**FINAL**

# REMEDIAL INVESTIGATION REPORT

# OPERABLE UNIT 7 OF THE LIBBY ASBESTOS SUPERFUND SITE

September 2014

Prepared for:

**MONTANA DEPARTMENT OF ENVIRONMENTAL QUALITY**
**Remediation Division**
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**REMEDIAL INVESTIGATION REPORT**

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## REVIEWS AND APPROVALS

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## ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| °C | Degrees Celsius |
| °F | Degrees Fahrenheit° |
| µm | Micrometers |
| AAS | Outdoor ambient air study |
| ABS | Activity-based sampling |
| AD | Address |
| AHERA | Asbestos Hazard Emergency Response Act |
| ASTM | American Society for Testing and Materials |
| ATSDR | Agency for Toxic Substances and disease Registry |
| BD | Building |
| bgs | Below ground surface |
| BNSF | Burlington Northern Santa Fe |
| CAMA | Computer assisted mass appraisal |
| CDC | Centers for Disease Control and Prevention |
| CDM | Camp, Dresser, and McKee |
| CERCLA | Comprehensive Environmental Response, Compensation, and Liability Act |
| CIC | Community Involvement Coordinator |
| CFR | *Code of Federal Regulations* |
| $cm^2$ | Square centimeters |
| COC | Chain of custody |
| CUA | Common use area |
| DEQ | Montana Department of Environmental Quality |
| DMP | Data management plan |
| DQO | Data quality objective |
| EDD | Electronic data deliverable |
| EPA | U.S. Environmental Protection Agency |
| ERS | Environmental resource specialist |
| ESAT | Environmental Services Assistance Team |
| FBAS | Fluidized Bed Asbestos Segregator |
| FS | Feasibility study |
| FSDS | Field sample data sheet |
| GIS | Geographic information system |
| GPS | Global positioning system |
| HEPA | High-efficiency particulate absorption |
| HHRA | Human health risk assessment |
| HASP | Health and safety plan |
| IDW | Investigation-derived waste |
| ISO | International Organization for Standardization |
| L/min | Liters per minute |
| LA | Libby amphibole |

## ACRONYMS AND ABBREVIATIONS (CONTINUED)

| | |
|---|---|
| LPM | Liters Per Minute |
| LUA | Limited use area |
| MARCH | OU7 document management archive |
| MBMG | Montana Bureau of Mines and Geology |
| MCE | Mixed cellulose ester |
| mph | Miles per hour |
| ND | Not detected |
| NIOSH | National Institute for Occupational Safety and Health |
| NOAA | National Oceanic and Atmospheric Administration |
| NUA | Non-use area |
| OU/OU7 | Operable unit/Operable Unit 7 of the Libby Asbestos Superfund Site |
| PDA | Personal digital assistant |
| PDF | Portable digital format |
| PLM | Polarized light microscopy |
| PLM-VE | Polarized light microscopy – visual estimation |
| POC | Point of contact |
| PPE | Personal protective equipment |
| PRI | Project Resources, Inc. |
| QA | Quality assurance |
| QARD | Site-wide Quality Assurance Reference Document for the Libby Asbestos Superfund Site Libby, Montana |
| QATS | Quality Assurance Technical Support |
| QC | Quality control |
| RAWP | Response action work plan |
| RDI | Removal design investigation |
| RI | Remedial investigation |
| ROD | Record of decision |
| ROM | Record of Modification |
| ROW | Right of way |
| RPD | Relative percent difference |
| $s/cm^2$ | Structures per square centimeter |
| s/cc | Structures per cubic centimeter |
| SAP | Sampling and analysis plan |
| SOP | Standard operating procedure |
| SSC | Site safety coordinator |
| SUA | Specific use area |
| TAAD | Troy Ambient Air Database |
| TAPE | Troy Asbestos Property Evaluation |
| Tetra Tech | Tetra Tech, Inc. |
| TEM | Transmission electron microscopy |
| TFO | Troy field office |
| TOAD | Troy Owner Access Database |
| TPIC | Third-party independent contractor |

## ACRONYMS AND ABBREVIATIONS (CONTINUED)

| | |
|---|---|
| UA | Use area |
| USACE | U.S. Army Corps of Engineers |
| USGS | U.S. Geological Survey |
| VCI | Vermiculite-containing insulation |
| VV | Visible vermiculite |
| VWC | Volumetric water content |
| WP | Work plan |

# EXECUTIVE SUMMARY

Tetra Tech, Inc. (Tetra Tech) was tasked by the Montana Department of Environmental Quality (DEQ) to conduct a remedial investigation (RI) at Operable Unit (OU) 7 of the Libby Asbestos Superfund Site and to prepare this report. The U.S. Environmental Protection Agency (EPA) is the lead agency for the Libby Asbestos Superfund Site and the DEQ is the lead agency for OU7 through a cooperative agreement with EPA. This report summarizes RI activities and findings at OU7 from 2007 in to early 2014.

The RI was undertaken to assess the nature and extent of Libby Amphibole asbestos (LA) contamination in OU7. The primary objectives of the RI were to:

- Adequately characterize the nature and extent of LA contamination in OU7 by investigating building interiors, soil, and ambient air;
- Provide community support and information on LA to OU7 residents during the investigations;
- Identify those parcels that meet current EPA removal action levels;
- Collect and provide accurate and reliable data on LA contamination in support of remedial design investigations and removal actions;
- Gather sufficient data to support a site-wide risk assessment; and
- Allow for the effective development and evaluation of alternative remedies to be included in the feasibility study (FS).

OU7 is located within the Kootenai River Valley northwest of Libby, Montana. It is one of eight OUs comprising the Libby Asbestos Superfund Site and encompasses the town of Troy, Montana. Troy is located approximately 18 miles west of Libby and, due to this proximity and historical connection to the vermiculite mine operation, it was determined that the presence of LA in Troy and its vicinity should be evaluated.

Vermiculite was mined and processed at the Rainy Creek vermiculite mine and associated processing operations located near Libby, Montana from the 1920s until 1990. The processed and exfoliated vermiculite was used primarily for insulation in buildings and as a soil amendment. Vermiculite from the Rainy Creek mine is contaminated with a unique combination of amphibole asbestos minerals, which has been classified as a combination of winchite, richterite, tremolite, magnesioriebeckite, edenite, and magnesio-arfvedsonite. Collectively, these amphibole asbestos minerals present in vermiculite from the Libby mine are referred to as LA. For decades, the processing of vermiculite ore

and generation and disposal of waste materials resulted in widespread amphibole asbestos contamination of the Libby community.

Asbestos exposure can cause a number of health problems in both humans and animals. Among these health problems are asbestosis, mesothelioma, cancer, pleural plaques, diffuse pleural thickening, and asbestos warts.

In 1999, EPA Region 8 dispatched an emergency response team to investigate media reports of amphibole asbestos contamination and high rates of asbestos-related disease in Libby. The Libby Asbestos Superfund Site consequently added to the National Priorities List in October of 2002.

EPA established removal action levels and clearance criteria for the Libby Asbestos Superfund Site based on screening-level risk calculations (EPA 2003), and eventually will issue a record of decision for all the OUs that it will set forth final removal action levels and clearance criteria. Until then, properties were evaluated against the following preliminary removal action levels and clearance criteria:

Attics/Walls

- Visual confirmation of open, non-contained, or migrating vermiculite insulation

Interiors

- Visual confirmation of vermiculite in the indoor living space
- Concentration of LA in an indoor dust sample greater than 5,000 LA structures per square centimeter (s/cm2) using Asbestos Hazard Emergency Response Act (AHERA) counting methods

Soils

- Visual confirmation of vermiculite or other vermiculite mine-related materials in specific use areas (SUA)(defined as a garden, former garden, planter, or other defined area of a yard likely to receive significant use and generally not covered with grass)
- Concentration of LA in an SUA or other yard soils by any analytical method greater than or equal to 1 percent LA

In addition to the EPA removal action criteria listed above, DEQ developed some additional lines of evidence which were used for removal action decision-making in OU7:

- Any knowledge of former miners, close relatives of miners, or any highly exposed persons living or visiting the building? = Yes

- Has a resident, past or present, been diagnosed with an asbestos-related disease? = Yes
- To the best of your knowledge, was vermiculite from the mine used in, or around, your home? = Yes
- Has the resident/business purchased any Libby vermiculite materials from W.R. Grace in the past? = Yes
- Has the property been used for a for-profit enterprise of distributing, treating, storing, or disposing of Libby vermiculite? = Yes
- Are Libby vermiculite additives in any of the building materials? = Yes

Between 2007 and 2013, Tetra Tech conducted several investigations at OU7, including Troy asbestos property evaluations (TAPE), an outdoor ambient air study, remedial design investigations, outdoor activity-based sampling (ABS), indoor ABS, and a soil background study. Results from each of these investigations are summarized below.

**Troy Asbestos Property Evaluation – Dust Samples**

TAPE Indoor dust samples were collected in 2007 and 2008. A total of 1,211 dust samples were collected from primary and secondary buildings (without dirt floors) in 2007. Four of the samples exceeded the EPA removal action level for indoor dust (an LA concentration greater than 5,000 s/cm$^2$ [EPA 2003]). One hundred and eighty-five samples had detected LA below 5,000 s/cm$^2$. LA was not detected in 1,022 of the 2007 dust samples.

A total of 573 dust samples were collected from primary and secondary buildings (without dirt floors) in 2008. Of these, results for two samples exceeded the EPA removal action level for indoor dust; an LA concentration greater than 5,000 s/cm$^2$ (EPA 2003). Ninety-seven samples had detected LA below 5,000 s/cm$^2$, and LA was not detected in 474 of the 2008 samples. The dust analytical results are summarized below (not including results for field QC samples).

**TAPE DUST SAMPLE ANALYTICAL RESULTS**

| Result | 2007 | 2008 |
|---|---|---|
| > 5,000 s/cm$^2$ | 4 | 2 |
| < 5,000 s/cm$^2$ | 185 | 97 |
| Non-detect | 1,022 | 474 |
| Total | 1,211 | 573 |

Notes:
> Greater than
< Less than
s/cm$^2$ Structures per square centimeter

**Troy Asbestos Property Evaluation – Soil Samples**

Soil samples were collected from use areas (UA) and indoor soil samples from accessible crawlspaces and dirt floors in primary and secondary buildings. Soil analytical results for all field soil samples, excluding road and alley samples, are presented in the table below by year, arranged by bin category. This table does not include results for field quality control (QC) samples.

The bin categories for LA in soil are:

Bin A = Non-detect
Bin B1 = Trace LA (less than 0.2 percent)
Bin B2 = Between 0.2 and 1 percent LA
Bin C = LA greater than or equal to 1 percent by weight (EPA action level for removal)

**TAPE SOIL SAMPLE ANALYTICAL RESULTS**

| | Number of Samples | | | | | | |
|---|---|---|---|---|---|---|---|
| Bin | 2007 | 2008 | 2009 | 2010 | 2011 | 2012 | 2013 |
| A | 1,274 | 894 | 426 | 227 | 146 | 91 | 51 |
| B1 | 637 | 327 | 228 | 49 | 62 | 15 | 10 |
| B2 | 13 | 5 | 2 | 0 | 0 | 0 | 0 |
| C | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Total | 1,924 | 1,226 | 656 | 276 | 208 | 106 | 61 |

Note:
Results include exterior, interior, and co-located soil samples.

**Troy Asbestos Property Evaluation – Soil Samples from Road and Alley Parcels**

The analytical results for soil samples collected from road and alley parcels are presented in the table below. This table does not include results from field QC samples.

**ROAD AND ALLEY SOIL SAMPLE ANALYTICAL RESULTS**

| Bin | Number of Parcels |
|---|---|
| A | 197 |
| B1 | 74 |
| B2 | 0 |
| C | 0 |
| Total | 271 |

**Removal Actions**

Each inspected parcel in OU7 was reviewed against the EPA and DEQ removal action criteria presented earlier to determine whether a removal action was necessary. The table below is a summary of the OU7 removal actions between 2008 and July 2013:

**OPERABLE UNIT 7 REMOVAL ACTIVITIES**

| Type of Removal Action | Completed in 2008 | Completed in 2009 | Completed in 2010 | Completed in 2011 | Completed in 2012 | Completed in 2013 | Total Completed |
|---|---|---|---|---|---|---|---|
| Interior Only | 1 | 1 | 55 | 5 | 1 | 1 | 64 |
| Exterior Only | 0 | 0 | 12 | 0 | 0 | 0 | 12 |
| Combination | 6 | 5 | 17 | 1 | 0 | 0 | 29 |
| TOTAL | 8 | 6 | 85 | 6 | 1 | 1 | 105 |

**Outdoor Ambient Air Program**

The outdoor ambient air study (AAS) program entailed quarterly monitoring of ambient air across OU7 over a three year period beginning in October 2009 and ending in June 2013. The primary objectives of the AAS were to measure LA concentrations in outdoor ambient air and combine the collected data with discrete data from other exposure routes to support the human health risk assessment (HHRA) and the evaluation of future remedial actions.

Monitoring station locations were established within four air zones to evaluate upwind, downwind and residential exposure scenarios. In three and half years of air sampling performed at 18 stations within four zones, LA detections were found at all but five stations. However, as shown in the summary of detected LA results below, out of up to 24 to 36 sampling events across each station, a total of 16 detections of LA were found. There were no notable seasonal or aerial trends within the ambient air stations.

| Station ID | N Sampling Events | N Samples with Detected PCME LA | PCME LA Air Concentration (s/cc) | | Mean Achieved Sensitivity (1/cc) |
|---|---|---|---|---|---|
| | | | Mean | Range of Concentrations | |
| **OU7 (Air Zone 1)** | | | | | |
| T1 | 35 | 2 | 3.2E-06 | 0.0E+00 - 7.4E-05 | 3.9E-05 |
| T2 | 35 | 2 | 2.2E-06 | 0.0E+00 - 3.8E-05 | 3.9E-05 |
| T11 | 35 | 1 | 1.1E-06 | 0.0E+00 - 3.9E-05 | 3.9E-05 |
| T12 | 32 | 1 | 1.2E-06 | 0.0E+00 - 3.9E-05 | 3.9E-05 |
| T21 | 24 | 0 | 0.0E+00 | all non-detect | 3.9E-05 |
| **OU7 (Air Zone 2)** | | | | | |
| T3 | 36 | 0 | 0.0E+00 | all non-detect | 3.9E-05 |
| T4 | 35 | 1 | 1.1E-06 | 0.0E+00 - 3.7E-05 | 3.9E-05 |
| T5 | 35 | 1 | 8.0E-07 | 0.0E+00 - 2.8E-05 | 4.4E-05 |
| T13 | 36 | 0 | 0.0E+00 | all non-detect | 3.9E-05 |
| T14 | 36 | 1 | 4.4E-07 | 0.0E+00 - 1.6E-05 | 4.6E-05 |
| T15 | 36 | 0 | 0.0E+00 | all non-detect | 3.9E-05 |
| T22 | 23 | 2 | 8.6E-06 | 0.0E+00 - 1.6E-04 | 4.6E-05 |
| **OU7 (Air Zone 3)** | | | | | |
| T6 | 36 | 1 | 9.9E-07 | 0.0E+00 - 3.6E-05 | 3.9E-05 |
| T16 | 35 | 0 | 0.0E+00 | all non-detect | 3.9E-05 |
| T23 | 23 | 2 | 3.5E-06 | 0.0E+00 - 4.0E-05 | 3.9E-05 |
| **OU7 (Air Zone 4)** | | | | | |
| T7 | 35 | 1 | 1.0E-06 | 0.0E+00 - 3.6E-05 | 3.9E-05 |
| T17 | 36 | 0 | 0.0E+00 | all non-detect | 3.9E-05 |
| T24 | 24 | 1 | 1.7E-06 | 0.0E+00 - 4.0E-05 | 3.9E-05 |

**Outdoor Activity-based Sampling Program**

The primary objective of outdoor ABS in OU7 was to obtain data needed to complete the HHRA by collecting data necessary to assess threats to human health from both residential and community-wide recreational sources of LA in air. Residential properties sampled during outdoor ABS in OU7 fall into two categories: (1) properties for which soil removal was not required based on the EPA and DEQ removal criteria, and (2) properties for which soil removal had been completed. Community-wide scenarios included recreational play in public parks, a school playground, and a Frisbee course. In addition, ABS activities were performed for driving and biking around the Troy area, which also included a child scenario consisting of a toddler trailer that was towed behind the adult on a bicycle. A total of 495 ABS air samples (including field QC samples) were collected under three residential-disturbance scenarios

and three community-wide disturbance scenarios in both the wet and dry seasons. Detected results for each scenario are presented in the tables below.

**DETECTED RESULTS FOR OUTDOOR ABS SCENARIOS AT NON-REMOVAL RESIDENTIAL PROPERTIES**

| Location | Sample ID | Season | Location Type | Location Description | Personnel Task | Scenario Description | Number of Structures | Result (s/cc) |
|---|---|---|---|---|---|---|---|---|
| AD-200438 | TB-00538 | DRY | SUA | Flowerbed | Digging/ Rototilling | Gardening in residential yards | 1 | 2.20E-04 |
| AD-200458 | TB-00296 | WET | CUA | Yard | Raking/Digging/ Mowing | Yard work in residential yards | 1 | 2.16E-04 |
| AD-200458 | TB-00300 | WET | SUA | Garden | Digging/ Rototilling | Gardening in residential yards | 1 | 2.16E-04 |
| AD-201096 | TB-00144 | WET | SUA | Driveway (unpaved) | Digging/ Bicycling (child) | Bicycling/playing on unpaved driveways | 2 | 4.37E-04 |
| AD-201096 | TB-00660 | DRY | CUA | Yard | Raking/Digging/ Mowing | Yard work in residential yards | 1 | 2.13E-04 |
| AD-201096 | TB-00665 | DRY | CUA | Former Garden | Digging/ Rototilling | Gardening in residential yards | 2 | 9.98E-04 |
| AD-201096 | TB-00670 | DRY | SUA | Driveway (unpaved) | Digging/ Bicycling (child) | Bicycling/playing on unpaved driveways | 4 | 8.59E-04 |

Note:
s/cc Structures per cubic centimeter

**DETECTED RESULTS FOR OUTDOOR ABS SCENARIOS AT POST-REMOVAL RESIDENTIAL PROPERTIES**

| Location | Sample ID | Season | Location Type | Location Description | Personnel Task | Scenario Description | Number of Structures | Result (s/cc) |
|---|---|---|---|---|---|---|---|---|
| AD-200594 | TB-00878 | DRY | CUA | Yard | Raking/ Digging/ Mowing | Yard work in residential yards | 2 | 4.39E-04 |
| AD-200634 | TB-00490 | WET | SUA | Driveway (unpaved) | Digging/ Bicycling (child) | Bicycling and playing on unpaved driveways | 1 | 2.12E-04 |
| AD-200634 | TB-00893 | DRY | SUA | Driveway (unpaved) | Digging/ Bicycling (child) | Bicycling and playing on unpaved driveways | 1 | 2.18E-04 |
| AD-200759 | TB-00955 | DRY | CUA | Yard | Raking/ Digging/ Mowing | Yard work in residential yards | 1 | 2.14E-04 |
| AD-200759 | TB-00963 | DRY | SUA | Driveway (unpaved) | Digging/ Bicycling (child) | Bicycling and playing on unpaved driveways | 1 | 2.15E-04 |
| AD-200880 | TB-00967 | DRY | SUA | Lean-To | Digging/ Bicycling (child) | Bicycling and playing on unpaved driveways | 1 | 2.15E-04 |

Note:
s/cc Structures per cubic centimeter

As the above tables show, detections of LA were found at both pre- and post-removal properties. There is a higher range of detections in the dry season versus the wet season at residential properties.

For the community-wide ABS scenarios shown below, there wase a high frequency of LA detections in the driving scenario, especially during the dry season.

**DETECTED RESULTS FOR OUTDOOR ABS COMMUNITY-WIDE SCENARIOS**

| Location | Sample ID | Season | Personnel Task | Scenario Description | Number of Structures | Result (s/cc) |
|---|---|---|---|---|---|---|
| AD-200335 | TB-00597 | DRY | Recreational sports & ball field | Recreational activities at parks and school yards | 1 | 2.20E-04 |
| AD-OU7NA | TB-00563 | DRY | Driving | Driving on paved and unpaved roads and alleys | 6 | 1.30E-03 |
| AD-OU7NA | TB-00565 | DRY | Driving | Driving on paved and unpaved roads and alleys | 2 | 4.09E-04 |
| AD-OU7NA | TB-00568 | DRY | Driving | Driving on paved and unpaved roads and alleys | 2 | 4.14E-04 |
| AD-OU7NA | TB-00570 | DRY | Driving | Driving on paved and unpaved roads and alleys | 3 | 6.39E-04 |
| AD-OU7NA | TB-00573 | DRY | Driving | Driving on paved and unpaved roads and alleys | 4 | 8.73E-04 |
| AD-OU7NA | TB-00578 | DRY | Driving | Driving on paved and unpaved roads and alleys | 13 | 2.80E-03 |
| AD-OU7NA | TB-00581 | DRY | Driving | Driving on paved and unpaved roads and alleys | 1 | 2.07E-04 |
| AD-200335 | TB-00414 | WET | Recreational sports & ball field | Recreational activities at parks and school yards | 3 | 6.37E-04 |
| AD-OU7NA | TB-00322 | WET | Bicycling (infant in trailer) | Bicycling on paved and unpaved roads and alleys | 1 | 2.20E-04 |

Note:
s/cc Structures per cubic centimeter

**Indoor Activity-based Sampling**

The primary objective of indoor ABS in OU7 was to obtain data needed to complete the HHRA, by collecting activity-based air samples from residential structures. Residential properties in OU7 fall into two categories: (1) properties for which removal was not required based on removal criteria, and (2) properties for which removal had been completed.

A total of 138 indoor ABS air samples (including field QC samples) were collected in 2012 and 2013 under two residential-disturbance scenarios. As shown in the table below, unexpectedly, there appears

to be a higher incidence of LA detections in the post removal indoor properties than the Pre-removal properties and, as expected, higher incidence of LA detections in the active versus the passive scenarios.

| Category | Type of ABS Scenario | Number of Samples | Average of Result | Average of Sensitivity |
|---|---|---|---|---|
| Indoor ABS from post-removal property | Active | 14 | 5.6E-05 | 5.1E-05 |
| Indoor ABS from post-removal property | Passive | 14 | 1.6E-05 | 1.8E-05 |
| Indoor ABS from non-removal property | Active | 26 | 3.0E-05 | 5.2E-05 |
| Indoor ABS from non-removal property | Passive | 26 | 1.2E-05 | 1.9E-05 |

**Soil Background Study**

A background soil study was performed for OU7 to determine the possible presence of LA in background soils associated with naturally-deposited glacial sediments in and around OU7. Soil and co-located ABS air samples were collected. A summary of the findings is as follows:

- The detection frequency was 7/11.
- The minimum concentration was 0 s/cc.
- The maximum concentration was 1.1E-03 s/cc.
- The average concentration was 3.2E-04 s/cc.
- The average sensitivity was 4.4E-04 s/cc (or this might be 2.1E-04 s/cc, if the indirect preparation factor is included in the sensitivity calculation).

The following conclusions were drawn from the results of the study:

- LA was detected in background soils in and around OU7 that are not thought to be affected by anthropogenic releases from vermiculite mining and processing activities.
- The types of LA structures in background soils within the Kootenai Valley indicate that a portion of the LA structures observed are unlikely to have originated at the Libby vermiculite ore body.
- The concentrations of LA in background soils (≤ 0.02 percent) is well below the detection limit of polarized light microscopy visual estimation method (PLM-VE), but can be reliably detected by preparing the samples using Fluidized Bed Asbestos Segregator (FBAS) method, following preparation using the rock flour method, and analysis by TEM. This analytical protocol appears to be a more sensitive metric of LA detection in soil than either ABS or field visible vermiculite observations.

The results support the conclusion that there is a non-zero level of LA in soils in and around OU7 that is not attributable to vermiculite mining and processing activities associated with the W.R. Grace mine. Further, these results support the data reported by Adams and others 2010; Langer et al 2010; and Gunter and Sanchez 2009, indicating that low level detections of amphibole fibers in soils are present within the Kootenai Valley, in part, originating from normal geologic, soil building processes unrelated to mining and milling of vermiculite ore from Vermiculite Mountain.

**Conclusions**

This final RI report for OU7 summarizes RI activities and findings from OU7 resulting from the TAPE, outdoor AAS, RDIs , outdoor ABS SAP, indoor ABS sampling, and the soil background study. It does not include an HHRA or ecological risk assessment because they are being prepared separately on a Libby Asbestos Superfund Site-wide basis. The EPA has custody of all electronic data collected for the OU7 RI.

The information presented in this final RI report indicates that LA from the Libby mine is present in OU7 as a result of mining activities, inadvertent transport and dispersal from the Libby mine by residents within OU7, commercial use of vermiculite from the Libby mine, and natural processes. Evaluation of the health effects to residents and ecological receptors within OU7 is the subjects of a separate report, which, together with this RI report, will provide the necessary information to formulate remedial alternatives for OU7.

# 1.0 INTRODUCTION

Tetra Tech, Inc. (Tetra Tech) was tasked by the Montana Department of Environmental Quality (DEQ) under DEQ Contract No. 407026, Task Order No. 113, to prepare a remedial investigation (RI) report for Operable Unit (OU) 7 of the Libby Asbestos Superfund Site. The U.S. Environmental Protection Agency (EPA) is the lead agency for the Libby Asbestos Superfund Site, and the DEQ is the lead agency for OU7 through a cooperative agreement with EPA. This RI report summarizes activities and findings conducted pursuant to the Troy Asbestos Property Evaluation (TAPE) work plan (Tetra Tech 2007), the Outdoor Ambient Air Study (AAS) work plan (Tetra Tech 2009a), the Removal Design Investigation (RDI) sampling and analysis plan (SAP) (Tetra Tech 2010a), the Final Human Health Risk Assessment Work Plan (Tetra Tech 2011a) the Activity-Based Sampling (ABS) SAP (Tetra Tech 2011b), the Indoor Activity-based Sampling SAP (Tetra Tech 2012a), and the Final Background Soil Study SAP (Tetra Tech 2012b). These investigations were undertaken to assess the nature and extent of Libby Amphibole asbestos (LA) contamination within OU7.

## 1.1 REPORT ORGANIZATION

This RI report is organized into 14 sections, which are followed by figures, appendices, and attachments. The tables appear within the text. The contents of Sections 1.0 through 14.0 are briefly described below.

Section 1.0 – Introduction: describes the report organization, purpose, objectives, site description, site history, and regulatory history

Section 2.0 – Physical Characteristics and Environmental Setting: describes the climate, surface water hydrology, geology, soil, hydrogeology, population and land use, and ecology of OU7 and the surrounding area

Section 3.0 – Libby Amphibole Asbestos: describes the physical and chemical properties, historical use, health effects, and EPA removal action levels

Section 4.0 – Troy Asbestos Property Evaluation and Remedial Design Investigation Activities: describes the TAPE objectives, investigation methods, sample analysis, data management and data quality assessment, RDI, and deviations from the TAPE work plan (Tetra Tech 2007)

Section 5.0 – Outdoor Ambient Air Study: describes the AAS objectives, investigation methods, analytical methods, field quality control (QC) samples, data quality assessment, and deviations from the work plan (Tetra Tech 2009a)

Section 6.0 – Outdoor Activity-Based Sampling: describes outdoor activity-based sampling (ABS) objectives and investigation methods, field QC samples, data quality assessment, and deviations from the SAP (Tetra Tech 2011b)

Section 7.0 – Indoor Activity-Based Sampling: describes indoor ABS objectives and investigation methods, field QC samples, data quality assessment, and deviations from the SAP (Tetra Tech 2012a)

Section 8.0 – Background Soil Study: briefly introduces the background soil study (Tetra Tech 2012b), which is included as Attachment 2 to this RI report

Section 9.0 – Additional Removal Activities: describes additional removal activities (Callahan Creek remediation, Burlington Northern Santa Fe Railroad right-of-way removal, and immediate residential removal actions) conducted during the course of the project

Section 10.0 – Field Results and Nature and Extent of Contamination: presents the results and nature and extent of contamination based on field observations and data gathered during TAPEs, RDIs, AAS, indoor and outdoor ABS, and background soil investigation

Section 11.0 – Contaminant Fate and Transport: presents the understanding of how LA is released, transported, deposited, transformed, and degraded in the environment

Section 12.0 – Risk Assessment: summarizes the status of the risk assessment for OU7

Section 13.0 – Summary and Conclusions: presents a summary of the RI activities and conclusions based on the RI results

Section 14.0 – References: lists the references cited in the report

In addition, the document contains Appendices A through C and two Attachments as described below.

Appendix A– Project Specific Laboratory Modifications and Standard Operating Procedures: presents the many analytical laboratory modifications and procedures that changed over the course of the RI

Appendix B- Troy Field Office Modification Records: contains records of variances from the field sampling plans

Appendix C- Preliminary List of Applicable or Relevant and Appropriate Requirements

Attachment 1-Libby Asbestos Site, Troy OU7 Residential/Commercial Cleanup Criteria, Specific Use Area Visible Vermiculite Action Level Technical Memorandum

Attachment 2-Background Soil Study Report-A stand alone report describing the details of the background soil study

## 1.2 REMEDIAL INVESTIGATION REPORT PURPOSE AND OBJECTIVES

The overall purpose of an RI is to collect necessary data such that the nature and extent of contamination can be adequately characterized and potential remedial alternatives evaluated. Specifically, the purpose of this RI report is to describe the approach used during the TAPE, AAS, ABS, background soil study, and removal activities; present the results of those investigations; and use the data to make informed recommendations about the need for remedial action. The following primary RI objectives for OU7 were identified:

- Adequately characterize the nature and extent of LA contamination in OU7 by investigating building interiors, soil, and ambient air;
- Provide community support and information on LA to OU7 residents during the investigations;
- Identify those parcels that meet current EPA removal action levels;
- Collect and provide accurate and reliable data on LA contamination in support of RDIs and removal actions;
- Gather sufficient data to support a site-wide risk assessment; and
- Allow for the effective development and evaluation of alternative remedies to be included in the FS.

Tetra Tech prepared this RI report in accordance with the EPA's *Guidance for Conducting Remedial Investigations and Feasibility Studies Under the Comprehensive Environmental Response , Compensation, and Liability Act (CERCLA)* (EPA 1988), as appropriate. This final RI report incorporates the annual updates to the draft final report (Tetra Tech 2010b) for 2010 (Tetra Tech 2011c) and 2011 (Tetra Tech 2013).

The EPA removal action levels and clearance criteria are outlined in the document *Libby Asbestos Site Residential/Commercial Cleanup Action Level and Clearance Criteria Technical Memorandum* (EPA 2003), that provides the initial guidance for identifying properties within the Libby Asbestos Superfund Site that require (or are eligible for) a removal action. Alterations to EPA action level and clearance criteria and their effect on subsequent sampling requirements and protocols are documented in this RI report. Similarly, this final RI report documents the results of additional investigations and their impact on the delineation of LA contamination within OU7 since issuance of the draft final RI report in 2010.

A site-wide risk assessment is being prepared under separate cover and, therefore, is not included in this final RI report.

## 1.3 SITE DESCRIPTION

OU7 is located within the Kootenai River valley northwest of Libby, Montana at an elevation ranging from 1,850 feet above mean sea level along the Kootenai River to 2,500 feet above mean sea level on the mountain slopes surrounding the valley. OU7 is approximately 8 miles long and 1.8 miles wide at its broadest point. The topography of OU7 varies from gently graded, open land along the Kootenai River to terraced hillsides and steep forested mountains adjacent to the river valley.

OU7 is one of eight OUs comprising the Libby Asbestos Superfund Site and encompasses the town of Troy, Montana. Troy is located approximately 18 miles west of Libby, and due to this proximity and historical connection to the vermiculite mine operation, it was determined that the presence of LA in Troy and its vicinity should be evaluated. Figure 1-1 displays the location of the Libby Asbestos Superfund Site in Montana. Figure 1-2 shows the location of each OU, described as follows:

- OU1 - Includes the former export plant and the Highway 37 embankments. OU1 is bounded by residential areas to the east and west, the Kootenai River to the north, and the Burlington Northern Santa Fe (BNSF) railroad lines and right of way (ROW) to the south.
- OU2 –Is defined by contamination discharged from the former screening plant. These areas include the former screening plant, the Flyway property, the ROW of Highway 37 and Rainy Creek Road adjacent to the screening plant and several privately owned properties.
- OU3 – Includes the former vermiculite mine and the adjacent land impacted by the operations (the ponds, Rainy Creek Road, Rainy Creek and a portion of the Kootenai River) and areas in which tree bark is contaminated with LA. The boundary of OU3 is defined based on the extent of the contamination released from the former vermiculite mine.

- OU4 – Is defined as the residential, industrial, commercial and public properties in and near the City of Libby, or those that received material from the mine that were not associated with W.R. Grace operations.
- OU5 – The boundary of OU5 includes the parcel of land which was the site of the old Stimson Lumber Company mill. This OU is bounded by the BNSF railroad to the north, Libby Creek to the east, and OU4 to the south and west.
- OU6 – Includes the BNSF railroad properties and ROW. It extends from the eastern boundary of OU4 to the western boundary of OU7 and includes the contamination associated with the Libby and Troy rail yards.
- OU7 – Is defined as all residential, commercial, and public properties in and around the City of Troy.
- OU8 – Includes all United States and Montana State Highways and secondary highways adjacent to and within all OUs of the Libby Asbestos Superfund Site.

The OU7 boundary was selected to ensure that TAPE investigations evaluated the majority of the older homes in and around Troy, which were more likely to be contaminated. Based on unique conditions, the DEQ and EPA conducted additional TAPE investigations for several properties outside of the OU7 boundary. Most of the additional investigations occurred in the Bull Lake area, south of OU7 along Highway 56. Only a few investigations occurred north of OU7 toward the town of Yaak, Montana. Figure 1-3 displays the boundary of OU7.

## 1.4 SITE HISTORY

Vermiculite was mined and processed at the Rainy Creek vermiculite mine and associated processing operations located near Libby, Montana from the 1920s until 1990. While it was in operation, the vermiculite mine near Libby may have produced 80 percent of the world's supply of vermiculite (Tetra Tech 2007). The processed and exfoliated vermiculite was used primarily for insulation in buildings and as a soil amendment.

The Libby vermiculite deposit is contaminated with amphibole asbestos. For decades, the processing of vermiculite ore and generation and disposal of waste materials resulted in widespread amphibole asbestos contamination of the Libby community. In 1999, EPA Region 8 dispatched an emergency response team to investigate media reports of amphibole asbestos contamination and high rates of asbestos-related disease in Libby. The Libby Asbestos Superfund Site (Figure 1-1) was consequently added to the National Priorities List in October of 2002. Subsequent environmental investigations have

found many areas in and around Libby contaminated with LA and the EPA eventually split the Libby Asbestos Superfund Site into eight OUs to further focus the remedial activities in each unit of the site.

Some of the workers at the vermiculite mine lived in Troy and commuted to the mine to work each day. The workers were exposed to asbestos-contaminated materials at the mine and processing facilities, and they transported asbestos-contaminated dust to their homes on their clothing and equipment. Residents of Troy also traveled to Libby for everyday activities such as shopping, working (other than at the mine), and attending school sporting events and likely came in contact with LA in Libby during these frequent visits.

Using funds provided by EPA through a cooperative agreement, DEQ hired Tetra Tech in 2005 to evaluate the extent of the LA contamination in OU7. A database of all OU7 properties was first assembled and TAPE inspections began in the spring of 2007. The intent was to perform a systematic screening of Troy area residences, public areas, schools, businesses, and road surfaces to gather sufficient information to determine how many Troy area properties were contaminated with LA. TAPE inspections were completed at approximately two-thirds of the OU7 parcels between 2007 and 2013.

In addition to the TAPE, Tetra Tech conducted an AAS, indoor and outdoor ABS studies, a background soil study, and removal activities during the course of the OU7 investigation.

## 1.5 REGULATORY HISTORY

Asbestos is a hazardous substance as defined by 40 Code of Federal Regulations (CFR) Section 302.4 of the National Oil and Hazardous Substances Pollution Contingency Plan. In November 1999, the EPA responded to requests from the State of Montana to investigate the potential exposure to asbestos related to the former mine operations and vermiculite processing. The EPA Response Team briefly inspected the former mine and processing facilities, interviewed local officials and members of impacted families, interviewed a pulmonologist in Spokane, Washington, and collected a small set of initial samples. A more detailed summary of the initial Libby Site evaluation is found in the EPA Action Memorandum dated May 23, 2000, *Request for a Time Critical Removal Action Approval and Exemption from the 12-month, $2-million Statutory Limit at the Libby Asbestos Site-Export Plant and Screening Plant former Processing Areas, Libby, Lincoln County, Montana* (EPA 2000). The initial investigation revealed two significant findings:

- There are a large number of current and historic cases of asbestos-related diseases centered around Libby, Montana.
- There is a likelihood that significant amounts of asbestos-contaminated vermiculite still remained in and around Libby.

These findings led the EPA to initiate a larger scale rapid investigation in December 1999 to meet the following goals:

- Obtain information on airborne asbestos levels in Libby in order to judge whether time-critical intervention was needed to protect public health
- Obtain data on asbestos levels in potential source materials (at the Export Plant and Screening Plant), and identify the most appropriate analytical methods to screen and quantify asbestos in source materials

Under CERCLA Section 104, the EPA has the authority to complete both removal and remedial actions. To date, all removals have been conducted using removal action authority to facilitate the timely removal of the most contaminated areas. The initial removal authority for time-critical actions began at the processing areas in May 2000 (EPA 2000). As additional areas requiring removal were identified, amendments to the initial action memo were approved and are briefly summarized below:

- May 23, 2000 – Initial Action Memorandum approving removal actions at the Export Plant and Screening Plant (EPA 2000)
- July 20, 2001 – Amendment increasing the funding ceiling for continued removal actions at the Export Plant and Screening Plant, and expanding the scope of removal actions to include Rainy Creek Road, Libby High School, Libby Middle School, Plummer Elementary (hereafter referred to as Kootenai Valley Head Start), and two private properties (EPA 2001)
- May 2, 2002 – Amendment increasing the funding ceiling and expanding the scope of removal actions to address residential/commercial properties at OU4 (EPA 2002)
- May 15, 2006 – Amendment increasing the funding ceiling for removal actions at additional properties at OU4 (EPA 2006a)
- June 27, 2006 – Amendment increasing the funding ceiling and expanding the scope of removal actions to include Troy, Montana (EPA 2006b)
- September 24, 2008 – Amendment increasing the funding ceiling and expanding the scope of removal actions to include specific creeks in Libby and Troy, Montana (EPA 2008)
- June 17, 2009 – Amendment increasing the funding ceiling for additional residential/commercial removal actions in Libby and Troy, Montana (EPA 2009a)
- August 13, 2009 – Amendment expanding the scope of removal actions to include the Cabinet View Country Club Golf Course (EPA 2009b)

- March 14, 2012 – Amendment to request and document approval for changes to removal action and removal protocols described in the original Action Memorandum and previous Amendments, and to outline a new neighborhood clean-up approach (EPA 2012a)
- August 28, 2012 – Amendment to address the removal of LA-containing vermiculite waste in the Rainy Creek floodplain in OU3 (EPA 2012b)

In October 2002, the Libby site was listed on the EPA's Superfund NPL, making it eligible to receive additional federal funds for investigation and removal, and transition the project from EPA's Removal Program to the Remedial Action Program. In 2009, the EPA declared a Public Health Emergency in Libby to provide federal health care assistance for victims of asbestos-related disease.

To facilitate a multi-phase approach to remediation of the Libby site, eight separate OUs were established. These were described in Section 1.3. OU1 and OU2 boundaries have been established; however, the boundaries for the other OUs will not be determined until the record of decision (ROD) is published. The OUs are shown on Figure 1-2.

# 2.0 PHYSICAL CHARACTERISTICS AND ENVIRONMENTAL SETTING

The following sections describe the physical characteristics and environmental setting for OU7, including the climate, surface water hydrology, geology, soil, hydrogeology, population and land use, and ecology.

## 2.1 CLIMATE

The climate within OU7 is typically mild during the summer and cold during the winter. August is the warmest month of the year with an average monthly high of 85.2 degrees Fahrenheit (°F). January is the coldest month of the year with an average monthly high of 32.7 °F. The average minimum monthly temperatures range from 19.8 °F in January to 50 °F in July. The average annual temperature is 46.7 °F (National Oceanic and Atmospheric Administration [NOAA] 2010).

Troy's wettest month of the year is November with an average precipitation of 3.66 inches. Maximum monthly averages occur in November and minimum monthly averages occur in August. These averages range from 3.21 inches to 1.19 inches, respectively. The average annual precipitation is 25.46 inches (NOAA 2010). In addition, the annual average snowfall is 30.4 inches, with the most snow typically falling in December and January (Western Regional Climate Center 2010).

## 2.2 SURFACE WATER HYDROLOGY

Surface water within OU7 mainly consists of rivers, lakes, and streams. There are no mapped wetlands for OU7 according to the Montana Natural Resource Information Service Land Cover Dataset map (Natural Resource Information Service 2010). The primary surface water body is the Kootenai River. The Kootenai River has a shallow gradient and flows southeast to northwest through the center of OU7. It traverses roughly eight miles of OU7, entering OU7 from the south, near Wilderness Plateau, and exiting OU7 to the northwest, near Kootenai Vista.

The northern portion of OU7 ranges in elevation from 1,800 feet above mean sea level in the valley to 2,500 feet above mean sea level in the surrounding mountains. Several creeks flow from the mountains into the Kootenai River. Ruby Creek enters on the west side of the Kootenai River and across from the Kootenai Vista. Further south, Brush Creek also enters the Kootenai River on the west side. Callahan Creek and Lake Creek enter the Kootenai River from the west at the southern end of OU7.

East of the Kootenai River, there are four lakes that range in elevation from 2,400 feet above mean sea level to 2,600 feet above mean sea level. These lakes include Alvord Lake, Slee Lake, Moose Lake, and Duck Lake. The largest lake in the area is Bull Lake, which is approximately 10 miles south of OU7.

The nature and extent of Libby vermiculite contamination in the Kootenai River is being investigated as part of OU3. Therefore, surface water of the Kootenai River passing through OU7 was not investigated as part of this RI.

## 2.3 GEOLOGY

OU7 lies within a system of linear, northwest to southeast trending , large thrust fault zones and intervening structural basins referred to as the Disturbed Belt or Precambrian Belt Series rocks. The mountains surrounding the Kootenai River canyon consist of a sedimentary sequence of rocks of shallow-water marine origin that are 35,000 to 50,000 feet thick and show a great variety of structural features. These metasediments are Proterozoic in age (900 to 1,500 million years old) and lie unconformably on the crystalline basement rocks below. Regional low-grade metamorphism has altered the original sedimentary rocks--sandstone, silty shale, and carbonates to quartzite, argillite, and impure dolomite (Montana Bureau of Mines and Geology [MBMG] 1963).

The mountains that bound the Kootenai River canyon and the bedrock underlying the valley consist of metamorphosed sedimentary rocks that formed in the Middle Proterozoic Period of the Precambrian Era. The ridge-forming units in the immediate vicinity of OU7 to the northeast and southwest consist primarily of four metasedimentary units. The thinly laminated dark-green to black argillite and light-green siltite of the upper member of the Wallace Formation grades vertically into the overlying green argillite and siltite of the Green facies of the Snowslip Formation. Chlorite is common on the bedding surfaces of the Green facies of the Snowslip (Harrison, Cressman, and Whipple 1992). Stratigraphically overlying the Wallace and Snowslip are the Shepard and Mount Shields formations. The Shepard Formation is a carbonate-bearing, green, platy, dolomitic to slightly dolomitic, interlaminated argillite and siltite that is commonly sheared and tightly folded. Overlying the Shepard Formation is the Mount Shields Formation consisting of black and green or white, thinly-laminated argillite and siltite in its uppermost member which conformably overlies a gray, silty, stromatolitic dolomite of the lower member (Harrison, Cressman, and Whipple 1992). Farther to the northwest beyond the closer ridges of

the canyon are the evenly-bedded, medium-dark to medium-light gray argillite and siltite of the Pritchard formation (Harrison and Cressman 1993).

The Kootenai River Valley is mainly composed of Quaternary unconsolidated fluvial glacial deposits of Pleistocene age (10,000 years to 1.6 million years before present) and alluvial and landslide deposits of Holocene age (recent to 10,000 years before present). The glacial deposits unconformably overlie the Precambrian rocks in the form of ground and end moraines, outwash, and other fluvial glacial deposits. The alluvium in the river valley consists of relatively well-sorted gravel, sand, silt, and clay and is present in the floodplains and low terraces along the drainages (Harrison, Cressman, and Whipple 1992). The landslide deposits consist of angular blocks of bedrock in finer grained matrices that are sometimes mixed with glacial debris (Harrison and Cressman 1993).

### 2.3.1 The Rainy Creek Igneous Intrusive Body

Approximately 20 miles to the east of OU7 and seven miles northeast of Libby is an alkaline-ultramafic igneous complex is known as the Rainy Creek Complex. The Rainy Creek Complex lies in the basin of Rainy Creek and is much less resistant to erosion than the surrounding Belt series metamorphic rocks. The rocks of this igneous complex formed by intrusion into the Precambrian Belt series (Wallace Formation) and are predominantly composed of biotite pyroxenite, magnetite pyroxenite, and biotite (Boettcher 1967). Hydrothermal alteration and extensive weathering of the ultramafic units resulted in the formation of a rich deposit of vermiculite. Later intrusions of alkaline magmas caused hydrothermal alteration of the pyroxenes resulting in formation of amphiboles. Approximately one-half of the amphiboles occur in the asbestiform habit (University of Idaho, modified from Bandli, 2002).

The biotite pyroxenite makes up approximately 20% of the intrusion and this unit is the source of all the mineable vermiculite. The vermiculite content varies significantly, but on average is 25 weight percent of the biotite pyroxenite body (Boettcher 1966). The biotite pyroxenite ranges in size from less than one millimeter to greater than 10 centimeters, and is composed of variable amounts of clinopyroxene (diopside), biotite, vermiculite, and hydrobiotite. In hand sample, it is dark green and friable (University of Idaho, modified from Bandli, 2002).

### 2.3.2 Vermiculite Mineralogy

Vermiculite is a general term applied to a group of platy, mica-like, hydrated silicate minerals that are the products of aqueous alteration of micas, primarily the biotite subgroup. They are often similar in appearance to the platy morphology of the replaced mica. Typically, the biotite grains alter to hydrobiotite or chlorite, then to vermiculite (Bush 1976). Vermiculites encompass a wide range of chemical compositions, vary in color from light yellow to green to brown to black, and generally have a bronze hue. Their distinctive characteristic is a prominent exfoliation when heated from 800 to 1,100 degrees Celsius (°C). When heated, the vermiculite plates expand at right angles to the cleavage (accordion-like) as the contained water is converted to steam ([U.S. Geological Survey] USGS 2005). Vermiculite was mined for 67 years from the Rainy Creek Complex and used in numerous consumer products in its expanded form.

## 2.4 SOIL

The soil types in OU7 reflect the geology of the area. The surface geology is comprised mainly of glacial and fluvial glacial deposits composed of Precambrian rocks that were broken and crushed, and then deposited as till by moving ice. The till is composed of poorly sorted beds of boulders, gravel, sand, and silt and clay lenses (MBMG 1978). Therefore, the soil characteristics include silty glaciolacustrine deposits and stratified sandy and gravelly outwash. Typically, below the surface, the profile of the silty glaciolacustrine deposits consists of silty loam. The stratified sandy and gravelly outwash consists of gravelly silt loam and very gravelly fine sandy loam (Natural Resources Conservation Service 2010).

Libby vermiculite was regularly added to native soil in and around the Libby Asbestos Superfund Site. Vermiculite has superior water retention properties, providing a good source of moisture for plants in poor soils. Raw, unexfoliated vermiculite was widely used as an amendment to aerate clayey exterior soils around the Libby Asbestos Superfund Site. This RI concentrates on soil amended with Libby vermiculite as opposed to undisturbed native soils.

## 2.5 HYDROGEOLOGY

The majority of OU7 is located in the Kootenai River canyon, which is underlain entirely by the pre-Cambrian rocks of the Belt Supergroup. Structural deformation of the Precambrian rocks has resulted in north- to northwest-trending folds, faults, and fracture systems. Natural recharge to the

groundwater system is from precipitation and streamflow, and natural discharge occurs at springs, by evapotranspiration, and as streamflow. Water moves readily between the groundwater system and the surface water system. During the wetter months of May through September, surface water flow is supplied by runoff, whereas during the remainder of the year, surface flow is maintained by groundwater discharge from the Precambrian rocks in the stream headwaters and from unconsolidated deposits in the river valley (MBMG 1978).

The alluvium and fluvial-glacial deposits are hydraulically connected and can be considered one aquifer. Because of the variability of the composition of the unconsolidated sediments, the transmissivity of the aquifer varies considerably. However, it is greatest along the relatively well-sorted sands, gravels, and cobbles of the alluvial deposits nearest the Kootenai River.

Small amounts of water are probably available from the fractures in the Precambrian bedrock but a study conducted by the MBMG of wells in the Libby area reported that very few wells are known to tap the Belt Series rocks (MBMG 1978). Glacial deposits composed of poorly sorted gravels and sands and silty clay lenses may contain adequate water for domestic use, but the clay lenses are of low permeability. Lakebed deposits of clay, silt, and fine-grained sediments have low permeability and yield little to no water to wells. Alluvium, which consists of coarser well-sorted sands, gravels, and cobbles, is more permeable and yields the greatest amounts of water to wells. Yields to alluvial wells will vary with the thickness of the deposits (MBMG 1978).

Water level fluctuation in aquifers depends on the distance from a stream and how well the aquifer is connected hydraulically to the Kootenai River or nearby stream. Aquifers that are close to the river or tributary streams and are in good hydraulic connection show rapid response to changes in river stage. Wells that are completed in the Precambrian bedrock or are relatively distant from rivers show smaller responses and a lag in water level response. Groundwater in the OU7 area varies in depth and includes land from 19 sections in three townships. A search of the MBMG Ground Water Information Center database shows that the average static water level in wells in these sections range from 30 feet to 64 feet with a minimum depth of zero feet and a maximum depth of 210 feet (Ground Water Information Center 2010).

## 2.6 POPULATION AND LAND USE

As of 2000, 18,837 people reside in Lincoln County (U.S. Census [Census] 2000). Of this number, approximately 1,982 live within the boundary of OU7, which is about 10.5 percent of the county's total population. Of this total, 957 reside within the Troy city limits (Census 2000).

As of 2000, the median household income in Troy is $19,635. The primary occupation categories in Troy were service (24.3 percent) and professional or management (19.5 percent). Unemployment is high in Troy; of those persons aged 16 years and over, only 52.5 percent are in the labor force. The labor force is comprised of 55.6 percent private wage and salary workers, 27.8 percent government workers, and 15.7 percent self-employed workers. Approximately 80 percent of the population 25 years and older have high school diplomas or higher (i.e. associate degree) and 13.7 percent have bachelor's degrees or higher (i.e. master's degree) (Census 2000).

The area of Lincoln County is approximately 2,350,838 acres. Most of the county is owned by governmental entities. Only 513,371 acres (22 percent) are privately owned. 1,759,003 acres (75 percent) are owned by the Federal government and 74,461 acres (3 percent) are owned by the State of Montana. The U.S. Forest Service and Plum Creek Timber are the largest landowners in Lincoln County owning 1,727,772 acres (73.5 percent) and 294,603 acres (12.5 percent), respectively (Census 2000).

OU7 covers approximately 3,560 acres within Lincoln County and is rural in nature. Approximately 44.3 percent of the land is classified as rural farming or agricultural property. Roughly 36.8 percent of the property in OU7 is rural residential, which includes both developed and vacant land. The remainder of the land is classified as exempt (federal, state, city property) at 7.8 percent, urban residential (both developed and vacant) at 5 percent, and commercial at 3.2 percent (Base Map Service Center 2010).

## 2.7 ECOLOGY

According to the U. S. Forest Service, the forested areas surrounding Troy are home to over 350 fish, mammal, bird, reptile, and amphibian species; however, it is unlikely that many of these animals would be encountered in developed areas that are inhabited by humans and generally free of dense forests. Troy and the surrounding area exhibit plant species that are dominant within the Kootenai

National Forest, as well as other non-native plant species imported primarily for landscaping purposes.

Threatened, endangered, and protected species in the area include grizzly bear, gray wolf, Canada lynx, and bald eagle. These animals have either been observed or could possibility be encountered in areas of OU7.

Gray wolves are the only threatened, endangered, or protected animals found in packs within OU7. There are currently nine documented gray wolf packs (four breeding packs) within the Kootenai National Forest, which includes OU7. Coyotes are the only other animal living in packs within OU7; however, the population of coyotes is larger than the population of wolves.

Approximately 30-40 grizzly bears roam the surrounding Kootenai National Forest. The population of Canada lynx in the surrounding forest is currently being studied. Bald eagles have been seen throughout the area.

# 3.0 LIBBY AMPHIBOLE ASBESTOS

This section includes a summary of the physical and chemical properties, historical use, and health effects of LA and LA-containing vermiculite.

## 3.1 PHYSICAL AND CHEMICAL PROPERTIES

Vermiculite is a naturally occurring mineral in the phyllosilicate family. It is formed by weathering or hydrothermal alteration of other minerals such as biotite or phlogopite. Pure vermiculite is non-toxic and does not contain asbestos. Asbestos is the commercial name for a specific group of long, very thin, naturally occurring, hydrated silicate minerals (Van Gosen 2007). The asbestos minerals are categorized into two groups based on morphology, serpentine and amphibole. Asbestos fibers from the serpentine group (chrysotile) are curly, sheet-like structures. Asbestos fibers from the amphibole group are needlelike structures. The vermiculite obtained from the Rainy Creek mine in Libby was contaminated with a unique combination of amphibole asbestos minerals, which has been classified as a combination of winchite, richterite, tremolite, magnesioriebeckite, edenite, and magnesio-arfvedsonite (Meeker et al. 2003). Collectively, these amphibole asbestos minerals present in vermiculite from the Libby mine are referred to as LA.

All asbestos minerals contain magnesium, silica, and water and are usually formed by regional or contact metamorphism or magmatic hydrothermal thermal reactions of magnesium-rich rocks such as dolomite. Asbestos minerals occur in bundles of long, thin mineral fibers. The bundles easily separate when crushed or are otherwise disturbed. The separation is a concern because asbestos fibers are extremely small and light and they can become airborne leading to potential exposure (Van Gosen 2007).

Asbestos fibers are typically less than one micron in diameter and range from several to hundreds of microns in length. The physical characteristics of asbestos include high tensile strength, flexibility, and resistance to heat, electricity, and most chemicals (Van Gosen 2007). The fibers do not evaporate in air, are insoluble in water, and do not migrate through soil. These properties are common to both groups (serpentine and amphibole) and have made them useful to a host of commercial and industrial purposes.

## 3.2 HISTORICAL USE

Gold miners discovered vermiculite near Libby in 1881. The Zonolite Company began mining the vermiculite at the Libby mine in the early 1920s. W.R. Grace purchased the mine in 1963. Due to its insulating properties after exfoliation (the addition of high heat to vermiculite ore would cause the mineral to expand), the Libby vermiculite was primarily used as attic insulation. It was also used as an additive to plaster for insulation purposes; as a feed and paint additive; as a soil amendment (water retention); as fire-proof roofing, wall board, wall paper, pipe and boiler coverings; and as fire-proof insulating packing for walls. Unexfoliated Libby vermiculite was readily available from free piles located in Libby. Therefore, in addition to the aforementioned uses, it was also used locally as bedding around water and sewer lines, as ore rock bedding under the Libby high school track, and in many other unique situations requiring soil fill.

Vermiculite was mined at the Rainy Creek mine using blasting and drag-lines. The ore was processed on site at the Rainy Creek mine by a dry mill until 1985, when the process was changed to wet milling. All ore processing operations at the mine ceased in 1990. The processed ore was transported by truck down Rainy Creek Road to a screening plant at the confluence of Rainy Creek and the Kootenai River. The screening plant sorted the ore by size and loaded the ore for transportation by railroad or truck to processing facilities in Libby and around the nation (Camp, Dresser, and McKee [CDM] 2009).

The ore that was shipped to Libby was exfoliated in two facilities; one near the lumber mill which was demolished in 1950, and the other in downtown Libby, which packaged and shipped the milled ore until 1990. It is estimated that W.R. Grace shipped nearly five million tons of vermiculite from the Libby mine to processing facilities across the nation, most of which was sold in a variety of commercial products within the United States (CDM 2009a).

## 3.3 HEALTH EFFECTS

Asbestos exposure can cause a number of health problems in both humans and animals. Among these health problems are asbestosis, mesothelioma, cancer, pleural plaques, diffuse pleural thickening, and asbestos warts. Asbestosis is the scarring of lung tissue that results from inhaling asbestos fibers. Mesothelioma is a form of cancer of the chest cavity lining that can be caused by inhalation of asbestos fibers. Cancer of the esophagus, stomach, colon, and pancreas can be caused by ingestion of asbestos.

Pleural plaques and diffuse pleural thickening can also result from inhalation of the fibers. Asbestos warts can be caused by skin exposure to asbestos.

Studies performed in the early 1980s by researchers from McGill University and the Centers for Disease Control and Prevention (CDC) National Institute for Occupational Safety and Health (NIOSH) found that former employees of the Libby vermiculite mine had significantly increased pulmonary morbidity and mortality from asbestosis and lung malignancies. Researchers at NIOSH who studied the annual chest x-rays of mine and mill workers with at least 5 years at the mine (between 1975 and 1982) also found an increased prevalence of the radiographic abnormalities associated with asbestos-related disease. The overall mortality rate among the group for mesothelioma was extremely high (4.2 percent) and was similar to that seen for crocidolite miners in South Africa (4.7 percent) and Australia (3.9 percent). Crocidolite is considered by many to be the most toxic form of asbestos. For comparison, the age-adjusted incidence of mesothelioma in the United States (1992 through 2002) was about 0.001 percent (1 case per 100,000) with the occurrence of cases being extremely rare prior to age 50 (Tetra Tech 2007).

More recent studies completed at the Libby Asbestos Superfund Site have also found increased mortality and morbidity among former workers, as well as others in the community without any direct occupational exposures to the mine or processing activities. A mortality study conducted by investigators from the CDC Agency for Toxic Substances and Disease Registry found markedly elevated death rates of asbestosis, lung cancer, and mesothelioma for the Libby community for the 20-year period examined (1979 through 1998). Mortality from asbestosis was approximately 40 times higher than the rest of Montana and 60 times higher than the rest of the United States (Tetra Tech 2007). Large-scale medical screening of over 7,300 individuals who worked or lived in Libby for at least six months prior to 1990 found significantly increased rates of asbestos-related radiologic abnormalities. The prevalence of pleural abnormalities increased with increasing exposure pathways, ranging from 6.7 percent for those who were not able to identify any specific exposure pathways aside from living in Libby to 34.6 percent for those who reported 12 or more specific exposure pathways. The majority of individuals (greater than 70 percent) with pleural abnormalities did not directly work for the mine or processing operations or with any secondary contractors for the mine (Tetra Tech 2007).

The needlelike structure of the amphibole asbestos fibers is particularly harmful to lung tissue. The fibers typically cause damage to the pleural lining of the lungs; this is the lining surrounding the lungs that expand and contracts during breathing. One of the most common asbestos related diseases for persons that have been exposed to LA is pleural fibrosis, which is scarring of the pleural lining. The scale of pleural fibrosis ranges from localized pleural plaques to diffuse plural thickening, but all types of pleural fibrosis reduce the flexibility of the pleural lining and can impair breathing function. Symptoms of pleural fibrosis can include shortness of breath, a chronic cough, phlegm, and/or chest pain (Center for Asbestos Related Disease [CARD] 2010). The fibers can also migrate into the mesothelium, which is the thin, flexible lining of the lung cavity. The fibers cause scarring in the mesothelium and can result in a type of cancer called mesothelioma. Scarring from asbestos fibers in the lung tissue itself is called asbestosis. This is an irritation of the lung cells by asbestos fibers, to the point where scar-like tissue grows in the lungs. The scar tissue is thicker and does not allow for optimum expansion and contraction during breathing. It also compromises blood flow to the lungs and forces the heart to pump harder, often causing enlarging of the heart (CARD 2010).

In February of 2007, the EPA approved funding for the Libby Action Plan, which contains a number of studies of analytical issues, epidemiology, and toxicology related to LA (EPA 2010). In general, these studies examine the issue of LA toxicity; until this toxicity has been determined, the risks associated with exposure to LA cannot be fully assessed.

## 3.4 EPA REMOVAL ACTION LEVELS

EPA established removal action levels and clearance criteria for the Libby Asbestos Superfund Site based on screening-level risk calculations developed to help characterize the relationship between asbestos levels in site media and the resultant level of health risk to residents (EPA 2003). These removal action levels allowed investigators to determine whether a removal action was warranted. The EPA clearance criteria were used to determine when a removal action is sufficiently complete (EPA 2003). EPA has conducted interim removal actions at OU4 (Libby) and OU7 since 2002. The EPA will issue a FS, Proposed Plan, and ROD for all OUs that will set forth final action levels and cleanup standards for remedial actions. Until this time, properties are evaluated in accordance with the removal action levels and clearance criteria.

The general removal approach by the EPA for the Libby Asbestos Superfund Site has been to identify and address sources of LA. Sources of LA can contaminate indoor air when disturbed, and hence, removal or isolation of the source greatly reduces the potential exposure from one of the most significant pathways. The need for removal has been assessed by determining source contamination in one or more of the following areas: 1) attic or interior walls (attics/walls), 2) indoor living space (interiors), or 3) outdoor soils (soils). To do so, the action levels listed below were used to determine if a removal action for the contaminated portion of the property in OU4 was warranted (EPA 2003):

Attics/Walls

- Visual confirmation of open, non-contained, or migrating vermiculite insulation

Interiors

- Visual confirmation of vermiculite in the indoor living space
- Concentration of LA in an indoor dust sample greater than 5,000 LA structures per square centimeter (s/cm$^2$) using Asbestos Hazard Emergency Response Act (AHERA) counting methods

Soils

- Visual confirmation of vermiculite or other vermiculite mine related materials in specific use areas (SUA). An SUA is defined as a garden, former garden, planter, or other defined area of a yard likely to receive significant use and generally not covered with grass
- Concentration of LA in an SUA or other yard soils by any analytical method greater than or equal to 1 percent LA

  The polarized light microscopy-visual estimation (PLM-VE) analytical results for soil samples are split into Bin categories:

  Bin A = Non-detect

  Bin B1 = Trace LA (less than 0.2 percent)

  Bin B2 = Between 0.2 percent and 1 percent

  Bin C = LA greater than or equal to 1 percent by weight (EPA action level)

A detailed discussion of TAPE results and EPA removal action levels is provided in Section 10.2.

# 4.0 TROY ASBESTOS PROPERTY EVALUATION AND REMOVAL DESIGN INVESTIGATIONS

This section describes the objectives, methods, and procedures utilized during TAPEs and RDIs, pursuant to the *Final TAPE Work Plan (Field Sampling Plan and Quality Assurance Project Plan) for the Troy OU7 of the Libby Asbestos Superfund Site* (Tetra Tech 2007) and the *Removal Design Investigation SAP for OU7 of the Libby Asbestos Site* (Tetra Tech 2010a), and references the documents as necessary for greater detail. The TAPE process was initiated in 2007 and was completed in 2013. This RI report provides details for all TAPEs conducted during the project.

## 4.1 TAPE OBJECTIVES

Previous investigations in Libby, Montana, revealed that LA was present in multiple environmental media including indoor air, outdoor ambient air, attic insulation, and soil. Because of the proximity of Libby and Troy and the historical connection the towns share, it was determined that the magnitude of LA and potential exposure of Troy residents to this were unknown, and that additional data would be needed to determine the nature and extent of LA to support removal action decisions. In response to this decision, the TAPE data acquisition process was formulated, and TAPE data quality objectives (DQO) were established to ensure acquisition of adequate data under appropriate conditions.

Two primary objectives of the TAPEs were (1) to determine eligibility of properties for removal actions and (2) to obtain sufficient information to evaluate the properties if the eligibility criteria should change. These criteria are described in Section 3.4 and in the *Draft Final Libby Asbestos Site Residential/Commercial Cleanup Action Level and Clearance Criteria Technical Memorandum* (EPA 2003), which is included as Attachment 1 of this report.

Figure 4-1 depicts all properties that have undergone a TAPE inspection since project inception.

## 4.2 FIELD INVESTIGATION METHODS

The following sections describe the TAPE and remedial design field investigation methods, including logistics and site access; community engagement; property; roads; and alley inspection procedures; quality control; field documentation; decontamination and investigation-derived waste (IDW) handling; record keeping; and sample management.

### 4.2.1 Logistics and Site Access

Logistics

The TAPE investigation focused on residential, commercial, and rural properties within OU7. Road and alley parcels were also inspected as part of the TAPE. Because the work relied heavily upon owner access to each property, Tetra Tech provided public outreach support (including facilitation support at public meetings, preparation of fact sheets, newspaper articles, and notices, and radio announcements) during implementation of the TAPE. In addition, a public information office, the DEQ Troy Information Center, was established in 2007 to assist OU7 parcel owners with all aspects of the TAPE process. It was centrally located at 303 N. Third Street in downtown Troy. The DEQ Troy Information Center also served as the Tetra Tech field office and was the TAPE logistical center for obtaining property access agreements, scheduling field activities, returning samples and field forms at the end of the day, and transferring sample custody from Tetra Tech to the appropriate entity.

Site Access

Prior to initiating the TAPE investigations, a digital property ownership layer was generated by digitizing property boundary lines in ArcGIS from scanned hard copies of ownership maps obtained from Lincoln County. Once each parcel in OU7 had been digitized, the parcels were then combined with the tabular ownership data from the Montana Department of Revenue's Computer Assisted Mass Appraisal (CAMA) tax database for Lincoln County. This resulted in a parcel geodatabase containing information on each property, such as the physical address, legal description, acreage, owner name and mailing address, and geographic coordinates. A unique two-letter, six-digit address number (AD number) was assigned to each parcel for tracking purposes. The unique AD numbers begin at 200001 (AD-200001) and went up; this was done so that the OU7 numbers would not overlap with any numbers used in OU4. The parcel geodatabase was imported into the TAPE Scribe Database so that TAPE inspection data could be tied to each parcel. This would allow for the ease of finding parcel-specific data for future property transactions. The parcel geodatabase was used to generate the mailing list for property access agreements. A more detailed discussion of parcel data and its management can be found in Section 4.4.

Approximately one month before the TAPE field activities began in 2007, access agreements were mailed to every property owner within the boundaries of OU7. A cover letter accompanied the access agreement and contained information on the proposed sampling effort as well as the contact

information for the DEQ Troy Information Center, EPA, Libby Information Center, and the main DEQ office in Helena, Montana. The cover letter explained the need for the signed access agreement and encouraged any property owners who had questions or concerns about the process to contact the designated parties. A separate access agreement was required for each parcel of land. This process was repeated in preparation for the 2008 and 2009 field seasons, with the exception that parcels that had already been inspected were not included in the access agreement mailing.

Once a signed access agreement had been received, the CIC contacted the property owner to schedule a TAPE inspection. For parcels where no response was received, a Tetra Tech field team person followed up by either calling or visiting the property in person to explain the purpose of the TAPE, describe the inspection and sampling process, and answer questions. Tetra Tech provided DEQ with a list of all properties where the property owners were non-responsive or unwilling to sign an access agreement despite attempts to contact them through reasonable means (telephone, visit to the property, and repeated mailings) at the conclusion of the yearly TAPE field activities.

Bulk access agreement mailings were not performed after the 2009 field season because the majority of properties within OU7 had undergone a TAPE inspection between 2007 and 2009, and given the publicity of TAPE inspections during that time, it was reasoned that property owners would request an inspection in subsequent years if they were interested.

#### 4.2.2 Community Engagement

The community relations program for OU7 was developed prior to the start of the initial field season in 2007 and continued through the duration of the project. Procedures and activities in support of community outreach for OU7 are summarized below.

The DEQ Troy Information Center office was staffed with a full-time CIC that was responsible for public outreach (including public meeting announcements, fact sheets, newspaper articles and notices, radio announcements, and mailings) as well as daily interfacing with the public.

Prior to each field season, the DEQ mailed a public meeting announcement postcard to individual property owners, citizens groups, and city and county officials to announce the date and the content of the meeting. The meetings offered the public a summary of the field activities that would take place that summer, as well as the opportunity to question both the EPA and the DEQ about activities being

conducted in OU7. Property owners were also sent property reports summarizing inspection results at their parcel(s). In addition, as new information regarding properties or events occurred in OU7, the DEQ sent the additional public informational mailings by letter or postcard.

The DEQ and the EPA also notified the public in OU7 regarding scheduled meetings in Libby through local newspapers and radio announcements. Meetings included the Libby Asbestos Superfund Site Technical Advisory Group and Community Advisory Group regularly scheduled meetings, as well as other EPA and city/county meetings.

### 4.2.3 Property Inspection, Sample Collection, and Data Recording Procedures

This section describes inspection, sample collection, and data recording procedures used for interiors of buildings (Section 4.2.3.1), and exteriors of properties (Section 4.2.3.2).

#### *4.2.3.1 Interior Inspection and Sampling*

Each land parcel within OU7 was assigned an AD number for tracking purposes. At the property inspected, the TAPE field team recorded all field data using the assigned AD number. The TAPE investigation commenced with a verbal interview with the property owner or resident that addressed all primary and secondary buildings and exterior use areas (UA) on the property. The purpose of the interview was to acquire background information on the property and was conducted by asking a specific set of questions provided on the forms loaded into the personal digital assistant (PDA). Interview topics included whether the property owner or resident was aware of any known or suspected vermiculite insulation or other LA-containing building materials in the house or outbuildings and possible introduction of other sources of LA within or near the property (including garden and landscaped areas and neighboring properties). The verbal interview addressed all primary and secondary buildings, exterior use areas located on the property, and verification of property boundaries. Each building on the property was assigned a unique building (BD) identification number (BD-2XXXXX) for tracking purposes.

<u>Visual Inspection</u>

The attic of each building (if present, safe, and reasonably accessible) was accessed and inspected by the field team for the presence of vermiculite insulation and/or other visible vermiculite (VV). The field team also inspected additional areas where vermiculite insulation may have been exposed in living

spaces (e.g. crawlspaces, closets, wall openings). The visual inspection included checking under other types of insulation (such as blown-in or fiberglass insulation) for vermiculite-containing insulation (VCI). Wall openings were inspected only if the cavity was open, sharing a common air space with the living spaces, and visible to the field team while inspecting interior living spaces. Visual inspections did not include inspecting the roof. The field teams did not inspect properties for non- vermiculite insulation and non-LA. However, damaged or friable suspect asbestos-containing materials observed during the inspection were noted in the PDAs and the locations were identified in the field logbook. If new or existing damage was present in the home and could have resulted in the exposure of the residents to vermiculite, then the field team may have, after consultation with or assistance from the property owner, installed temporary barriers (caulking cracks, taping over openings) to prevent additional vermiculite from entering the living space. Any temporary barrier installed by the field team was detailed in the logbook. Habitable buildings with more extensive damage and greater potential exposure were addressed in a separate response effort, referred to as an Environmental Resource Specialist (ERS) response. ERS activities are summarized in Section 10.1. All visual inspection findings were documented in the PDA, with additional pertinent information recorded in the field logbook and on the field sketch.

Dust Sampling

During the 2007 and 2008 field seasons, dust samples were collected from primary buildings and secondary buildings with finished floors (i.e. material other than dirt). The collection of dust samples was ceased after the 2008 field season because EPA and DEQ agreed that dust sampling analytical results did not contribute sufficient information from which removal decisions could be made. The cessation of dust sampling required a modification to the TAPE work plan (Tetra Tech 2007); this and other deviations from the work plan and the rationale behind them are discussed in Section 4.7.

Dust samples were collected from every level in every primary and secondary building (without a dirt floor) regardless of whether vermiculite insulation or other LA-containing building materials were observed. The samples were collected using a microvacuum (microvac) sampling technique. The dust samples were collected following the procedures detailed in EPA-LIBBY-2012-10 (Appendix A) with TAPE-specific modifications detailed below.

Each indoor dust sample was composed of a 10-point composite sample to improve representativeness of the dust sample for each level of the building. The TAPE field team selected sample aliquot locations based on the team's visual inspection of the building and estimation of where contaminated dust was most likely to be found.

The microvac pumps were calibrated prior to, and following sample collection, using a secondary standard rotometer. The sampling area for each dust sample point (aliquot) consisted of 100 square centimeters ($cm^2$) and was delineated using a fixed template provided with the sampling cassettes. A specific sampling pattern within the template was established and followed for each aliquot collected. Field teams used stopwatches to record both the 30-second aliquot intervals and the total composite sample time. The cassette contained dust from a total 1,000 $cm^2$ surface area and a total of approximately 5 minutes of sampling time.

Once the dust sample was collected, a pre-printed label with the sample identification number was attached to the cassette. The sample identification number consisted of a unique number "TT-XXXXX" with "TT" indicating that it was a "Troy TAPE" sample and "XXXXX" being a unique five-digit number.

Soil Sampling

Soil samples were collected from all accessible soil surfaces within the interior of buildings, such as garages, pump houses, sheds, and crawlspaces. Soil was sampled from interior surfaces regardless of the results of the visual inspection. TAPE interior surface soil samples were collected as 30-point composites with each aliquot being collected from zero to three inches in depth. Any variations to this sampling protocol are documented in the logbook and are limited to factors such as limited volume or square footage, limited access to the interior area, or inability to sample at the specified depth. Interior soil samples were placed in a plastic bag and a pre-printed sample identification label (TT number) was adhered to the exterior of the bag. The sample was then placed into another plastic bag labeled with the TT number. Soil sampling protocols followed Standard Operating Procedure (SOP) CDM-Libby-05.

#### *4.2.3.2 Exterior Inspection and Sampling*

Visual Inspection

All areas of OU7 properties not covered with buildings were inspected for vermiculite. These exterior areas were grouped into four general use area (UA) types: (1) specific use area (SUA) (e.g., gardens,

flowerbeds, etc.), (2) common use area (CUA) (e.g., yard), (3) limited use area (LUA) (e.g., field), and (4) non-use area (NUA) (wooded area). A unique UA identification number (UA-2XXXXX) was assigned to each UA that was delineated and inspected. For the TAPE, visual point inspections generally correlated with soil sample aliquot locations. A minimum of five and a maximum of 30 visual point inspections were performed per UA.

Soil Sampling

After the visual inspection of the property was conducted, the TAPE field team collected a 30-point composite soil sample from each exterior UA. Each composite was obtained from either a depth of 0 to 3 inches for CUAs and LUAs or from a depth of 0 to 6 inches for SUAs. No soil samples were collected from areas with paved surfaces such as parking lots. Each aliquot was examined for the presence of VV. The amount of VV was categorized as none, low, intermediate, or high, and described as expanded or unexpanded. Once all of the soil aliquots had been obtained and examined for VV, the sample was placed in a plastic bag and labeled with the corresponding sample identification number (TT number).

### 4.2.4 Road and Alley Inspection, Sample Collection, and Data Recording Procedures

The potential exists for vermiculite to migrate from properties where it was used as attic insulation or as a soil amendment to other parcels. This could occur as a result of erosion and transport from contaminated yards and gardens or from accidental spills during transport from Libby to Troy. Also, it is possible that waste or borrow material containing Libby vermiculite was used in the construction of OU7 roads. Because most roads and alleys are on public land with few access restrictions, there may be exposure to LA from contact with road and alley soils. To address this concern, OU7 road and alley parcels were added to the TAPE inspection process in 2009. The following paragraphs outline the general procedures for road and alley inspections; for more complete procedural information, please refer to the *Protocol Modification to the TAPE Work Plan, Inspection and Sampling of Roads and Alleys for Visible Vermiculite* (Tetra Tech 2009b). Figure 4-2 depicts the road and alley parcels.

Parcel Identification and Classification

Prior to inspection and soil sampling, Tetra Tech identified all road and alley parcels within OU7 and assigned a unique AD number to each. The parcels were then assigned a “classification” based on the type of road or alley (such as paved, unpaved, and paved with no curb and gutter, or driveway) and

location (within or outside of city limits). Individual parcel maps were prepared for each road and alley parcel and were included in the AD file for use by field staff during the inspections and sampling.

Visual Inspection and Soil Sampling

Tetra Tech first inspected each road and alley parcel for the presence of VV. Similar to the exterior use area procedure, the amount of VV was quantified as none, low, intermediate, or high and whether or not it was expanded or unexpanded. The field crew recorded the presence or absence of VV either on the surface or in the soil aliquots, regardless of the road or alley surface material (asphalt, concrete, gravel, dirt) in the PDA and in the corresponding logbook. After completing the visual inspection of each road or alley, the field team then collected soil samples following the procedures described the Final TAPE work plan (Tetra Tech 2007). One soil sample was collected from 0 to 6 inches below ground surface from each road and alley parcel not fully covered by asphalt or concrete. Samples consisted of 30-point composites; the aliquots were collected at evenly spaced intervals alternating from left, to center, to right on dirt/gravel surfaced roads or alleys. Where road surfaces were paved but road edges were not, a 30-point composite sample was collected from evenly spaced locations along the road edge/borrow ditch. Each aliquot was inspected for VV. All aliquot locations were recorded on the field sketch. Once all aliquots had been obtained, the sample was mixed, placed in a plastic bag and labeled with a sample identification number.

### 4.2.5 Quality Control Samples

Field QC samples were collected as part of the TAPE investigation to assess satisfaction of data quality objectives. The basis for the QC sample scheme (type, frequency, etc.) is outlined in the *Site-wide Quality Assurance Reference Document* (U.S. Army Corps of Engineers [USACE] 2013) for the Libby Asbestos Superfund Site. The procedure for the collection of dust field blank samples was modified as necessary for OU7. All field QC samples collected during the TAPE were submitted blind to the laboratory.

Field Blank Dust Samples

Each field team collected one field blank dust sample per day. Each field blank dust sample was archived and one field blank dust sample per week was randomly selected for analysis from the archived samples. Field blank dust samples were collected at locations selected by the TAPE field team. They

were collected by attaching a cassette to the pump and pumping for 30 seconds to one minute at the same rate as for dust sample collection. However, the end of the cassette was exposed to indoor air at the selected sampling location, rather than passed over a surface of any kind. Data for the field blank dust samples were evaluated to assess whether a potential existed for airborne asbestos to cause analytical detections of asbestos in dust, or for cross-contamination to occur during sampling.

Dust Lot Blank Samples

Dust lot blank samples were submitted to the laboratory for each lot of cassettes received from the supplier. One lot blank cassette was submitted from each 50-cassette box prior to field use. Data for dust lot blank samples was used to evaluate whether cartridges were received asbestos-free from the supplier. Tetra Tech did not use a cassette from a given lot until the dust lot blank results confirmed the cartridges were asbestos-free. The Tetra Tech TAPE Field Team Leader was responsible for cassette clearance and usage.

Dust Field Duplicates

Dust field duplicate samples were collected at a frequency of one sample per 20 composite dust samples or a rate of five percent. Dust field duplicate samples were collected by locating a second sampling template adjacent to every original sample aliquot location within the building level. Each duplicate aliquot location was sampled using the dust sampling procedures described above and contained the same number of sample aliquots as the original sample. Data for dust field duplicates were used to evaluate the potential variability in LA concentrations in a building. These data were not used to evaluate precision in sampling or analytical techniques.

Equipment Blanks

Soil field equipment blanks were collected at a rate of one per calendar week of sampling per field team. Field equipment blanks were collected by pouring distilled water over the sampling equipment into a decontaminated stainless steel sampling bowl, pouring the rinse water from the bowl into a sample bottle, placing the sample bottle in a re-closable plastic bag, and submitting it for analysis by EPA Method 100.2 (EPA 1994). Data from field equipment blank samples were used to evaluate whether the decontamination procedures resulted in sampling equipment that was asbestos-free. Soil field

equipment blank samples with elevated results could indicate inadequate equipment decontamination procedures.

Equipment rinsate blanks were not collected after 2011 because limits of detection for LA using Polarized Light Microscopy (PLM) are not low enough to capture concentrations expected in equipment rinsate blanks, and because the frequency of LA detections had been extremely low in rinsate blanks previously collected in OU7.

Soil Field Duplicates

Soil field duplicate samples were collected at a frequency of one sample per 20 composite soil samples or a rate of 5 percent. Field duplicate samples were collected as samples co-located in the same exterior use area (yard or landscaped area, for example) and contained the same number of sample aliquots, but were collected from adjacent aliquot locations. Data for soil field duplicates were used to evaluate the potential variability in LA concentrations in a specific exterior use area. These data were not used to evaluate precision in sampling or analytical techniques.

#### 4.2.6 Field Documentation

The primary tool for field documentation of TAPE inspection information was the Trimble GeoXT PDA. In addition to the PDA, the field teams also recorded information in field logbooks, on property sketches, and with photographs. All TAPE data, including responses from property owner interviews, results of the visual inspection at the property, and sample collection data, were entered into the PDA. This ensured consistency between properties and between TAPE field teams and aided in limiting data entry errors. Use of the PDA also allowed for compilation of TAPE data into the TAPE Scribe Database. A description of Scribe is provided in Section 4.4.3.

The field teams also used field logbooks. Relevant information that was not recorded on the PDA field forms was recorded in logbooks. Each field team maintained a field logbook for recording the date and time of each property inspection, the property ID, building ID, use area ID numbers, the number and type of samples collected at the property including sample ID numbers and any other pertinent information. The field logbook served as an independent (backup) record for all activities conducted and samples collected at a property, in the event that the PDA data were lost or corrupted.

TAPE inspection information was also recorded on individual field sketches. Property maps comprised of aerial photographs were provided to the teams for reference; however, the quality of the photographs did not allow for their use as a base map for each property. The field sketches show the property boundary, building locations, exterior use areas, fences, pavement, large trees, and other major identifiable features of the property. In addition to identifying these items, the field sketch served as an additional means for recording the location of any VV detected during the exterior visual inspection. Requirements for field sketches are discussed in greater detail in the TAPE work plan (Tetra Tech 2007).

Each TAPE field team was provided with a digital camera for photo-documenting the primary characteristics of each parcel. The field teams photographed primary and secondary buildings, attic insulation, any VCI or VV identified on the premises, UAs, and other noteworthy features. The home owner or resident was asked for permission before any photographs were taken. A description of each photograph was recorded in the field logbook.

#### 4.2.7 Decontamination and Investigation-Derived Waste Handling

Decontamination

Dust sampling activities required minimal decontamination since a new cassette and disposable paper template were used for each sample collected. The air pump and the tubing that connects the cassette to the air pump were decontaminated between samples with a damp paper towel to avoid transferring dust from one location to another. The used paper towels were placed in a labeled asbestos waste bag.

Reusable stainless steel scoops, bowls, and augers were used for soil sampling and required decontamination after each soil sample was collected. Decontamination occurred in the location where the soil sample was collected and included spraying the equipment with distilled water and drying it with paper towels. The water was allowed to fall onto the ground surface in the area just sampled and the paper towels were placed in a labeled asbestos waste bag.

Field team members wore nitrile gloves during dust and soil sampling. Any visible soil on hands or clothing was removed by washing with soap and water. Additional personnel decontamination procedures, including requirements for decontamination zones, are outlined in the project health and safety plan (Appendix A of the TAPE work plan [Tetra Tech 2007]).

Investigation-Derived Waste Handling

Investigation-derived waste (IDW) included used wet wipes, wet paper towels, disposable gloves, used respirator cartridges, used plastic tubing, disposable protective outerwear, plastic floor coverings, and other minimal waste. It was possible, but not likely, that these investigation-derived waste materials were contaminated with asbestos. Therefore, all investigation-derived waste was double-bagged in asbestos waste bags, labeled with asbestos labels, and stored in an approved containment area at the Tetra Tech Troy field office (TFO) until it was transferred to an approved landfill (Lincoln County outside of Libby) for final disposal. Non-IDW generated by the TAPE field teams, such as food containers and waste paper, was separately bagged and disposed of as solid waste.

### 4.2.8 Record Keeping and Sample Management

At the end of each field day, the TAPE field teams downloaded the PDA and cameras, cataloged and stored all environmental and QC samples, and turned in the appropriate logbook and paperwork to the Tetra Tech Sample Coordinator. The TAPE Scribe Database is capable of generating chain-of-custody (COC) information. Once inspection data on the PDA had been reviewed for accuracy and imported into Scribe, the Sample Database Coordinator created COC forms for all of the samples collected (water, soil, air). Scribe automatically assigned a number to each COC form so it could be tracked in the database. COC forms were reviewed by the field teams that collected the samples, and then signed, dated, and placed with the samples in a sample storage bin in the TFO/equipment shed. The Tetra Tech Sample Coordinator stored the samples in a secure area until the samples were transferred, and accepted via a signed COC form, to the appropriate laboratories.

The field teams were also responsible for accurately labeling and downloading digital photographs daily to a computer at the TFO/equipment shed.

After each field sample had been collected and labeled with the appropriate TT number, a field team member completed, signed, and placed a custody seal on the cassette (dust) or around the sealed bag (soil). At the end of the field day, the field teams placed their samples into numbered bins in the Tetra Tech TFO/equipment shed and wrote the corresponding number of samples on a specific form for the Troy sample coordinator. The Sample Coordinator then printed a COC form for each bin. The field team confirmed that each COC matched the samples in the numbered bin, signed the COC, and placed it in the corresponding bin to await transfer to the appropriate laboratories.

## 4.3 SAMPLE ANALYSIS

The analytical methods and standard operating procedures (SOP) used for TAPE dust and soil sample analysis are summarized in the sections below. Copies of all project-specific laboratory modifications and SOPs are provided in Appendix A.

### 4.3.1 Dust Sample Analysis

Indoor dust samples were analyzed by transmission electron microscopy (TEM) in accordance with American Society for Testing and Materials (ASTM) Method D5755-03, using modified counting rules (length greater than or equal to 0.5 micrometers [µm]; aspect ratio greater than or equal to 3:1) and sample preparation techniques in accordance with applicable project specific laboratory modifications and SOPs (Appendix A).

### 4.3.2 Soil Sample Analysis

Soil samples were analyzed using PLM-VE. Sample preparation was performed in accordance with project-specific SOP ISSI-Libby-01, *Soil Sample Preparation* (Appendix A) and sample analysis was performed in accordance with project-specific SOP SRC-Libby-03, *Analysis of Asbestos Fibers in Fine Soil by Polarized Light Microscopy* (Appendix A). The coarse fraction of each soil sample was archived instead of being analyzed, because the results were assumed at the time to have little bearing on whether a property was eligible for removal.

## 4.4 DATA MANAGEMENT

A complete description of the data management procedures for the TAPE program can be found in the *Final TAPE Work Plan (Field Sampling Plan and Quality Assurance Project Plan) for the Troy OU7 of the Libby Asbestos Superfund Site* (Tetra Tech 2007) and in the *Data Management Plan Version 3.0* (Tetra Tech 2009c). Additional information on project-wide data management procedures can be found in the EPA Data Management Plan (EPA 2010).

The following sections describe the management of data associated with parcel inspections, sample collection, and sample analyses.

### 4.4.1 Parcel and GPS Data

Parcel Data

A digital parcel database was assembled from hard copy property ownership maps by digitizing the parcel boundaries using ArcGIS software. Unique two-letter, six-digit AD numbers were assigned to each parcel for tracking purposes. The parcel geodatabase was imported into the TAPE Scribe Database, so that inspection data could be tied to each parcel and so that it could be used to generate the address list for access agreement mailings. The parcel geodatabase resulting from the assembly process was completed in January 2007. Since that time, the State of Montana has periodically released updates to the cadastral (geography) and CAMA databases. Tetra Tech processed revisions from both sources before they were incorporated into the parcel geodatabase. Parcels that had been subdivided, combined, or had any other boundary changes were updated and the changes noted in the OU7 parcel geodatabase.

Occasionally, parcels were legally combined or subdivided. For parcels that were legally combined, one or more AD numbers were removed from the parcel geodatabase. When a parcel was subdivided, a new (unique) AD number was assigned, and if any data were associated with the subdivided parcel, it was reassigned to the new AD number as well. See Appendix A in the Troy Data Management Plan Version 3.0 (Tetra Tech 2009c) for a complete explanation of the process used for parcel combinations and subdivisions.

In instances where an adjacent parcel shared a building or major UA with a parcel scheduled for inspection (both owned by the same landowner), although the parcels were listed as separate properties in CAMA, if the two parcels had not been legally combined, the field teams would designate one of the parcels as a reference parcel. Reference parcels are associated with and tracked in the database with the adjacent parcel(s).

Global Positioning Data

In 2007, field teams attempted to collect global positioning system (GPS) geographical coordinates from each exterior use area to demarcate the soil sampling locations. However, due to tree cover and other obstructions it was often difficult, if not impossible, to acquire enough satellites to obtain accurate coordinates. Therefore, in 2008, field teams began only recording only one set of coordinates for each

parcel. The GPS reading was collected approximately 20 feet outside the main entrance to the primary building on the parcel, on a defined UA (yard, driveway, etc.). The information was captured using the field forms in the PDA. The latitude, longitude, and other GPS data are exported from the handheld computer and imported into the TAPE Scribe Database.

### 4.4.2 Field Data

The sections below describe the various field forms and documents related to the TAPE investigation and how documents are managed.

Access Agreements

Signed access agreements were required prior to any field activities on a subject property. Some landowners provided verbal approval and scheduled an inspection over the phone. In these cases, the field team obtained a signature immediately prior to conducting the inspection. The original, signed, access agreements were placed in the file folder for each parcel (by AD number) in the DEQ Troy Information Center. The original access agreements were also scanned into a portable data format (PDF) and included in the electronic data archive for the property.

Field Logbooks

Information pertaining to the field inspection was recorded in field logbooks. Each field team maintained a field logbook for recording the date and time of each property inspection; the names of the people who allowed property access and completed the interview; the property identification and building designations; locations, quantities, and types of visible vermiculite; and the number and type of samples collected at the property, including sample numbers and any other pertinent information.

The field logbook serves as an independent (backup) record for all activities conducted and samples collected at a property in the event that data on the mobile device are lost or corrupted. The field logbooks were scanned into individual PDF files and stored as part of the electronic data archive for each property. The hard copy original logbooks were stored in the DEQ Troy Information Center.

Property Sketches

A property sketch was generated for each property inspected. The sketches depict the locations of primary and secondary buildings and UAs. Property boundaries (as depicted on aerial photographs overlain with property boundaries and verified by the property owner) were also delineated. The property sketch also documents the approximate area of each building and UA, as well as the location(s) of fences, large trees, and other potential obstructions to possible future remedial activities. Property sketches also show the locations where soil samples were collected, as well as the locations of any observed VV. The sketches were prepared on 8.5- by 11-inch graphing paper and were mandatory for each property inspected. Original hard copy property sketches were placed in the file folders in the DEQ Troy Information Center. Property sketches were also scanned into an individual PDF file and stored in the electronic data archive for the parcel.

Point-of-Contact Forms

A point-of-contact (POC) form was required for every occupied property inspected (with the exception of properties that were entirely non-use), regardless of whether it was occupied at the time of inspection by the owner, a tenant, or an employee. Information requested on the POC form included the AD number, the primary BD number, the date completed, names and birthdates of the occupants, telephone numbers, the mailing address, and the physical address. Original POC forms were placed in the project files in the DEQ Troy Information Center. A scanned copy (PDF format) of the POC form was maintained in the electronic data archive for the property.

Additionally, information recorded on the POC forms was manually entered into a temporary POC database ("Troy_POC.mdb", Microsoft Access format). The information in the temporary POC database was reviewed and then imported into the TAPE Scribe Database.

Digital Photographs

The field teams took digital photographs to document certain features of a parcel for future reference and to potentially assist in development of a removal action plan for the property. The property owners were asked for permission before photographs were taken, unless the photographs were taken from public rights-of-way. All photographs were taken using digital cameras and a description of each photograph was recorded in the logbook. The photographs were downloaded at the DEQ Troy

Information Center the same day as the inspection, and saved in a directory of field photographs. The images were retrieved from the directory by the Sample Database Coordinator and placed with the other scanned documents (access agreement, logbook, property sketch, and POC form) for the parcel in the electronic data archive folder.

### 4.4.3 Electronic Data

The following sections describe the management of electronic data, including inspection data entered into the PDA and the transfer of data from the PDA to the TAPE Scribe Database.

Personal Digital Assistant Setup and Modification

Each field team was provided a PDA for data and GPS point acquisition. The geographic software ArcPad was loaded onto each of the PDAs to support parcel GIS layers. In addition, aerial photographs of OU7 were uploaded onto the PDAs so that the parcel database layer could be overlaid on the aerial images. A custom software application was developed named PocketScribe, which uses a Microsoft SQL Server Compact database to store lookup values and inspection results. PocketScribe was pre-populated with basic parcel information from the parcel database, but the remaining data associated with inspections were hand-entered into the PocketScribe field forms on the PDA. Changes were occasionally made to the PocketScribe field forms to make data entry more efficient or to suit the objectives of the project. Suggested changes were discussed with the Field Team Leader and DEQ Project Officer before the Sample Database Coordinator made any changes to PocketScribe and the TAPE Scribe Database. If changes were made, the modified PocketScribe application was loaded onto the PDA and the field teams were trained on the modifications.

Data Collection

TAPE field data were entered into the PocketScribe field forms on the PDA during inspections. Data entered included parcel information (AD number and any reference parcels), information gathered about the property during the interview (age of buildings, historical use, persons living on the premises, past or present, etc.) location and building information (number of primary and secondary buildings, number of attics, building types – sheds, garages, homes, etc.), sample information (number of aliquots, location of samples, etc.), and the presence or absence of VV on the parcel (interior and exterior). Certain screens alerted field team members of missing critical data and would not allow data entry to

move forward until this information was entered. Once all data entry had occurred and the forms were saved, the PDA was turned off and taken to the next parcel or back to the Sample Database Coordinator in the DEQ Troy Information Center for incorporation of the data into the TAPE Scribe Database. If, for some reason, the handheld computer became inoperable in the field, the field teams could collect data using a printout of the field forms. In the event the hard copy field forms were used, the Sample Database Coordinator would enter the data into an operable PDA and import the data into the TAPE Scribe Database. The hard copy field forms were then scanned into a PDF file and added to the electronic archive folder for the parcel. The original hard copy was filed with the other paperwork for the property in the DEQ Troy Information Office.

Data Upload to TAPE Scribe Database

Scribe is a database application developed by the EPA's Environmental Response Team to manage environmental data. It is capable of capturing sampling, observational, and monitoring data, as well as importing electronic data such as analytical laboratory results and sampling location data like GPS coordinates.

The TAPE Scribe Database contains parcel, field, and analytical data from the TAPE. During the field season, the Sample Database Coordinator was responsible for importing data from the handheld computer into the TAPE Scribe Database at the end of each field day. After completion of a parcel inspection, the field team gave the handheld computer to the Sample Database Coordinator, who reviewed the data to ensure that the correct AD number was used and that BD, UA, and TT number entries in the logbook correlate with the property sketch and POC form. Field data from the handheld computers were exported as an extensible markup language file and then imported into a desktop Access database where the Sample Database Coordinator verified them. After verification, the data were imported into the TAPE Scribe Database where they were processed and distributed to users.

Multiple users can simultaneously view the data in the TAPE Scribe Database. Scribe.NET is a method of storing and sharing Scribe projects between various desktop clients. Scribe projects are "published" from the Scribe desktop client so that other users can "subscribe" to the published projects. The Tetra Tech Scribe Database Administrator was responsible for managing the importation of TAPE data into Scribe and for constructing queries that are used for data evaluation and presentation.

Troy Owner Access Database

The Troy Owner Access Database (TOAD) is a Microsoft Access database used by the CIC and other office staff to track landowner information, communication with landowners or other agencies regarding the parcel, and inspection scheduling information. For each mailing that was sent to a landowner, the mailing category and date were tracked in TOAD. The TAPE Scribe Database works in conjunction with TOAD to provide the most up-to-date landowner contact information for each parcel. In addition, the Scribe parcel visit data are depicted in TOAD so the user can quickly view the number and types of visits to the parcel. Custom reports show detailed communications or can summarize data by various parameters. Specific parcel information includes physical address, owner names and contact information, and other important parcel features such as parcel status (granted, limited, or denied).

### 4.4.4 Analytical Data

Analytical data were delivered to Tetra Tech in electronic and PDF format from the laboratories responsible for analyzing TAPE dust, soil, and health and safety-related air samples. Tetra Tech data verification and management personnel were copied on the messages and an assessment of data quality for each deliverable was subsequently initiated. Section 4.5 below describes the data quality assessment process. Each electronic data deliverable (EDD) and associated data package was filed on the Tetra Tech server in Helena, Montana, and serves as a backup copy.

## 4.5 DATA QUALITY ASSESSMENT

Tetra Tech assessed the quality of both field and laboratory data generated during the TAPE project to ascertain whether the data satisfy the project DQOs specified in the TAPE work plan (Tetra Tech 2007). The field data verification process is summarized in Section 4.5.1, and the laboratory data verification process is summarized in Section 4.5.2. A statement of data quality is provided in Section 4.5.3.

### 4.5.1 Field Data Verification

Tetra Tech verified the accuracy and completeness of TAPE field documentation in accordance with the procedures outlined in the data management plan (DMP) (Tetra Tech 2009c).

### 4.5.2 Analytical Data Verification

The TAPE analytical data were received from the laboratory in two formats: (1) data package (i.e., sample receipt documentation and bench sheets) in PDF format and (2) EDDs in Excel spreadsheet format. Once the analytical results were received from the laboratory, the electronic data were imported into the TAPE Scribe Database. This allowed the field data to be linked to analytical data. The electronic data subsequently underwent a three-step verification process to identify and correct any inconsistencies between the laboratory bench sheets, EDDs, and the final TAPE Scribe Database. A description of this data verification process can be found in the DMP (Tetra Tech 2009c). The analytical data were also verified to ascertain whether laboratory procedures were consistent with analytical method requirements and were consistent between laboratories and analysts, in accordance with project-specific data verification SOPs (Appendix A).

Dust Analytical Data Verification

Tetra Tech conducted data review and data entry verification of the dust sample analytical data in accordance with project-specific SOP EPA-LIBBY-09. The SOP describes a standardized method for review of raw TEM data and verification of TEM data entry into the project database. Libby-specific procedures described in this SOP were modified, as needed, for the OU7 TAPE project. A copy of this SOP is provided in Appendix A.

Tetra Tech took corrective action for transfer errors by summarizing apparent inconsistencies, omissions, or other suspected errors and providing them to the laboratory liaison. The laboratory determined which items were authentic errors that required correction and corrected the EDD and/or bench sheets as needed. The laboratory liaison forwarded the list of errors to the appropriate laboratories for response. The revised bench sheets were submitted to the laboratory liaison and forwarded to Tetra Tech. Tetra Tech downloaded the revised documents, reviewed them, and replaced the previous ones as appropriate.

Soil Analytical Data Verification

Tetra Tech conducted data review and data entry verification of the soil sample analytical data in accordance with project-specific SOP EPA-Libby-10. This SOP describes a standardized method for review of raw polarized light microscopy (PLM) data and verification of PLM data entry into the project

database. Libby-specific procedures described in this SOP were modified, as needed, for OU7. A copy of this SOP is provided in Appendix A.

Tetra Tech completed a verification worksheet (Excel format) and recorded any inconsistencies, errors, and omissions discovered during the review process. The validation worksheet was provided to the laboratory liaison overseeing the contracted laboratory. The laboratory liaison notified the laboratory of errors and inconsistencies. The laboratory subsequently made any necessary changes to the deliverables. Upon receipt of the corrected EDDs and bench sheets, the data were replaced in the TAPE Scribe Database and verified as discussed above.

#### 4.5.3 Statement on Data Quality

One of the primary objectives of the TAPE was to identify parcels in OU7 that met the removal criteria specified by the EPA (EPA 2003) (Attachment 1). This objective was met in all field seasons through visual inspections for vermiculite and collection and analysis of environmental samples. These samples were analyzed at analytical sensitivities sufficient to identify LA above the applicable EPA removal action levels (Attachment 1). In addition, TAPE field and analytical data were verified to ensure accuracy and reliability. As a result, TAPE data met the TAPE DQOs and were sufficient to determine whether or not inspected properties required additional investigation through the RDI process and/or subsequent removal action, if necessary.

DQOs were developed to identify the data quality and quantity needed to support project objectives, and to assess whether the data collected were of adequate quality and quantity to satisfy project needs. DQOs for the TAPE project were presented in the TAPE work plan (Tetra Tech 2007).

The objective of the TAPE was to identify those parcels in OU7 that meet the removal criteria identified in EPA's *Action Level and Clearance Criteria Technical Memorandum for the Libby Asbestos Site Residential/Commercial Cleanup* document (Attachment 1). Three questions and associated decision rules were established for the project:

1) **Did VCI and/or VV need to be removed from properties in OU7?** If uncontained, migrating VCI or VV was observed in indoor living spaces or working spaces, or uncontained VCI or VV was observed in attics, the property required a removal action to remove the VCI and/or VV.

2) **Did specific properties in OU7 contain levels of LA in soil that needed to be addressed by short-term removal actions?** If VV was observed in an SUA, a short-term removal action was necessary to

remove it. If the average concentration of LA in soil in any UA exceeded the EPA action level for soil, any detectable LA in soil on the property should be removed in a short-term removal action.

**3) Did specific properties in OU7 contain levels of LA in dust that needed to be addressed by short-term removal actions?** If the average concentration of LA in dust over an interior building level (floor) exceeded the EPA action level for indoor dust, the entire building level from which the dust sample was collected was subject to a removal action.

#### *4.5.3.1 Sampling Design*

To address the first question, interior inspections for VCI and VV were performed in each building at each property inspected in OU7.

To address the second question, a soil sampling program was implemented. A composite sampling design was used in order to measure the average concentration of LA in each UA. One composite sample consisting of 30 subsamples was collected from each UA. The sampling depth depended on the type of UA. Samples were collected from 0 to 3 inches below ground surface (bgs) in CUAs and LUAs and from 0 to 6 inches bgs in SUAs (to account for anticipated exposure to deeper soil horizons [e.g., during gardening]).

To address the third question, an interior dust sampling program was implemented. A composite sampling design was used in order to measure the average concentration for each building level (floor). One composite sample consisting of 10 subsamples was collected from each floor. A specific number of subsamples was collected from each of three designated sub-area types, as applicable to the building: accessible areas (four subsamples), infrequently accessed areas (four subsamples), and inaccessible areas (two subsamples).

#### *4.5.3.2 Limits on Decision Errors*

The TAPE established a limit on decision errors of no more than a 10 percent chance of not initiating a removal action when one was required (Tetra Tech 2007). Applicable removal criteria, as identified in the *Action Level and Clearance Criteria Technical Memorandum for the Libby Asbestos Site Residential/Commercial Cleanup* (Attachment 1) are outlined in Section 3.4 above. Discussions regarding whether the limit on decision errors was achieved are presented below, organized by the three primary types of data collection for the TAPE - visual inspections, dust samples, and soil samples.

**Visual Inspections.** If no VV or VCI was observed during a visual inspection of a property, there should be no more than a 10 percent chance that VV or VCI was, in fact, present on the property. Although achievement of this limit on decision errors has not been verified quantitatively, a number of efforts were made to ensure that identification of VV was as accurate as possible.

At the beginning of the 2008 field season, Tetra Tech conducted VV re-verifications at 101 parcels for the identification and possible quantification of VV in exterior soils. A total of 527 UAs initially inspected in 2007, were re-inspected to confirm, semi-quantify, and describe VV in exterior soils. Detailed information about the parcel/UA selection process is provided in TFO-00008 (Appendix B). In addition, at the start of the 2008 field season, several Tetra Tech field personnel were trained by experienced CDM field personnel for approximately four days on the recognition of VV in exterior soils. Additionally, pursuant to TFO-00008, CDM conducted audits of Tetra Tech's VV re-inspection process. The results of the audits demonstrated that procedures being used in OU7 were adequate for identifying VV.

**Soil.** If the measured average concentration of LA in soil in a UA was less than the action level for soil, there should be no more than a 10 percent chance that the true average exceeded the action level. If the concentrations of a contaminant could be measured accurately in the individual samples as well as in their composite, and if the compositing process is carried out properly, then the concentration measured in the composite sample was expected to be equal to the average of the concentrations measured in the individual samples (assuming no measurement errors). Although achievement of this limit has not been verified quantitatively, an EPA study of the accuracy of the soil analytical method found that 77 percent of the reference soil samples were assigned to the appropriate bin, and of those not accurately assigned, 96 percent were assigned to a higher-concentration bin than the reference concentration (Tetra Tech 2007). Based on these results, the method appeared to overestimate rather than underestimate the true concentration of LA in soil, so the risk of false negative decisions was presumed to be low. In addition, both field and laboratory duplicate samples were included in the TAPE analytical program to assess sampling and analytical precision, thus minimizing the chance for false negative errors (i.e., not initiating a removal action when one was required).

**Dust**. For dust, if the measured average concentration of LA in dust over an interior building level (floor) was less than the action level for indoor dust, there should be no more than a 10 percent chance that the true average exceeded the action level. To assess the variability of the composite samples, duplicate samples were collected and analyzed from randomly selected properties within OU7. However, interior

dust sample collection was discontinued after the 2008 field season because the dust sample data did not provide sufficiently useful information for removal-action decision making (i.e., very few analytical results exceeded the removal criterion of 5,000 LA s/cm$^2$). Supporting documentation for this modification is provided in TFO-00012 (Appendix B). Since the dust sample collection program was discontinued, sampling and analytical precision (based on field duplicate pair results) was not determined.

#### *4.5.3.3 Analytical Sensitivity*

OU7 dust and soil sample analytical sensitivities were commensurate with those for OU4, which are based on the removal criteria outlined in EPA's *Action Level and Clearance Criteria Technical Memorandum for the Libby Asbestos Site Residential/Commercial Cleanup* (Attachment 1). The removal criteria are 1 percent LA for soil and 5,000 s/cm$^2$ (using AHERA counting rules) for dust. The dust and soil analytical sensitivities achieved for the TAPE samples were low enough to detect LA at levels below these removal criteria.

#### *4.5.3.4 Summary*

In summary, the objective of the TAPE was to identify those parcels in Troy that met the removal criteria identified in EPA's *Action Level and Clearance Criteria Technical Memorandum for the Libby Asbestos Site Residential/Commercial Cleanup* document (Attachment 1). This objective was met through (1) visual inspections for VCI and VV; (2) the collection and analysis of interior dust samples; and (3) the collection and analysis of interior and exterior soil samples. These samples were analyzed at analytical sensitivities sufficient to identify LA at the EPA removal action levels.

## 4.6 REMOVAL DESIGN INVESTIGATIONS

RDIs were conducted at properties where the results of a TAPE inspection revealed that the parcel met one or more EPA removal action criterion. The primary objective of an RDI was to obtain the additional data necessary to design removal activities at properties identified for a removal action. RDI field methods are briefly described below; a detailed description of the RDI process can be found in the RDI SAP for OU7 (RDI SAP) (Tetra Tech 2010a).

Tetra Tech conducted the exterior portion of the RDI inspections, and a contractor to the USACE performed the interior portion of the RDI inspections.

For the exterior RDI process, Tetra Tech reviewed field and analytical inspection data before providing results of the RDI to the USACE contractor responsible for preparing design drawings and performing removal activities. Once design drawings were prepared, Tetra Tech again reviewed the RDI results to ensure the removal was based on accurate information. Removal activities were then performed by the USACE contractor.

For the interior RDI process, Tetra Tech provided the USACE removal contractor with a summary document outlining TAPE findings and listing known vermiculite in buildings, attics, crawl spaces, living spaces, and outbuildings. Additionally, the removal contractor received the property access agreements, removal registry agreement, TAPE field sketches, photos, logbook notes, and property reports.

The removal contractor was also given a building drawing provided by another USACE contractor, which outlined the interior removal scope of work. The removal contractor's supervisor completed a building walkthrough inspection before initiation of the project to verify accuracy of the TAPE findings. An additional interior inspection was completed by the removal contractor's supervisor during the removal process to document any containment breaches or VCI leaking from the removal areas. The removal contractor's supervisor completed a final walkthrough of the building to document completion of the removal process.

## 4.7 DEVIATIONS FROM THE TAPE WORK PLAN

Procedures were continually monitored to ensure that the objectives of the TAPE work plan (Tetra Tech 2007) were accomplished. Modifications to the procedures described in this document were occasionally necessary to fulfill project objectives or to accommodate changes to project objectives. However, no deviations affected the reliability of the data. This section summarizes deviations from the TAPE work plan that occurred during the duration of the project.

### 4.7.1 Troy Field Office Record of Modifications

Field procedures were continually monitored to ensure that the objectives of the TAPE project were accomplished. There were circumstances where modifications to the procedures described in the TAPE

work plan were necessary to complete project objectives or to accommodate changes to project objectives.

Routine modifications (e.g., field team could not reach required depth of soil sample) were recorded in the field forms on the PDA. All minor handwritten entry errors in the logbook, property sketch, or POC were corrected by utilizing a single strikeout through the information to be changed, initials of the field staff member recording the modification were provided, and the date of documentation changes were noted. The corrected information was entered in proximity to the erroneous entry whenever possible. Errors encountered after the document had been scanned were corrected using electronic comments attached to the PDF file using Adobe Acrobat. Additionally, some properties that are situated outside the OU7 boundary underwent TAPE inspections. These out-of-study area properties were included in the TAPE program on a case-by-case basis and under consultation with DEQ and EPA.

Project-wide modifications were occasionally necessary and required the DEQ Project Officer to consult with the EPA Remedial Project Manager for the modification. Table 4-1 below summarizes the TFO modifications for the TAPE project. Copies of the TFO Record of Modification forms are provided in Appendix B.

**TABLE 4-1: TROY FIELD OFFICE RECORDS OF MODIFICATION**

| Record of Modification Number | Record of Modification Date | Description of Modification |
|---|---|---|
| TFO-00001 | 5/8/2007 | Changed required number of dust sampling aliquots from 30 to 10, comprised of 4 accessible, 4 infrequent, and 2 inaccessible locations. |
| TFO-00002 | 5/8/2007 | Maximum number of visual point inspections is set at 30 for an individual use area. The point inspection locations are evenly distributed and coincide with sample aliquot locations. |
| TFO-00003 | 7/12/2007 | Utilize prioritization scheme for dust sampling. |
| TFO-00004 | 7/12/2007 | Property-specific modification that does not affect the overall project. |
| TFO-00005 | 8/1/2007 | Dust sample to be collected only if visible vermiculite observed, or if the resident worked at mine or has asbestos-related disease. |
| TFO-00006 | 8/23/2007 | Revert back to dust sampling in all buildings (supersedes TFO-00005). |
| TFO-00007 | 11/15/2007 | Collect soil samples from specific use areas (SUAs) with visible vermiculite. |
| TFO-00008 | 4/29/2008 | Revisit 2007 use areas to complete an inspection and/or collect a soil sample for the purpose of confirming, semi-quantifying, and describing visible vermiculite. |
| TFO-00009 | 6/3/2008 | Collect 'field split' samples in use areas with sufficient soil volume. |
| TFO-00010 | 6/13/2008 | Cease implementation of TFO-00008. |
| TFO-00011 | 12/22/2008 | Cease the collection of a separate "field split" sample as required in TFO-00009 and instead increase soil sample volume by 50%. |

**TABLE 4-1: TROY FIELD OFFICE RECORDS OF MODIFICATION (CONT.)**

| Record of Modification Number | Record of Modification Date | Description of Modification |
|---|---|---|
| TFO-00012 | 12/22/2008 | Cease collection of dust samples as part of TAPE inspection. |
| TFO-00013 | 5/27/2009 | Perform aggressive attic inspections to thoroughly identify the presence of vermiculite attic insulation for future inspections. This also required re-visits to properties where attics were not inspected due to limited access. |
| TFO-00014 | 6/17/2009 | Include road and alley parcels as part of TAPE inspection process. |
| TFO-00001 (RDI) | 7/21/2010 | Revises the RDI SAP page 23, Section 4.4.5, Sample Labeling and Identification, to read as follows: "The sample labeling scheme is as follows: TD-XXXXX; where TD identifies that a sample is collected in accordance with the RDI SAP and XXXXX represents a 5-digit numeric code." |
| TFO-01 (RDI) | 8/5/2010 | Modifies RDI procedures for the Bruce Cole property (AD-202036), to limit the RDI to a reasonable area surrounding the barn. This is a property-specific modification that does not affect the overall project. |
| TFO-00015 | 5/4/2010 | Eliminates collection of equipment rinsate blanks for soil sampling. |
| TFO-00016 | 5/4/2010 | Adjusts the schedule of visual observations and soil sampling frequency for TAPE inspections to be consistent with the final OU4 General Property Investigation Work Plan by CDM-SMITH, dated April 2010. |

Notes:
CDM Camp, Dresser, and McKee
RDI Removal design investigation
SAP Sampling and analysis plan
SUA Specific use area
TAPE Troy Asbestos Property Evaluation
TFO Troy field office
WP Work plan

### 4.7.2 Opportunistic Samples

The TAPE inspection protocol involved the collection of soil samples from all exterior use areas with the exception of NUAs. A NUA is a portion of a parcel, or an entire parcel, that is comprised of native soil and vegetation and shows no evidence of past or present human disturbance. It is also not currently used by residents or other individuals. Since the presence of LA-contaminated vermiculite is a result of human transport and placement, the possibility of LA-contaminated vermiculite being present in a NUA was considered to be low. As a result, NUAs were excluded from the general sampling effort. NUAs were, however, visually inspected by the field teams to ensure there were no signs of human disturbance (old roads, former house foundations, etc.) as part of the TAPE investigation. If the field team determined that an NUA was disturbed, it was re-categorized (i.e. LUA or SUA) and sampled accordingly. In some cases, property owners requested that a soil sample be collected from an NUA on their property. Between TAPE project inception and completion, a total of 12 NUAs on developed parcels and three NUAs on entirely undeveloped parcels were sampled.

# 5.0 OUTDOOR AMBIENT AIR STUDY

This section describes the objectives, methods, and activities utilized during the AAS program. The information presented in this section is detailed in the OU7 AAS work plan (Tetra Tech 2009a). The OU7 AAS was initiated in fall 2009 and ended in late summer 2013. AAS sampling beyond the scope of this RI is currently ongoing at three station locations as part of OU7 post-removal ambient air monitoring.

## 5.1 OUTDOOR AMBIENT AIR STUDY OBJECTIVES

As discussed earlier in this document, previous investigations by EPA in Libby determined that LA was present in multiple environmental media. Since the potential existed for similar LA contamination in the OU7 area due to its proximity and historical mining connection to Libby, DEQ asked Tetra Tech to formulate an outdoor ambient air data collection work plan to assess outdoor ambient air as a potential exposure pathway. The AAS results will be used and presented in the site wide risk assessment.

Initial field activities included selection of site monitoring stations, assembly and installation of monitoring equipment, and initiation of outdoor ambient air sample collection. The outdoor AAS has included monitoring of ambient air in four distinct “air zones” across OU7. Seven monitoring station locations within four air zones were sampled to evaluate upwind, down-wind, and residential exposure scenarios. The four air sampling zones were originally identified based on geographic location and land use coverage. Personnel and work zone monitoring was conducted to ensure worker safety and to evaluate real-time airborne levels of asbestos in ambient air.

The primary objectives of the AAS were to measure LA concentrations in outdoor ambient air and combine the collected data with discrete data from other exposure routes to support the HHRA and the evaluation of future remedial actions. Specifically, as part of the DQOs, the AAS was designed to determine (1) whether the levels of LA in outdoor ambient air contribute a risk of cancer or non-cancer effects, either alone or in combination with other exposure pathways; (2) whether that risk is within an acceptable range of risks under a reasonable maximum exposure scenario; and (3) whether the data identify any significant differences of the levels of LA in outdoor ambient air as a function of time or location in OU7. A more thorough discussion of the AAS DQOs can be found in Section 3 of the AAS work plan (Tetra Tech 2009a).

## 5.2 OUTDOOR AMBIENT AIR STUDY FIELD INVESTIGATION METHODS

The following sections provide a summary of the tasks implemented to complete the AAS.

### 5.2.1 Site Access, Logistics, and Pre-Sampling Activities

Prior to beginning the sampling, DEQ and Tetra Tech contacted the property owners or representatives where sampling was proposed to determine their desire to participate and to explain the details and expectations of the sampling program. The property owners or representatives were advised on the study's duration, and were informed of the importance of obtaining samples consistently over an extended period. Tetra Tech personnel explained the program and the potential impact to the property (e.g., installation of the housing unit, sampling at regular intervals, and the expected duration of the program) to each resident or representative. Tetra Tech also requested that the residents or representatives inform them two days prior to any site disturbance around a sampling station. DEQ obtained signed access agreements for each property specifically for the AAS investigation. The access agreements were scanned to PDF and added to their respective AAS scanned data archive folder and the original agreements were added to the AAS paper files (sorted by sampling period) in the DEQ Troy Information Center.

Prior to beginning field activities, Tetra Tech held a field planning meeting, completed an inventory of equipment and supplies and equipment procurement, identified station locations, and constructed the outdoor ambient air and meteorological sampling stations. The following sections discuss these pre-sampling activities.

<u>Field Planning Meeting/Equipment Inventory and Procurement</u>

The Tetra Tech Field Manager conducted a field planning meeting with field and QA staff to discuss objectives of the field work, equipment and training needs, SOPs and the schedule of events, health and safety measures, equipment requirements, and required site documentation.

Following the meeting, field staff conducted an inventory of equipment in the field office and determined what supplies would be purchased and what additional equipment would need to be procured.

Identify Outdoor Ambient Air Sampling Locations

Outdoor ambient air sampling stations were located in four air sampling zones throughout OU7. The four air sampling zones were identified based on geographic location and land use coverage and are depicted on Figure 5-1. Final sampling locations were identified prior to initiation of sampling based on site logistics, landowner agreement, and site security. Tetra Tech assigned unique sample station identification numbers to all stations. Seven outdoor ambient air sampling locations were selected within the four distinct outdoor ambient air sampling zones of OU7.

The predominant winds in Troy tend to flow in southeast and northwest directions, following the river corridor within which OU7 is located. Two sampling stations (one each) were placed in proximity to the northwest and southeast boundaries of OU7. This ensured that there were upwind and downwind sample collection stations for both predominant wind directions. Two stations (one each) were also located on the northwest and southeast borders of downtown Troy in order to have upwind and downwind sample stations in the area with the highest population density. One sample station was placed at the DEQ Troy Information Center to measure LA concentrations in downtown Troy. A station was also placed in the northeastern portion of OU7 and the last station was placed along Iron Creek Road in the southwestern portion of OU7 (Figure 5-1). Station locations were moved to new locations within the same four air zones during Year 2 and Year 3 sampling. Year 2 and Year 3 station locations are shown on Figure 5-1. Section 5.7 provides a description of the new station locations and rationale for the choice of the new locations.

Outdoor Ambient Air Sample Station Installation

Tetra Tech obtained the ambient air sampling station boxes formerly used in OU4 from a Libby storage facility on October 7, 2009. Eight station boxes were obtained with the following serial numbers: 3794-1, 3794-3, 3794-7, 3794-9, 3794-11, 3794-12, 3794-13, and 3794-14. Using Hudson sprayers and disposable towels, Tetra Tech decontaminated the boxes prior to transporting them to the TFO. Tetra Tech purchased materials and supplies to set up the sampling station boxes during the weeks of October 12 and 19, 2009.

Tetra Tech obtained access agreements from property representatives before the sampling station boxes were installed on October 20 and 21, 2009. Contact dates and names of those individuals granting access to each site were recorded on Field Sampling Data Sheets (FSDS) along with all field data

collected. The basic design for the FSDS was created before field-testing began and was modified during the first two sampling periods.

Once the sampling stations were placed at each location, the GPS coordinates of each station were recorded and site photographs were taken.

Outdoor ambient air sampling stations were set up to collect samples from the height of an adult's breathing zone, approximately 5 feet above ground level by using adequate lengths of Tygon® tubing that reached from the sampling pump in the equipment box to a sampling stand designed to hold the air sampling cassette. A rain and snow shield was placed over the equipment to prevent moisture-related impacts to sampling results.

Meteorological Station Installation

Meteorological data (wind speed, direction, temperature, humidity, and precipitation) were collected from a portable weather station installed at the DEQ Troy Information Center and Field Office. Tetra Tech assembled the station at the DEQ Troy Information Center to collect meteorological data during the outdoor ambient air sampling events. The station consisted of a CR200 data logger capable of recording the average wind speed, wind direction, temperature, humidity, pressure and precipitation at hourly intervals during each 120-hour sampling event. The meteorological station was supplied with power from the field office.

Sampling Equipment Setup and Calibration

Sampling station equipment was designed to use battery-operated air sampling and heating equipment in order to work independent of onsite electrical sources. In order to sample continuously for 5 consecutive days with data recording capabilities, the battery operating criteria required Tetra Tech to purchase air sampling equipment that could use 12-volt deep cycle batteries. Tetra Tech purchased air sampling pumps that met these criteria. The selected pumps were SKC AirChek2000, which are equipped with battery eliminator attachments that can be connected directly to the 12-volt batteries. Although the air pump set-up met the manufacturer's specifications, Tetra Tech could not be sure that the apparatus would work successfully for the OU7 ambient air project requirements without first completing a test run. After pump calibration, separate short-term test runs of the sampling equipment and power supply system were completed in the days prior to actual sampling startup. Results of

short-term testing indicated that the equipment was functioning properly and was ready for full-length sampling periods. Once again, after pumps were calibrated, implementation of the OU7 air monitoring program was initiated in October 2009. Section 10.3 provides a summary of AAS sampling completed during the period of 2009 through 2013 and provides a summary of data collected.

### 5.2.2 Field Documentation

Field documentation generated during this sampling program included the following: FSDSs, field logbooks, photographs (as necessary), and sample custody documentation. FSDSs, logbooks, and photographic documentation are discussed below. Sample custody documentation is presented in Section 5.2.5.

Field Sample Data Sheets

The field staff documented sampling information from each sampling event on FSDSs, which were maintained for the duration of the project. The sheets provided an account of sampling at each station including sample start and finish dates, start and finish times, sample station identification number, sample identification number, pump number, pump fault, start and finish flow rates, total sample volume, total sample time, primary battery voltage, photographs taken (if any), and any unusual conditions (e.g. significant weather events, construction, or municipal activities). Information was manually recorded on each FSDS.

Field Logbook

In addition to the FSDS, field staff entered field-related information into a field logbook. Information recorded in the field logbook included phone calls with other field staff or owners or operators of sampling station properties, photographs taken, unique weather or situational activities that could have potentially impacted sample results, and pump issues and maintenance. Copies of the logbook pages were regularly scanned into PDF files and stored in the electronic data archive. The original field logbook is maintained in the DEQ Troy Information Center.

Photographic Documentation

Digital photographs were recorded for each sampling location at the first collection event and any time thereafter if the equipment was moved or changed. Photographs were documented on the FSDS and in

the field logbook; the information recorded included the time of day, the orientation of the photograph, and pertinent site features. The photographs were each assigned a unique number and were included in the electronic data archive.

### 5.2.3 Equipment and Personnel Decontamination

Decontamination of field equipment used for assembly, station repair, or necessary maintenance (e.g. during maintenance or replacement of sampling pumps or enclosures) was completed where the sample station was located. Decontamination included wiping the equipment with pre-moistened cleaning wipes or spraying the equipment (as appropriate) with distilled water and drying it with paper towels. The water was allowed to fall on the ground in the area of the sampler, and the paper towels or cleaning wipes were placed in a labeled asbestos waste bag for disposal.

Visible soil on hands or clothing was removed by washing with soap and water. Prior to achieving a successful negative exposure assessment following the first sampling period, PPE included disposable gloves, disposable protective outerwear, work boots, disposable boot covers, and respirators. After completion of the negative exposure assessment, the PPE was reduced to Level D for the remainder of the AAS investigation. The respirators were cleaned and decontaminated as discussed in the outdoor ambient air health and safety plan and respirator cartridges were placed in labeled asbestos waste bag for later disposal.

### 5.2.4 Containment and Disposal of Investigation-Derived Waste

IDW included the following used items: wet wipes, wet paper towels, disposable gloves, respirator cartridges, disposable plastic tubing, disposable protective outerwear, plastic floor coverings (from sampling station decontamination), and other minimal waste. Only small amounts of tubing were used during calibration, replacement, and repair of any defective or worn tubing. As it was possible that these IDW materials contained asbestos, all IDW was double-bagged in appropriate asbestos bags, labeled with asbestos labels, and stored in an approved containment area at the DEQ Troy Information Center until it was properly disposed of at the Lincoln County Landfill, outside of Libby, Montana. Non-sampling waste generated by the field staff, such as food containers and waste paper, was separately bagged and properly disposed of as solid waste.

### 5.2.5 Record Keeping and Chain of Custody

An individual file (both paper and electronic) was maintained for each air sampling station. Originals of all field forms were kept in each individual air sampling station file in the DEQ Troy Information Center for the duration of the project, so that information is available if questions arise. Scanned PDF copies of all field forms were stored in electronic files (electronic data archive) for each sampling station. In addition to field forms, signed and released copies of COC forms from the laboratory were stored in the DEQ Troy Information Center. A backup electronic copy of the OU7 Outdoor Ambient Air Scribe Database and individual electronic air sampling station files were stored in the Tetra Tech office in Helena, Montana, and were updated periodically during the sampling and reporting phases of the project.

COC procedures were implemented to handle all outdoor ambient air samples. The Tetra Tech Sample Database Coordinator prepared each COC report and the Tetra Tech field staff members printed the reports and stored them with the outdoor ambient air samples. The COC reports were then transferred to the laboratory when the samples were shipped at the end of each 5-day sampling event.

### 5.2.6 Sample Management

Samples were labeled with unique identification numbers, “TA-XXXXX” with “TA” indicating that it was a “Troy AAS” sample and “XXXXX” being a five-digit number. The TA sample labels were supplied by the Sample Database Coordinator and were signed out by the field staff (i.e., controlled). One sample label was placed on each sampling cassette. The sample identification number was recorded on the FSDS and was also written on the outside of the plastic bag used to hold the sampling cassette during transport.

Tetra Tech sample team members placed custody seals on each sample as described in Section 4.4.11 of the AAS work plan (Tetra Tech 2009a). Sample storage bins were used to transport the outdoor ambient air samples back to the DEQ Troy Information Center office. Samples were handled as little as possible and transportation times minimized so as to limit excess vibration and potential for dislodging of fibers from the filter material. Temporary storage of the samples was in storage bins in a secured (locked) area at the field office. All samples collected from the AAS, including QC samples, were transferred to the laboratory at the end of each 5-day sampling event or were securely held at the DEQ Troy Information Center until laboratory space became available. A hard copy of the COC report was

prepared by Tetra Tech and accompanied each shipment of samples to the laboratory. Additional information on sample management and COC procedures is found in Section 5.4.

## 5.3 AIR SAMPLE ANALYSIS

Outdoor ambient air samples were prepared and analyzed by TEM in accordance the International Organization for Standardization (ISO) Method 10312 Annex E and applicable project-specific modifications, including LB-000016, LB-000019, LB-000028, LB-000029, LB-000030, LB-000053, and LB-000066 (Appendix A). Detailed analytical requirements (e.g., counting rules, required sensitivities) are listed in analytical requirements summary associated with the AAS work plan (Tetra Tech 2009a).

## 5.4 DATA MANAGEMENT

Management of the outdoor ambient air data was conducted under the guidance and supervision of the Tetra Tech Sample Database Coordinator. The following sections describe management of the field data, the electronic data archive, and analytical data.

### 5.4.1 Field Data

As stated previously, a portion of the field data was manually recorded on FSDS and in a logbook at the beginning and end of every sampling event, as well as during periodic daily checks to ensure sample accuracy. After completion of each sampling round, field staff scanned the FSDS and logbook pages (to PDF files) for submittal to the electronic data archive. Field staff also downloaded digital photographs to a computer at the DEQ Troy Information Center office. Scanned access agreements, FSDSs, digital photographs, and any other documentation pertinent to the outdoor ambient air program were placed in the electronic data archive. Tetra Tech maintained a separate electronic data archive for the AAS project. The folders for the AAS electronic data archive were organized by sample station. The AAS electronic data archive was stored on the CIC's computer at the DEQ Troy Information Center office and a backup copy was maintained on the Outdoor Ambient Air web portal. All hard copies of field documentation were stored in files at the DEQ Troy Information Center office.

### 5.4.2 Electronic Data

Electronic field data were recorded through the data logging capabilities of the SKC, Inc. AirCheck2000 pumps used for the AAS. The pertinent sample data from each pump and sampling event was

downloaded by field staff into a temporary Microsoft Access import database called the Troy Ambient Air Database (TAAD) that was used for pre-processing the pump data before importing data into the OU7 Outdoor Ambient Air Scribe Database. The field staff then reviewed the data for accuracy before the data were published to the OU7 Outdoor Ambient Air Scribe Database by the Sample Database Coordinator. Part of the data review process included checking the FSDS against the data downloaded from the pump.

Subscribers to the Scribe.net OU7 Outdoor Ambient Air Scribe Database were able to download a complete database that contains all station, sample, analytical, and custom queries.

After the data were successfully downloaded and the OU7 Outdoor Ambient Air Scribe Database was published to the internet and the air sampling pumps were cleared of the sample event data and programmed for the next sample event.

### 5.4.3 Analytical Data

Outdoor ambient air analytical data were received from the laboratory in both PDF and EDD format. An EDD was provided for each individual sample from the laboratory in Excel format. Additionally, scanned laboratory bench sheets necessary for data validation were received from the laboratory in PDF format. The information contained in the EDDs was entered into the OU7 Outdoor Ambient Air Scribe Database. The ambient air data verification process is summarized in Section 5.6.2.

## 5.5 QUALITY CONTROL SAMPLES

Three types of QC samples were collected as part of this investigation: lot blanks, field blanks, and co-located samples. All QC samples were submitted “blind” to the laboratory using unique sample identification numbers similar to those of real field samples.

**Lot Blanks.** Before any cassettes were used, a cassette from each filter lot was randomly selected and submitted for analysis. One lot blank cassette was submitted from each 50-cassette box prior to field use. Data for lot blank samples were used to evaluate whether cassettes were received asbestos-free from the supplier. Tetra Tech did not use a cassette from a given lot until the lot blank results confirmed the cassettes were asbestos-free. The lot blanks were analyzed for asbestos fibers by the same TEM method used for field sample analysis.

**Field Blanks.** One field blank was collected per 5-day sampling event for the duration of the ambient air monitoring program. The field blanks were collected by opening the sample cassette package and exposing the cassette to the full range of field efforts, including sample handling, car travel, 10 seconds attached to the air sample pump (not turned on), sample cassette retrieval, return to office, packaging, and transport to the laboratory. The field blanks were analyzed for asbestos fibers by the same TEM method used for field sample analysis. Data for the field blank ambient air samples were evaluated to assess whether a potential existed for sample cross-contamination during sample handling.

**Co-located Samples**. Co-located samples were collected to determine the analytical variability of the TEM method for asbestos fibers. One co-located sample was collected per sampling event. Field co-located samples were collected from the same location during Year 1 of the project (Station Number T4QC). Station T4QC was located approximately seven feet from sampling station T4 at the DEQ Troy Information Center. At the start of outdoor AAS program in Year 2 (beginning with period 37) and continuing through Year 3 (Period 101), co-located samples were collected at rotating station locations so that the same number of co-located samples was collected at each station throughout the year. Section 5.7 discusses outdoor AAS changes regarding collection of co-located samples. All co-located samples were handled and sent to the laboratory for analysis using the same TEM method used for field samples.

## 5.6 DATA QUALITY ASSESSMENT

The primary objective of data quality assessment was to ascertain the accuracy and usability of the data collected in meeting the DQOs of the project. This objective was achieved; by verifying the accuracy and completeness of the field data collected, as described in Section 5.6.1, and by verification of the analytical data, as described in Section 5.6.2. The field data verification process was performed on AAS field data as it was collected and on analytical data as it came available throughout the life of the project.

### 5.6.1 Field Data Verification

Outdoor AAS field data from each sampling round were reviewed for completeness, accuracy, and consistency to ensure adequate quality of the collected outdoor ambient air samples. Pump history files were first reviewed to ensure performance met acceptable criteria. To ensure data were consistent, the pump history data were then compared to data manually recorded on FSDSs and in the ambient air

logbook. Data from the pump history files, FSDSs, and the logbook were then checked against the TAAD to again ensure that data were consistent among the data summary locations, and that notable events had been documented throughout. If discrepancies were noted on the FSDS forms or in the logbook, the field team was contacted, and changes to the appropriate files were made via electronic notes in the PDF files. Discrepancies or errors identified in the TAAD database were addressed by manually correcting the entries in the database. Finally, a review of the OU7 Scribe Database occurred after data entry to verify that transfer of data from TAAD was complete and no errors were present.

### 5.6.2 Analytical Data Verification

Outdoor AAS samples were analyzed using TEM according to ISO Method 10312 Annex E and applicable project-specific modifications, including LB-000016, LB-000019, LB-000028, LB-000029, LB-000030, LB-000053, and LB-000066 (Appendix A). Detailed analytical requirements (e.g., counting rules, required sensitivities) are listed in analytical requirements summary associated with the AAS work plan (Tetra Tech 2009a).

Tetra Tech conducted data review and data entry verification of the outdoor AAS data in accordance with SOP EPA-LIBBY-09 (Appendix A). Tetra Tech followed the data review and data entry verification procedures outlined in this SOP, with some minor deviations for OU7. The deviations are explained, where applicable, in the subsections below.

Tetra Tech's data review and data entry verification process involved three steps: (1) selection of data records for review and verification, (2) review of the original laboratory bench sheets, and (3) verification of transfer of results from the bench sheets onto the EDDs. Tetra Tech also reviewed field QC sample results for adherence to minimum frequency requirements and procedures and QC limits specified in the work plan (Tetra Tech 2009a). The data review and data entry verification process are described in the subsections below.

<u>Selection of TEM Records for Review</u>

SOP EPA-LIBBY-09 specifies review and verification of a minimum of 10 percent of the sample records. Tetra Tech deviated from this minimum requirement and reviewed 100 percent of the sample records for sampling periods 1 through 5, approximately 50 percent of the sample records for sampling periods 6 through 18, approximately 25 percent of the sample records for sampling periods 19 through 36, and

approximately 10 percent of the sample records for sampling periods 37 through 101. The decision to exceed the minimum review and verification requirement early in the study was in part due to the high incidence of significant errors (e.g., incorrect transfer of structure counts from bench sheets to EDD) noted during analysis of early samples, and in part because the structure of the database was changed in early 2010 at the request of EPA, resulting in emergence of a number of data formatting and structural issues. Details regarding errors and issues identified during data review and verification are provided in the individual AAS quarterly memoranda.

SOP EPA-LIBBY-09 also specifies a query of the OU7 Scribe database to select specific percentages of analytical records to verify—representing laboratory and analyst, as well as detected and non-detected results. Outside of Year 1, analytical data verification record selection generally conformed to the record selection process described in detail in SOP EPA-LIBBY-09 (Appendix A).

Consistency Review of Laboratory Bench Sheets

Tetra Tech inspected the information recorded on the original hand-written laboratory bench sheets in accordance with the procedure for reviewing consistency of laboratory bench sheets outlined in Section 5 of SOP EPA-LIBBY-09 (Appendix A), with minor OU7-related modifications. The bench sheets were reviewed to identify any data omissions, apparent inconsistencies, or potential errors in structure. The objective was to assess whether the raw structure data were recorded in accordance with method counting rules, as modified by the applicable project-specific modifications (Appendix A).

**Corrective Action –** Tetra Tech summarized all apparent inconsistencies, omissions, and suspected errors, and provided these to the Environmental Services Assistance Team (ESAT). The ESAT determined which of these were authentic and would require correction, and then forwarded them to the appropriate laboratories for response.

Verification of Data Transfer from Bench Sheet to Database

To ensure that data from laboratory bench sheets were transferred through the EDDs into the OU7 Scribe database without error or omission, Tetra Tech compared selected analysis-specific information in the laboratory bench sheets to that in associated EDDs. Tetra Tech followed the verification of data transfer procedure outlined in Section 6.0 of SOP EPA-LIBBY-09 (Appendix A), with minor modifications for OU7. The bench sheets include the laboratory COC form, sample check-in form, preparation log, and

hand-written data record sheets. This process (1) compared analysis-specific information in the EDD to the original laboratory job documentation (e.g., internal laboratory COC, preparation logs, bench sheets, etc.); and (2) verified (by recalculation) reported air sensitivities for amphibole and chrysotile, the area analyzed, and, for indirect preparations, the indirect preparation dilution factor. Using the bench sheets, Tetra Tech also recounted the countable LA structures across all grid openings evaluated and compared this number (and the calculated concentrations) to the total number of LA structures in the EDD.

The final step in the verification of data transfer process was to verify that the data in the EDDs were loaded into the OU7 Scribe database without error or omission.

**Corrective Action –** Tetra Tech summarized all apparent inconsistencies, omissions, and suspected errors, and provided these to ESAT. The ESAT determined which of these were authentic and would require correction, and then forwarded them to the appropriate laboratories for response.

Review of Field Quality Control Sample Results

Tetra Tech reviewed the field QC sample (co-located and field blank sample) results, and initiated any necessary corrective actions. Each co-located sample pair was compared using the Poisson rate test, included in laboratory modification LB-000029 (Appendix A), to determine whether the results differed statistically from one another at the 95 percent confidence level. The Poisson rate test is suitable for this analysis because fiber counts on TEM grids are considered independent and random.

**Corrective Action** –For co-located field sample pairs, Tetra Tech reviewed the Poisson rate test results and investigated the basis for any significant statistical differences and need for any appropriate corrective actions. Poisson rate test results that indicate the co-located samples are similar at the 95 percent confidence interval are considered good. Test results in the 90 to 95 percent confidence interval range are considered acceptable, and test results that fall below the 90 percent confidence interval are considered poor for similarity.

If test results were found below the 90 percent interval, Tetra Tech investigated the basis for the discrepancy and implemented as-needed corrective action in sampling procedures. Tetra Tech reported the results from the original sample (as opposed to co-located or laboratory recount sample results). A possible exception to this rule was ESAT inter-laboratory recount results. If, during validation, an inter-

laboratory recount result was deemed more representative than the original result, Tetra Tech discussed the findings with the DEQ and reported whichever result was determined the most representative.

For field blanks, Tetra Tech reviewed and the results for all field blanks for adherence to the QC limits specified in project-specific laboratory modification LB-000029 (Appendix A). All field blank results have been within QC limits.

### 5.6.3 Statement on Data Quality

The objectives of the AAS were to obtain the data necessary to determine: (1) whether levels of LA in outdoor ambient air pose a risk of cancer or non-cancer effects, either alone or in combination with other exposure pathways; (2) whether that risk is within an acceptable risk range under a reasonable maximum exposure scenario; and (3) whether the data identify any significant differences in levels of LA in outdoor ambient air as a function of time or location in OU7. The outdoor AAS DQOs appear in the work plan (Tetra Tech 2009a).

To address AAS objectives, Tetra Tech collected and analyzed outdoor ambient air samples for LA. A total of 639 outdoor ambient air samples (including co-located and blank samples) were collected from project inception through completion of the project.

To ensure accuracy and reliability, outdoor AAS field data and analytical results underwent review and verification in accordance with the procedures specified in the work plan (Tetra Tech 2009a). Data quality appeared adequate for supporting exposure scenario and risk assessment calculations. The AAS data will be used and presented in the site wide risk assessment.

## 5.7 DEVIATIONS FROM THE OUTDOOR AMBIENT AIR STUDY WORK PLAN

All minor FSDS entry modifications were made utilizing a single strikeout through the information to be changed, initials of the field staff member recording the modification were provided, and the date documentation changes were noted. The corrected information was entered in proximity to the erroneous entry where possible. Errors encountered after the FSDSs had been scanned were corrected using electronic comments attached to the PDF file using Adobe Acrobat, and explained or clarified the erroneous entry.

The following provides a summary of the deviations from the outdoor AAS work plan (Tetra Tech 2009a) that apply to the entire outdoor AAS investigation and data. All associated TFO forms were submitted to the DEQ Project Officer for review and approval. The signed copies were placed in the DEQ Troy Information Center office. Some deviations were not documented through a TFO; however, they were approved by DEQ without submittal of a formal TFO due to time constraints or funding limitations.

TFO-00001

The choice of 0.45-µm filter pore size was originally made to coincide with the EPA Asbestos Sampling SOP #2015, Section 5.2 - Filter Cassettes, Paragraph 5.2.1 – TEM Cassette Requirements, which recommends "the cassette shall be loaded with an mixed cellulose ester (MCE) filter of pore size 0.45 µm, and supplied from a lot number which has been qualified as low background for asbestos determination." However, sampling at high air volumes can create excessive filter loading which may lead to air pump faults. This problem is greater using the finer-mesh 0.45-µm filter pore size and may be reduced by using an alternative 0.8-µm pore size filter. Prior to beginning the OU7 ambient air study, Tetra Tech discovered that the 0.8-µm filter was used for TEM air monitoring in Libby during the OU4 ambient air study. Tetra Tech also selected 0.8-µm filters for the OU7 ambient air sampling to minimize sample pump faults due to the high air volumes being collected (approximately 14,400 liters were typically collected during the Libby study). The OU7 study proposed to collect between 14,400 and 21,600 liters of air per sample which increases the likelihood of pump faults due to filter loading; therefore, upon discovering the protocol that was used during the OU4 study, Tetra Tech decided to alter the procedure during the OU7 study as well. A copy of TFO-00001 is in Appendix A.

TFO-00002

Because of periodic overloading of the sample filters, TFO-00002 was implemented to change the sampling pump air flow from 3 liters per minute to 2 liters per minute (resulting in a decrease in total air volume sampled from 21,600 to 14,400 liters). Reducing the air flow eliminated the incidence of overloaded sample filters and still allowed generation of high-quality data that met sensitivity analysis requirements without counting of excessive filter grid openings during TEM analysis. This modification was implemented as a permanent procedural change beginning in Year 1, Sample Period 13. A copy of TFO-00002 is in Appendix B.

TFO-00003

This TFO specified the relocation of ambient air monitoring stations for Year 2 sampling to achieve more comprehensive coverage of the four air zones in OU7. A copy of TFO-00003 is in Appendix B.

TFO-00004

This TFO modified sampling protocol so as to rotate the co-located sampling station (Station TQC) among all seven ambient air sampling stations for Year 2 sampling. Analytical protocol was not affected; however, moving the co-located sampling station allowed an evaluation of analytical variability among all seven monitoring stations. Co-located field samples were collected (station TQC) at rotating station locations during each sample period throughout Quarter 5. Station TQC was placed next to the monitoring stations. Station TQC was moved after each sampling period (beginning with monitoring station T11) and was cycled through each of the remaining stations (T12, T13, T14, T15, T16, T17). After cycling through station 17, station TQC was returned to station T11 to start the process anew. Cycling of station TQC continued throughout Year 2 so that a minimum of five co-located samples were collected at each of the seven monitoring stations. A copy of TFO-00004 is in Appendix B.

TFO-00005

This TFO requested relocations of ambient air monitoring stations for Year 3 sampling to achieve more comprehensive coverage of the four air zones identified in OU7. For Year 3 sampling efforts, four sampling stations were re-established at locations used during Year 1. One station was located within each of the four unique "air zones" as shown Figure 5-1. Two sampling stations were re-established at locations in proximity to the northern (T21) and southern (T24) boundaries of OU7. This ensured that there are upwind and downwind sample collection stations for both directions the wind was blowing. One sample station (T22) was located in the densely populated area of downtown Troy and a final station (T23) was re-located south of Troy along the Iron Creek Road. A copy of TFO-00005 is in Appendix B.

Quarter 12 and Quarter 13 Station/Sampling Schedule Changes

Due to an interruption in project funding, Quarter 12 sampling was not initiated until February 18, 2013, leaving a gap of two months from the final Quarter 11 sampling period. Additionally, the Quarter 12

sampling schedule was reduced to one period per month; three sampling periods (97, 98, and 99) were completed during Quarter 12.

Quarter 13 covered three sampling periods (100, 101, and 102) during May, June, and July of 2013. Period 100 sampling was completed at four stations as per TFO-00005; however, sampling periods 101-102 efforts were reduced to three stations (T21, T22, and T24). Quarter 12 and Quarter 13 sampling changes were approved by the DEQ; however, due to time constraints and funding limitations, TFO forms were not completed to document the changes.

**Analytical Data Review and Verification**

Tetra Tech conducted review and verification of the analytical data in general accordance with project-specific SOP EPA-LIBBY-09 (Appendix A), with minor modifications for OU7.

The SOP specified data review and data entry verification of a minimum of 10 percent of the sample records. It also specified criteria for selecting sample records for review and verification. However, 100 percent of the period 1 through 5 sample records, approximately 50 percent of the period 6 through 18 sample records, and approximately 25 percent of the period 19 through 23 sample records underwent data review and data entry verification. For these period 1 through 23 records, rather than random selection from the OU7 Scribe Database as specified in the SOP, records were hand-selected for review and verification based on result type (detected LA) and sample type (field duplicate pairs and field blanks). This deviation occurred because the sample records had not been published in the Scribe Database, which would have allowed application of the random selection process described in the SOP. For periods 24 through 72, approximately 10 percent of the sample records underwent data review and data entry verification. These records were randomly selected, in general accordance with project-specific SOP EPA-LIBBY-09 (Appendix A).

Sample records from period 102 did not undergo analytical data review and verification as the laboratory data did not come available until after the expiration of Tetra Tech's task order in July 30, 2013. Data from sample period 102 is now part of the on-going OU7 post-removal ambient air monitoring and will be verified by another contractor at a later date.

# 6.0 OUTDOOR ACTIVITY-BASED SAMPLING

This section summarizes objectives, methods, and procedures of the outdoor ABS sampling program, which was initiated in April 2011. For in-depth details on the outdoor ABS program, refer to the *Final Activity-Based Sampling, Sampling and Analysis Plan for Operable Unit 7 of the Libby Asbestos Superfund Site* (Tetra Tech 2011b).

## 6.1 SAMPLING OBJECTIVES AND INVESTIGATION METHODS

Investigations in Libby, Montana, revealed presence of LA in multiple environmental media including indoor air, outdoor ambient air, attic insulation, and soil. Because of the proximity of Libby and Troy as well as their historical connections, it was determined that the magnitude of LA and potential exposure of Troy residents to this were unknown, and that additional data were needed to complete an HHRA. Therefore, two outdoor ABS events were performed; one in May 2011 (wet season) and one in August 2011 (dry season). Data from this investigation was used in the preparation of the HHRA. ABS investigation methods are summarized below.

### 6.1.1 Pre-Sampling Planning

The HHRA work plan (Tetra Tech 2011a) identified outdoor ABS data for two residential property categories and for community-wide scenarios as data gaps. Samples representing the following residential property categories were collected:

- Residential properties for which removal of soil was deemed unnecessary
- Residential properties for which removal of soil had been completed

Community-wide ABS samples were also collected to evaluate threats to human health from community-wide (non-residence-specific) exposures to LA in air.

Prior to start of field activities, residential UA sampling locations were selected using a query of the OU7 database. To the extent possible, the residential locations were selected from representative locations across OU7. Property owners were contacted to determine their willingness to participate prior to the start of ABS. This process continued until the desired number of sampling locations had been selected.

Before the field crews mobilized, Tetra Tech prepared detailed property maps identifying the OU7 residential properties to be included. Maps were also prepared and reviewed to establish the recommended routes for the recreational biking and driving ABS scenarios.

### 6.1.2 Investigation Methods and Activities

Although a wide variety of activities may result in outdoor disturbances of LA, it was not feasible to evaluate every type of disturbance. As a result, six disturbance scenarios were identified as realistic and representative of outdoor LA disturbances within OU7. Air sample collection is summarized in Section 6.1.2.1 and soil sample collection is summarized in Section 6.1.2.2.

#### *6.1.2.1 Air Sampling*

Air samples were collected during the six disturbance scenarios described below. Residential ABS air sample collection locations are depicted on Figure 6-1, and recreational ABS air sampling locations are depicted on Figure 6-2. The outdoor ABS bicycling sampling route is depicted on Figure 6-3. The outdoor ABS driving route is depicted on Figure 6-4.

**Scenario 1: Yard Work in Residential Yards**

This scenario assumed three activities involving residential yard work that were considered realistic examples of relatively vigorous disturbances:

- Raking the lawn or yard with a metal-tined leaf rake
- Digging in the soil with a shovel
- Mowing the yard with a gasoline-powered rotary lawn mower

Because risk is related to long-term average exposure across all of these activities, ABS samples were composited from the three types of disturbance activities. Samples were collected from residential properties where removal of yard soil had not been required and from residential properties where removal of yard soil had been completed.

Adult residents, teenagers, and groundskeepers are the most likely receptors and age groups under this scenario, so samples were collected at the adult breathing height. Sampling occurred for a total of one hour on a single filter cartridge; air was collected during 21 minutes of raking, 18 minutes of digging, and 21 minutes of mowing.

To account for seasonal factors and provide ABS samples representative of potential exposures over multiple seasons, two sampling events occurred—one in the spring (wet season) and one in the late summer (dry season). A total of 82 ABS air samples (41 high volume and 41 low volume, not including field QC samples) were collected for this scenario. The low volume samples were analyzed only if the high volume samples were overloaded. Of these, 41 samples were collected during dry season and 40 samples during wet season.

**Scenario 2: Gardening in Residential Gardens**

This scenario assumed two activities involving residential garden soil that were considered realistic examples of relatively vigorous disturbances:

- Rototilling
- Digging with a trowel

Samples were collected at residential properties where no removals had occurred and properties where removals and restoration had occurred. Similar to ABS Scenario 1, a long-term average exposure across both of these activities was represented by collecting composite ABS samples. Sampling occurred for one hour on a single filter cartridge; air was collected during 45 minutes of digging and 15 minutes of rototilling.

A total of 76 ABS air samples (38 high volume and 38 low volume, not including field QC samples) were collected under this scenario. The low volume samples were analyzed only if the high volume samples were overloaded. Of these, 38 samples were collected during both the dry season and the wet season.

**Scenario 3: Bicycling and Playing on Unpaved Residential Driveways**

This scenario assumed that a child riding a tricycle and playing on an unpaved driveway may disturb the soil. Activities under this scenario included bicycling on and digging in driveways, with samples collected at a child's breathing height at both categories of residential properties.

The tricycle-riding activity occurred at each selected driveway for 30 minutes. The child actor rode a tricycle up and down the driveway, covering the entire area of the driveway during that time period.

Digging occurred at each selected driveway for 30 minutes. The child actor sat on the ground on the driveway while digging or scraping the top 2 to 6 inches of soil, pushing soil and rocks to the side, and

then replacing them. The digging activity occurred for approximately 5 minutes at each of six discrete and widely distributed locations along the driveway (Tetra Tech 2011b).

Similar to ABS Scenario 1, long-term average exposure across bicycling and playing activities was represented by collecting composite ABS air samples. Samples were collected at the child breathing height because a residential child is the primary receptor and age group under this scenario.

A total of 80 ABS air samples (40 high volume and 40 low volume, not including field QC samples) were collected under this scenario. The low volume samples were analyzed only if the high volume samples were overloaded. Of these, 40 samples were collected during both the dry season and the wet season.

**Scenario 4: Driving on Paved and Unpaved Roads and Alleys**

Scenario 4 assumes driving in a motorized vehicle on roads and alleys within OU7 may result in exposure to LA from disturbance of road surfaces. Composite ABS air samples were collected during driving on paved and unpaved roads and alleys within OU7 to represent combined exposures from a variety of road surfaces.

Driving occurred for 60 minutes. The driving actor drove a full-sized automobile on both paved roads and unpaved roads and alleys. Travel was evenly distributed across road types throughout OU7. During sampling, the front windows of the vehicle were fully open, and the back two windows were open approximately one inch. Driving routes were documented using a portable GPS unit.

The driving ABS activity occurred during non-rain periods when the roads were free of excess water or runoff. Samples were collected at the breathing height of an adult while seated in a vehicle because adults (drivers and passengers) are the most frequent receptors and age group under this scenario based on length of exposure time and duration. Although children and teenagers are also potential receptors under this scenario, differences in breathing heights among adults, children, and teenagers in a motor vehicle were not considered large enough to warrant additional sampling.

A total of 40 ABS air samples (20 high volume and 20 low volume, not including field QC samples) were collected under this scenario. The low volume samples were analyzed only if the high volume samples were overloaded. Of these, 20 samples were collected during both the wet season and the dry season.

**Scenario 5: Bicycling on Paved and Unpaved Roads, Alleys, and Trails**

Composite ABS air samples were collected during bicycling on paved and unpaved roads and alleys within OU7 to represent combined exposures from a variety of road surfaces. This scenario (riding bicycles on roads, alleys, and trails within OU7) was assumed to cause more soil disturbance than would other recreational activities such as walking or running.

The bicycling activity occurred for 60 minutes. The actor-samplers used non-motorized, 2-wheeled bicycles. A bicycle trailer was attached to the back of one of the bicycles, and a personal air monitor was mounted inside the trailer to collect the child ABS sample. Two bicycle riders were fitted with air sample pumps and monitoring cassettes affixed in the breathing zone. The bicyclists rode in single file along a predetermined route for 60 minutes. The distance maintained between the riders depended on visibility, terrain, and safety considerations. The riders alternated positions (leading and trailing) throughout the scenario. The trailing rider rode in the dust cloud of the rider in front as much as was safe and practical. During these events, the bicycle riders varied their speed between 3 and 15 miles per hour (mph), attempting to maintain an average speed of 8 mph. Bicycling routes were documented using a portable GPS unit.

The bicycling ABS scenario occurred during non-rain periods when the roads were free of rainwater runoff. All age groups—adults, teens, and children—are likely receptors under this scenario. The breathing height of a very young child (infant), who might be a passenger in a bicycle trailer is significantly closer to the ground than that of an adult, teenager, or non-infant child. To evaluate the effect breathing height has on exposure to LA, samples were collected at two breathing heights: adult while seated on a bicycle and infant passenger in a bicycle trailer. Although the breathing heights of teenagers and non-infant children seated on a bicycle differ from that of adults, the difference was not considered large enough to warrant additional sampling at different breathing heights.

Twenty 60-minute bicycling-scenario events occurred—10 in the spring (wet season) and 10 during the late summer (dry season). A total of 80 ABS air samples (40 high volume and 40 low volume, not including field QC samples) were collected under this scenario. The low volume samples were analyzed only if the high volume samples were overloaded.

**Scenario 6: Recreational Activities at Parks and School Yards**

This scenario involved recreational activities in playgrounds and ball fields at public parks and school yards in OU7. Activities at playgrounds included playing on playground equipment (i.e., swing sets, merry-go-rounds, jungle-gyms, and see-saws) and digging in sand boxes, and were performed at the playgrounds at the Troy elementary school and at Roosevelt Park (Figure 6-2). Recreational activities at the ball fields and parks included baseball, football, soccer, Frisbee, and Frisbee golf. These recreational activities were performed at the Troy elementary school, Roosevelt Park, and the Timber Beast Disk Golf Course (Figure 6-2). These playground and recreational activities were assumed to cause more soil disturbance than do other less vigorous recreational activities.

Composite ABS samples were collected to represent a variety of playground and ball field activities under a combined ABS exposure scenario. The adult breathing height was used for recreational activities at ball fields because adults and teenagers are more likely to participate in these activities. The child breathing height was used for playground activity sampling because children are more likely to play at playgrounds.

Ten one-hour samples representing the child-receptor playground scenario and 10 one-hour samples representing the adult-receptor ball field scenario were collected. One ABS sampling event occurred during the wet spring season and a second event occurred during the dry late summer. A total of 80 ABS air samples (40 high volume and 40 low volume, not including field QC samples) were collected under this scenario. The low volume samples were analyzed only if the high volume samples were overloaded.

#### *6.1.2.2 Soil Sampling*

Two side-by-side 30-point composite soil samples were collected at 0- to 1-inch and 0- to 6-inch depths associated with each residential ABS scenario: (1) yard work in residential yards, (2) gardening in residential gardens, and (3) bicycling and playing on unpaved driveways. Composite samples were collected from across the yard, garden, and driveway areas to represent the entire UA and in accordance with ABS sampling protocols used for the other OUs.

Soil samples were collected and homogenized in accordance with the ABS SAP (Tetra Tech 2011b). To ensure availability of sufficient soil material for potential future analysis, the target mass of each

composite sample was 2.5 kilograms. Each aliquot was inspected for VV in accordance with project-specific SOP CDM-LIBBY-16 (Appendix A), with the following modifications:

- The entire UA was inspected for VV regardless of previous investigation results or removal activities.
- The approximate location and level of any VV was documented on a field sketch that detailed the location of each scenario area. If available, land surveys were used as baselines for these sketches. If a land survey was not available, an aerial photo was used. Soil sampling and vermiculite observations occurred during each sampling event.
- Field personnel documented square footage of each UA where soil samples were collected.
- Field personnel documented the predominant type of ground cover at each UA where soil samples were collected. Ground-cover options included: (1) grass, (2) bare soil, (3) gravel and rock, (4) post-removal grass, (5) post-removal bare soil, (6) and post-removal gravel and rock (see Assessment of Soil Cover, below).
- Field personnel documented a perimeter GPS outline of each UA where air and soil samples were collected.

Before collection of ABS soil samples, in-situ soil moisture was measured at each ABS sample location using a soil moisture meter. Soil moisture was measured at a minimum of 10 locations between 0 and 3 inches bgs. ABS sampling did not occur if the average volumetric water content (VWC) exceeded 30 percent, or if the VWC at any measurement point exceeded 50 percent. The soil moisture result for each area was recorded in the field logbook. On days when ABS activities occurred, meteorological data were downloaded from the local weather station at the Troy DEQ Information Center and archived in electronic format for future reference. The hourly recorded data included:

- Temperature (degrees Fahrenheit)
- Relative humidity (percent)
- Wind speed (mph)
- Wind gusts (mph)
- Wind direction
- Precipitation (inches)

A total of 251 soil samples (including field QC samples) were collected and archived. These samples were retrieved from archive and analyzed in late 2013 and early 2014.

**Assessment of Soil Cover – Photograph Method**

The extent of soil cover was estimated for each sampling event and recorded on the field form and in the field logbook using the method described below. Digital photographs were used for a qualitative estimation of vegetative cover within UAs. Individual sample quadrats (1 square meter) were photographed. The percent of each quadrat covered by vegetation or other material was determined by superimposing a grid frame of 25 equal-sized squares onto the digital picture.

1. Three sample quadrats were randomly located within the UA by tossing the quadrat to three locations from the central area of the UA. The percent soil cover within the UA was the average of the three quadrat estimates.
2. Each quadrat was labeled according to the AD and UA numbers, location, date, and quadrat number (1, 2, or 3). The full quadrat label was written on a board and placed at the top frame of each photograph.
3. A tripod and camera bar was used to position the camera vertically over the approximate center of the quadrat at about 4 feet above ground surface. A digital camera with a zoom lens was used to take each picture. The picture area was zoomed in or out to have the quadrat frame encompass most of the viewing area.

The digital photographs were analyzed as follows:

1. A grid was superimposed onto the digital picture to align exactly with the quadrat to have 25 equal-sized squares.
2. Five grid squares were randomly selected from each of the three quadrats (total of 15) for evaluation of soil cover. The number of grid squares covered by grass, bare soil, gravel or rock, post- removal grass, post-removal bare soil, or post-removal gravel or rock was counted. A grid square with greater than 50 percent of one of the above categories was counted as that category. A grid square with less than 50 percent of any category was assigned the category most represented by materials or vegetation present.
3. The percent soil cover values were summarized for each quadrat and the average cover values calculated for each UA.

## 6.2 SAMPLE ANALYSIS

Outdoor ABS air and soil samples were submitted to the ESAT Sample Coordinator for distribution to a subcontracted analytical laboratory. The analytical methods and requirements used for the outdoor ABS air and soil samples are outlined in the sections below. Copies of applicable project-specific laboratory modifications and SOPs are provided in Appendix A.

### 6.2.1 Air Sample Analysis

ABS air samples were prepared and analyzed by TEM in accordance ISO Method 10312 Annex E and applicable project-specific laboratory modifications including LB-000016, LB-000019, LB-000028, LB-000029, LB-000030, LB-000053, and LB-000066 (Appendix A). Detailed analytical requirements (e.g., counting rules, required sensitivities) are listed in ABS SAP Requirements Summary SABSOU7-0511 (Tetra Tech 2011b).

### 6.2.2 Soil Sample Analysis

Soil samples were archived until late 2013, at which time they were brought out of archive and analyzed using the PLM-VE and PLM-gravimetric methods. Sample preparation was performed in accordance with project-specific SOP ISSI-Libby-01, *Soil Sample Preparation* (Appendix A). The fine fraction was analyzed in accordance with project-specific SOP SRC-Libby-03, *Analysis of Asbestos Fibers in Fine Soil by Polarized Light Microscopy* (Appendix A). The coarse fraction was analyzed in accordance with project-specific SOP SRC-Libby-01, *Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy* (Appendix A).

### 6.2.3 Laboratory Documentation and Reporting

Sample results data was delivered to the EPA in accordance with the current version of the EPA Data Management Plan (EPA 2010).

## 6.3 DATA MANAGEMENT

Management of outdoor ABS field and analytical data is described in, and did not deviate from, the outdoor ABS SAP (Tetra Tech 2011b).

## 6.4 FIELD QUALITY CONTROL SAMPLES

The following field QC samples (including field duplicates) were collected in association the outdoor ABS samples. Field QC samples associated with air samples include lot blanks and field blanks. Each of these QC sample types is described below and summarized in Table 6-1.

**TABLE 6-1: OUTDOOR ACTIVITY-BASED SAMPLING FIELD QUALITY CONTROL SAMPLES**

| Media | QC Sample | Collection Frequency | Analysis Frequency | Analysis Request | Acceptance Criteria |
|---|---|---|---|---|---|
| Air | Lot blank | 1 per case (50 cassettes) | 100% | TEM | ND for all asbestos fibers |
| Air | Field blank | 1 per day | 100% | TEM | ND for all asbestos fibers |
| Soil | Co-located sample | 1 per 20 field samples | Archived [a] | PLM-VE | <30% RPD |

Notes:

[a] Co-located soil samples were collected and archived, then analyzed pending need to analyze the associated field samples.

% Percent
ND Not detected
PLM-VE Polarized light microscopy - visual estimation
RPD Relative percent difference
TEM Transmission electron microscopy

### 6.4.1 Lot Blanks (Air)

Before an air sampling cassette was used, a cassette from each filter lot was randomly selected and submitted for analysis. One lot blank cassette was submitted from each 50-cassette box prior to field use. The lot blanks were analyzed for asbestos fibers by the same method used for field sample analysis. The entire batch of cassettes would have been rejected if an asbestos fiber had been detected on a lot blank.

### 6.4.2 Field Blanks (Air)

One field air blank was collected each day for the duration of the ABS sampling program. The field blanks were analyzed for asbestos fibers by the same TEM method used for field sample analysis. Field blanks were collected by opening the sample cassette package and exposing the cassette to the full range of field efforts including sample handling, car travel, attaching it to the air sample pump for 10 seconds (not turned on), retrieval of the sample cassette, returning it to the office, packaging it, and transporting it to the laboratory.

### 6.4.3 Co-located Samples (Soil)

Co-located samples for ABS soil sampling activities were collected at a rate of 1 per 20 field samples. Co-located samples were collected from areas undergoing sampling during the ABS activities discussed in the previous sections. Individual composite aliquots for each co-located sample were collected from soil adjacent to the original sample location. Co-located samples were collected in accordance with the ABS

SAP (Tetra Tech 2011b). Co-located samples were not sent for analysis but were archived, along with the parent samples, for future analysis.

## 6.5 DATA QUALITY ASSESSMENT

The primary objective of data quality assessment was to determine whether obtained data met project DQOs. Field and analytical data verification procedures for the 2011 ABS air data followed those specified in the outdoor ABS SAP (Tetra Tech 2011b). The sections below summarize verification procedures; for additional details on these procedures, refer to the SAP.

### 6.5.1 Field Data Verification

Outdoor ABS field data were reviewed for completeness, accuracy, and consistency to ensure adequate quality of the outdoor ABS samples. Data recorded on the FSDSs and in logbooks were then checked against the OU7 Scribe Database to ensure consistency of data throughout. Finally, a review of the OU7 Scribe Database occurred after data entry to verify that the data were complete and no errors were present.

### 6.5.2 Analytical Data Verification

Outdoor ABS air samples were analyzed using TEM per ISO Method 10312, modified in accordance with project-specific SOPs and laboratory modifications described in the SAP (Tetra Tech 2011b).

Tetra Tech conducted data review and data entry verification of the 2011 outdoor ABS data in basic accordance with project-specific SOP EPA-LIBBY-09 (Appendix A). Tetra Tech followed the data review and data entry verification procedures outlined in this SOP with minor deviations for OU7. Noteworthy deviations are explained in the subsections below.

Tetra Tech's data review and data entry verification process entailed three steps: (1) selection of data records for review and verification, (2) review of the original laboratory bench sheets, and (3) verification of transfer of results from the bench sheets onto the EDDs. Tetra Tech also reviewed field QC sample results for adherence to minimum frequency requirements and procedures and QC limits specified in the SAP (Tetra Tech 2011b). The data review and data entry verification process are described in the following subsections.

**Selection of TEM Records for Review**

SOP EPA-LIBBY-09 specifies review and verification of a minimum of 10 percent of the sample records. Tetra Tech deviated from this minimum requirement and reviewed 20 percent of the outdoor ABS air sample records, in part because of the incidence of errors (e.g., data formatting and structural issues related to database structural changes).

SOP EPA-LIBBY-09 also specifies a query of the Scribe Database to select specific percentages of analytical records to insure that a representative number of records from each laboratory and analyst, as well as detected and non-detected results, are verified. The process for selecting analytical data records to verify conformed to SOP EPA-LIBBY-09 (Appendix A).

**Consistency Review of Laboratory Bench Sheets**

Tetra Tech inspected the information recorded on the original hand-written laboratory bench sheets in accordance with the consistency review of laboratory bench sheets procedure outlined in Section 5 of SOP EPA-LIBBY-09 (Appendix A), with minor OU7-related modifications. The bench sheets were reviewed to identify any data omissions, apparent inconsistencies, or potential errors in structure. The objective was to assess whether the raw structure data were recorded in accordance with ISO 10312 counting rules (as modified by applicable project-specific modifications introduced above).

**Corrective Action –** Tetra Tech summarized all apparent inconsistencies, omissions, and suspected errors, and provided these to the ESAT. The ESAT then forwarded them to the appropriate laboratories for response.

**Verification of Data Transfer from Bench Sheet to Database**

To ensure that data from laboratory bench sheets were transferred, through the EDDs, into the OU7 Scribe Database without error or omission, Tetra Tech compared selected analysis-specific information in the laboratory bench sheets to that in the Scribe Database. Tetra Tech followed the verification of data transfer procedure outlined in Section 6.0 of SOP EPA-LIBBY-09 (Appendix A), with minor modifications for OU7. The bench sheets include the laboratory COC form, sample check-in form, preparation log, and hand-written data record sheets. This process compared analysis-specific information in the database to the original laboratory job documentation (e.g., internal laboratory COC, preparation logs, bench sheets, etc.). The process included verifying (by recalculation) the reported air

sensitivities for amphibole and chrysotile, the area analyzed, and, for indirect preparations, the indirect preparation dilution factor. Using the bench sheets, Tetra Tech also recounted the countable LA structures across all grid openings evaluated and compared this number (and the calculated concentrations) to the total number of LA structures indicated in the database.

**Corrective Action –** Tetra Tech summarized all apparent inconsistencies, omissions, and suspected errors, and provided these to the ESAT. The ESAT then forwarded them to the appropriate laboratories for response.

**Review of Field QC Sample Results**

Tetra Tech reviewed field QC sample (co-located and field blank sample) results, and used the findings to refine sampling procedures where applicable. For the co-located field samples, each co-located sample pair was compared using the Poisson rate test, included in project-specific laboratory modification LB-000029 (Appendix A), to determine whether the results differed statistically from one another at the 95 percent confidence level. The Poisson rate test is suitable for this analysis because fiber counts on TEM grids are considered independent and random.

**Corrective Action** –For field co-located samples, Tetra Tech reviewed the Poisson rate test results and investigated the basis for any significant statistical differences and need for any appropriate corrective actions (modification of sampling protocol). Poisson rate test results are considered good when the field duplicate samples are similar at the 95 percent confidence interval. Test results in the 90 to 95 percent confidence interval range are considered acceptable, and test results below the 90 percent confidence interval are considered poor for similarity.

For field blanks, Tetra Tech reviewed the results for adherence to the QC limits specified in SAP (Tetra Tech 2011b). All field blank results were within QC limits.

### 6.5.3 Statement on Data Quality

The objective of the outdoor ABS investigation was to obtain the data necessary to complete an HHRA. The air data needed to complete the HHRA—identified in the HHRA work plan for OU7 (Tetra Tech 2011a)—include ABS data for outdoor air within the two residential property categories and within community areas. The outdoor ABS DQOs are presented in the SAP (Tetra Tech 2011b).

To address outdoor ABS objectives, Tetra Tech collected and analyzed outdoor ABS air samples for LA. The results were used in the HHRA. Tetra Tech also collected soil samples, which were originally archived, but were retrieved from archive and analyzed beginning in late 2013. To ensure accuracy and reliability, outdoor ABS field data and analytical results were reviewed and verified in accordance with the procedures specified in the SAP (Tetra Tech 2011b). Data quality appeared to be adequate for supporting exposure scenario and risk assessment calculations.

## 6.6 DEVIATIONS FROM THE OUTDOOR ACTIVITY-BASED SAMPLING AND ANALYSIS PLAN

Ten residential gardens at post-removal residential properties were planned for sampling under the residential garden scenario. However, only eight residential gardens met this criterion and were sampled during the wet season ABS event in May 2011. For the August 2011 dry season ABS event, nine residential gardens that met the criterion were identified and sampled. All other ABS exposure scenarios and locations, and the number of samples collected, accorded with specifications in Table 4-1 in the SAP (Tetra Tech 2011b). The reduction in the number of properties for this sampling did not affect the results.

# 7.0 INDOOR ACTIVITY-BASED SAMPLING

This section summarizes objectives, methods, and procedures of the indoor ABS sampling program. Indoor ABS sampling program began in September 2012 and ended in March 2013. Refer to the *Indoor Activity-Based Sampling, Sampling and Analysis Plan for Operable Unit 7 of the Libby Asbestos Superfund Site* (Tetra Tech 2012a) for in-depth details.

## 7.1 SAMPLING OBJECTIVES AND INVESTIGATION METHODS

The primary objective of the indoor ABS sampling program at OU7 was to obtain the data needed to complete the HHRA. The data needed to complete the HHRA were identified in the HHRA work plan for OU7 (Tetra Tech 2011a) and include ABS data for indoor air from both non-removal and post-removal residential properties.

### 7.1.1 Pre-Sampling Planning

Residential sampling locations were selected by querying the OU7 Scribe Database. To the extent possible, residential locations were selected to represent both non-removal and post-removal properties, as well as a cross-section of properties across OU7. Property owners were contacted to determine their willingness to participate prior to the start of ABS. This process continued until the desired number of sampling locations had been selected.

Before mobilization to each property where signed access agreements had been obtained, each property owner or tenant was contacted by phone to determine a specific time for the sampling.

Prior to sampling, each residential structure was assessed to determine the number of rooms on each "living floor" of the main structure where sampling would occur. A "living floor" is defined as a regularly occupied area, and excludes unfinished basements and attics. The total sampling time for each period (passive and active) was divided evenly among the all the rooms where routine living activities occur. Areas such as bathrooms and closets were not sampled.

### 7.1.2 Investigation Methods and Activities

Prior to sampling, the extent of porous and non-porous surface cover in each room was estimated and recorded on the field forms and in the field logbooks. Digital photographs were used to document porous and non-porous surfaces in each room. One photo was taken in each room.

Each room was visually inspected to document the presence or absence of visible vermiculite and to note the relative level of dust. Dust levels in each room were categorized as low, medium, or high and noted in the field form and logbook.

#### *7.1.2.1 Indoor ABS Sampling Scenarios*

The following sections provide a general summary of the indoor ABS scenarios evaluated with additional information related to receptors, duration of sampling, number of samples, and temporal considerations.

**Receptors and Age Groups** - Adult residents are the most likely receptors and age group for the active indoor ABS scenarios, so samples were collected at the adult breathing height during all active ABS sampling. Although teenagers may participate in housecleaning work, the breathing height of teenagers does not differ enough from that of adults to warrant collection of additional samples for teenager breathing height. Similarly, child exposures during active ABS scenarios were represented by activities that included simulated play with children and pets, such as rolling, crawling, and sitting on the floor surfaces. All air samples were collected as the adult actor; however, multiple breathing heights were involved with the up and down movements of the actor during the simulated play tasks.

Breathing heights of adults, teens, and most children during the passive ABS scenarios would not differ significantly, so only samples from an adult breathing zone height were collected during the passive scenarios.

**Duration of Sampling** – Each of the two sampling scenarios consisted of four hours of continuous sampling on a single filter cartridge with specific time spent in each room determined by the number of rooms in the house.

**Sample Compositing** - Because risk is related to long-term average exposure to all residential activities, indoor ABS samples were composited to represent a variety of indoor activities. These activities fall into two general categories: (1) active indoor residential activities, and (2) passive indoor residential activities.

**ABS Scenario 1: Active Resident**

The active resident sampling scenario involved a variety of dust-generating activities including walking, continuously standing up and sitting down on upholstered chairs and/or cushions, sweeping non-carpeted floors, dusting, simulated play with children and pets, and vacuuming carpeted floors. Brooms and High Efficiency Particulate Air (HEPA) vacuums used during active disturbance activities were provided by the home owners as agreed upon in the access agreements. If HEPA vacuums were used by the owners, the filters were removed prior to sampling to simulate worst case exposures. When there was carpeted and non-carpeted flooring in the same room, sweeping or vacuuming was conducted on both flooring types.

The various active resident sampling activities were conducted for equal periods of time in each room. Table 7-1 depicts the execution of a hypothetical active resident sampling effort (assuming no upholstered furniture is in rooms 2 or 5):

**TABLE 7-1: HYPOTHETICAL ACTIVE RESIDENT SAMPLING CHRONOLOGY**

| Location | Minutes Elapsed Since | Activity | Duration of Activity (minutes) |
|---|---|---|---|
| Room 1 | 0 to 13.0 | Walk on flooring | 13 |
| | 13.0 to 26.0 | Sit on upholstered furniture | 13 |
| | 26.0 to 39.0 | Sweep non-carpeted flooring | 13 |
| Room 2 | 39.0 to 39.5 | Transition to room 2 | 0.5 |
| | 39.5 to 59.5 | Walk on flooring | 20 |
| | 59.5 to 79.5 | Vacuum carpeted flooring | 20 |
| Room 3 | 79.5 to 80.0 | Transition to room 3 | 0.5 |
| | 80.0 to 93.0 | Walk on flooring | 13 |
| | 93.0 to 106.0 | Sit on upholstered furniture | 13 |
| | 106.0 to 119.0 | Sweep non-carpeted flooring | 13 |
| Room 4 | 119.0 to 119.5 | Transition to room 4 | 0.5 |
| | 119.5 to 132.5 | Walk on flooring | 13 |
| | 132.5 to 145.5 | Sit on upholstered furniture | 13 |
| | 145.5 to 158.5 | Vacuum carpeted flooring | 13 |
| Room 5 | 158.5 to 159.0 | Transition to room 5 | 0.5 |
| | 159.0 to 179.0 | Walk on flooring in room 5 | 20 |
| | 179.0 to 199.0 | Vacuum carpeted flooring | 20 |
| Room 6 | 199.0 to 199.5 | Transition to room 6 | 0.5 |
| | 199.5 to 212.5 | Walk on flooring in room 6 | 13 |
| | 212.5 to 225.5 | Sit on upholstered furniture | 13 |
| | 225.5 to 238.5 | Sweep carpeted flooring | 13 |
| **Total Active ABS Scenario Time (minutes)** | | | **238.5** |

**ABS Scenario 2: Passive Resident**

Passive resident sampling activities were mostly sedentary. Movement was restricted to walking between rooms and sitting on upholstered chairs or cushions. While seated the actor read, watched television, played video games, or completed paperwork.

The actor transitioned to each room when required, walked around the perimeter of the room once upon initial entry, and then remained seated for the duration of the time required in the room. Table 7-2 shows the execution of a hypothetical passive resident sampling effort:

**TABLE 7-2: HYPOTHETICAL PASSIVE RESIDENT SAMPLING CHRONOLOGY**

| Location | Minutes Elapsed Since Start | Activity | Total Time of Activity in Location (minutes) |
|---|---|---|---|
| Room 1 | 0 to 0.5 | Walk around perimeter of room 1 | 0.5 |
| | 0.5 to 40.5 | Sit in room | 40 |
| Room 2 | 40.5 to 41.0 | Transition to room 2 and walk around perimeter | 0.5 |
| | 41.0 to 81.0 | Sit in room | 40 |
| Room 3 | 81.0 to 81.5 | Transition to room 3 and walk around perimeter | 0.5 |
| | 81.5 to 101.5 | Sit in room | 40 |
| Room 4 | 101.5 to 102.0 | Transition to room 4 and walk around perimeter | 0.5 |
| | 102.0 to 142.0 | Sit in room | 40 |
| Room 5 | 142.0 to 142.5 | Transition to room 5 and walk around perimeter | 0.5 |
| | 142.5 to 182.5 | Sit in room | 40 |
| Room 6 | 182.5 to 222.5 | Transition to room 6 and walk around perimeter | 0.5 |
| | 222.5 to 242.5 | Sit in room | 40 |
| | | **Total Passive ABS Scenario Time (minutes)** | **243** |

### *7.1.2.2 Air Sample Collection*

Indoor ABS air sampling occurred over a 4-hour period for each scenario and was repeated two times at each sample location (September 2012 [dry season] and March 2013 [wet season]). Each time-interval was subdivided by the number of representative activities as detailed in Table 7-3. Indoor ABS air samples were collected by actors performing activities in accordance with the active or passive scenarios. At each location, one actor engaged in the prescribed activities in each of the scenario areas. The order of activities began with the scenario of least disturbance (passive) and finished with the active scenario that likely created the most measurable disturbance.

All indoor ABS air samples were collected using cassettes that contained a 25-millimeter diameter MCE filter with a pore size of 0.8 µm. One actor conducted the scripted activities and wore an air sample pump (calibrated at five liters per minute [L/min] for the first two days of sampling, then reduced to 4

L/min [to reduce filter overloading] for all subsequent indoor ABS samples) during two scenarios. Each air sample was collected using battery-powered sampling pumps capable of operating for extended periods at 4 L/min. The specific model used was the SKC AirChek 5000 personal air sampling pump. The pump flow rate was adjusted to 4 L/min to obtain sample volumes of at least 1,000 liters over the 4-hour sample period.

The monitoring cassette was affixed to the shoulder of the actor performing adult scenario activities so that the cassette was in the proper breathing zone visualized as a hemisphere approximately six to nine inches around an individual's face. The top cover from the cowl extension on the sampling cassette was removed (i.e., open-face) and the cassette was oriented face down. The actors monitored the cassette throughout the scenario to ensure it remained directed toward the activity and was free of obstructions. Table 7-3 summarizes the two indoor ABS scenarios and associated samples.

**TABLE 7-3: EXPOSURE SCENARIOS, LOCATIONS, ACTIVITIES, AND NUMBER OF SAMPLES FORINDOOR ACTIVITY-BASED SAMPLING**

| ABS Scenario | | Location | Property Category | Property Sub-Category | Receptor | Age Group | Sample Height | Activity | Activity Time | Samples per Scenario and Category | Events per Location (a) | Total Samples per Scenario-Property Category |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| No. | Description | | | | | | | | | | | |
| 1 | Active Resident | Inside Residence | Removal Not Required | No V V / ND | Resident | Adult | Breathing Zone for Adult | Vacuuming / Dusting / Cleaning / Walking From Room to Room | 250 Minutes of Total Combined Activities | 5 | 2 | 10 |
| | | | | V V / ND | | | | | | 5 | 2 | 10 |
| | | | | No V V / Trace (0.2 to <1) | | | | | | 5 | 2 | 10 |
| | | | | V V / Trace (0.2 to <1) | | | | | | 5 | 2 | 10 |
| 2 | Active Resident | Inside Residence | Removal Completed | Interior Removal Only | Resident | Adult | Breathing Zone for Adult | Vacuuming / Dusting / Cleaning / Walking From Room to Room | 250 Minutes of Total Combined Activities | 7 | 2 | 14 |
| | | | | Exterior Removal Only | | | | | | 7 | 2 | 14 |
| | | | | Interior / Exterior Combination Removal | | | | | | 6 | 2 | 12 |
| 3 | Passive Resident | Inside Residence | Removal Not Required | No V V / ND | Resident | Adult | Breathing Zone for Adult | Sitting / Reading / Watching Television/ Writing | 250 Minutes of Total Combined Activities | 5 | 2 | 10 |
| | | | | V V / ND | | | | | | 5 | 2 | 10 |
| | | | | No V V / Trace (0.2 to <1) | | | | | | 5 | 2 | 10 |
| | | | | V V / Trace (0.2 to <1) | | | | | | 5 | 2 | 10 |
| 4 | Passive Resident | Inside Residence | Removal Completed | Interior Removal Only | Resident | Adult | Breathing Zone for Adult | Sitting / Reading / Watching Television/ Writing | 250 Minutes of Total Combined Activities | 7 | 2 | 14 |
| | | | | Exterior Removal Only | | | | | | 7 | 2 | 14 |
| | | | | Interior / Exterior Combination Removal | | | | | | 6 | 2 | 12 |

Notes

a One wet season and one dry season event

VV Visible vermiculite

ND Non-detect

< Less than

#### *7.1.2.3 Indoor ABS Activities Documentation*

Logbooks and field forms were maintained for each residence sampled. Sample information and visual inspection results were recorded on sketches and in the logbook as needed. Sketches included a basic home floor plan, visual inspection locations and findings, and a summary of surface types and materials in the rooms.

## 7.2 FIELD QUALITY CONTROL SAMPLES

Field QC samples were collected in association with air samples and included lot blanks and field blanks. These are described below and summarized in Table 7-4.

### 7.2.1 Lot Blanks

Before any air sampling cassette was used, a cassette from each filter lot was randomly selected and submitted for analysis. One lot blank cassette was submitted from each 50-cassette box prior to field use. The lot blanks were analyzed for asbestos fibers by the same method used for field sample analysis. The entire batch of cassettes would be rejected if any asbestos fiber is detected on a lot blank.

### 7.2.2 Field Blanks

One field air blank was collected at each sampling location each day for the duration of this sampling program. The field blanks were analyzed for asbestos fibers by the same TEM method used for field sample analysis. The field blanks were collected by opening the sample cassette package and exposing the cassette to the full range of field efforts, including sample handling, car travel, attaching to the air sample pump for ten seconds (not turned on), retrieving the sample cassette, returning to the office, packaging, and transporting to the laboratory.

**TABLE 7-4: INDOOR ACTIVITY-BASED SAMPLING FIELD QUALITY CONTROL SAMPLES**

| Sample Matrix | Sample Type | Collection Frequency | Analysis Frequency | Analysis | Acceptance Criteria |
|---|---|---|---|---|---|
| Air | Lot Blank | 1 per case (50 cassettes) | 100% | Asbestos by TEM | ND for all asbestos fibers |
| Air | Field Blank | 1 per day | 100% | Asbestos by TEM | ND for all asbestos fibers |

Notes:
% Percent
TEM Transmission Electron Microscopy
ND Non detect

## 7.3 GENERAL PROCESSES

This section describes the general field processes used to support the indoor ABS sampling effort and includes references to project-specific SOPs where applicable. Field equipment maintenance was conducted and documented on a routine basis.

### 7.3.1 Equipment and Personnel Decontamination

Air monitoring pumps, tubing and calibration equipment were wet wiped with moistened, disposable, paper towels before and after each ABS sampling. Personnel decontamination procedures, including requirements for decontamination zones, are described in Section 10.1 of the HASP (Appendix A of the SAP [Tetra Tech 2012a]). PPE consisted of disposable gloves, disposable protective outerwear, work boots, disposable boot covers, and respirators. The respirators were cleaned and decontaminated as described in Section 10.2.1 of the HASP ((Appendix A of the SAP [Tetra Tech 2012a]).

### 7.3.2 Investigation-Derived Waste

IDW included used wet wipes, paper towels, disposable gloves, used respirator cartridges, used plastic tubing, disposable protective outerwear, plastic coverings, and other minimal waste. Because it was possible that these IDW materials could be contaminated with asbestos, all IDW was double-bagged in appropriate asbestos bags, labeled with asbestos labels, and stored in an approved containment area at the DEQ Troy Information Center office until it was properly disposed of at an approved landfill. Non-sampling waste from the ABS field teams, such as food containers and waste paper, was separately bagged and properly disposed of as solid waste.

### 7.3.3 Recordkeeping and Sample Chain of Custody

At the end of each day the ABS field teams returned to the DEQ Troy Information Center office to download and transfer data recorded on the OU7 indoor ABS forms. The field teams transferred the air samples and QC samples, provided copies of the appropriate logbook pages to the Field Sample Coordinator (or the coordinator's designee), and downloaded digital photographs. Photographs were electronically labeled and entered into the OU7 Document Management Archive (MARCH). MARCH is a Microsoft Access database used to manage electronic documents and associated metadata in accordance with EPA Appendix C requirements (EPA 2013a). Individual photographs were not routinely printed. Data from the field forms were downloaded or entered on a daily basis into a local

field Scribe Database. Once all data were processed, the Scribe project was informally published to Scribe.NET. The field database was incorporated into the primary OU7 Scribe Database, LibbyTTOU7Field, and was formally published to Scribe.NET.

An individual file (paper and electronic) was maintained for each indoor ABS property and building where ABS was done. Originals of all field forms were kept in each individual property file in the DEQ Troy Information Center office for the duration of the project. PDF copies of all field forms and appropriate logbook pages, and digital photographs were stored in each individual electronic property file. A backup electronic copy of the files was stored away from the DEQ Troy Information Center office, and was updated periodically for the duration of the sampling and reporting phases of the project. Copies of all QA/QC records, forms, and field logbooks were available to the DEQ, EPA, or to the Troy property owners at any time during the ABS project.

After the OU7 ABS form electronic information and data were entered in the local field Scribe Database, the Field Sample Coordinator checked all property, building, and sample identification numbers for accuracy. The Field Sample Coordinator printed a hard copy of the COC form and stored it with the associated samples. The COC form was transferred to the ESAT Laboratory Coordinator with the samples.

Until samples had been transferred to the ESAT Laboratory Coordinator, they were securely held by Tetra Tech. Samples were stored in storage bins in locked vehicles or in a secured (locked) area of the DEQ Troy Information Center. ABS samples, including QC samples, were regularly transferred to the ESAT Laboratory Coordinator. The ESAT Laboratory Coordinator provided Tetra Tech with a copy of the signed COC form. The ESAT Laboratory Coordinator transferred the samples to the on-site laboratory for preparation, the off-site laboratory for analysis, or to the storage archive.

### 7.3.4 Field Logbooks

Documentation of field activities was recorded in field logbooks maintained specifically for this sampling program. Logbooks are controlled documentation (i.e., sequentially numbered) and maintained by DEQ and EPA.

The logbook is an accounting of activities at the site and noted problems or deviations from the governing plans and observations relating to the sampling and analysis program. A new logbook page

was started for each indoor ABS property visited. The header information included the address. When closing out a logbook page with lineout and signature, the author printed his/her name underneath the signature. Original logbooks were maintained in the DEQ Troy Information Center.

### 7.3.5 Sample Labeling and Identification

A unique alphanumeric code, or sample identification number, identifies each sample. The coding system provided a tracking record to allow retrieval of information about a particular sample and to ensure that each sample was uniquely identified. Sample identifications are sequential and do not include information about any particular ABS scenario or building. Sample identifications correlate with sample location identifications that are identified in the field logbooks.

The sample labeling scheme followed the format:

TB-XXXXX

where:

TB identifies that a sample was collected in accordance with the indoor ABS SAP and XXXXX represents a unique 5-digit sample number.

Preprinted adhesive sample labels were signed out to sampling personnel by the sample database manager. The labels were controlled to prevent duplication in assigning sample IDs. The labels were affixed to the inner and outer sample bags for ABS air samples.

### 7.3.6 Photographic Documentation

Photographs were taken with a digital camera at sampling locations and during each sampling event. Photos generally included an overview of the room, close up shots of flooring materials, equipment, actors, and any other notable photos to support later health risk assessments that the sampler might identify. A description of each photograph was recorded in the field logbook in accordance with photographic log protocol described in Appendix I of TAPE work plan (Tetra Tech 2007). A list of photographs in the field logbook clearly stated where each photograph was taken (e.g., ABS scenario or ABS room). Electronic photograph files were saved each day to a project-designated computer at the DEQ Troy Information Center and assigned appropriate metadata in MARCH so that photographs for a particular ABS property or scenario can easily be retrieved.

## 7.4 AIR SAMPLE ANALYSIS

Indoor ABS air samples were submitted to the ESAT Sample Coordinator for distribution to a subcontracted analytical laboratory. ABS air samples were prepared and analyzed by TEM in accordance the International ISO Method 10312, Annex E and applicable project-specific laboratory modifications including LB-000016, LB-000019, LB-000028, LB-000029, LB-000030, LB-000053, and LB-000066 (Appendix A).

All asbestos structures (including LA and all other asbestos types) that have appropriate diffraction patterns and EDS spectra, and length greater than 5 µm, a width greater than or equal to 0.25, and an aspect ratio greater than or equal to 3:1, were counted. Additional details regarding analytical requirements (e.g., counting rules, required sensitivities) are listed in indoor ABS SAP Requirements Summary No. OU7_TB_RI_102212 (Tetra Tech 2012a). Copies of project-specific laboratory modifications and SOPs are provided in Appendix A.

## 7.5 LABORATORY DOCUMENTATION AND REPORTING

Sample results data was delivered to the EPA in accordance with applicable version of the EPA Data Management Plan (EPA 2012c).

## 7.6 DATA MANAGEMENT

Data management was under the supervision of the Tetra Tech Database Manager in the DEQ Troy Information Center. Field crews generated field data on paper copies and digital photographs. All field data was managed according to EPA reporting requirements specified in the EPA Data Management Plan (EPA 2012c).

These reporting requirements were developed to help satisfy EPA's removal objectives at the Libby Asbestos Superfund Site. The reporting requirements guide data collection processes and data reporting procedures for spatial information, tabular data, and documents (EPA 2012c).

### 7.6.1 TABULAR DATA

The Tetra Tech Database Manager was required to format and submit all tabular data in accordance with EPA reporting requirements (EPA 2012c). Operational electronic data was QC reviewed, entered into a local field Scribe database, and informally published to Scribe.Net within one day of

data collection; and, after data auditor checks, formally published to Scribe.NET as part of the primary OU7 Scribe Database, LibbyTTOU7Field. This ensured that EPA had consistent and up-to-date information.

### 7.6.2 DOCUMENTS AND RECORDS

The Tetra Tech Database Manager was required to format and submit all operations documents and records in accordance with EPA reporting requirements (EPA 2012c). Hard copy documents were stored in file cabinets at the DEQ Troy Information Center. Electronic copies were barcoded and assigned the appropriate metadata in the MARCH database. When requested, these records and associated metadata were delivered to EPA.

## 7.7 QUALITY CONTROL PROCEDURES

The QC objectives, internal QC checks, and audits completed for the project are described in this section.

### 7.7.1 QUALITY CONTROL OBJECTIVES

The QC objectives of the OU7 Indoor ABS project were to obtain 100 percent usable and accurate data. This was achieved through inspection and sampling using standardized PDA data entry procedures, auditing field operations, observing COC procedures, analyzing field quality control samples and laboratory quality control samples, and analytical data review, verification, and validation. The DQOs are described in detail in the SAP (Tetra Tech 2012a).

### 7.7.2 DATA VERIFICATION AND VALIDATION

The data verification process is described in Section 7.7.2.1 and the data validation process is described in Section 7.7.2.2.

#### *7.7.2.1 Data Verification*

Tetra Tech's Analytical Coordinator conducted data verification of all sample analytical data generated. One hundred percent of the field sample data underwent verification.

Data verification included checking that results were transferred correctly from the original hand-written field and analytical laboratory documentation to the project database. The goal of data

verification was to identify and correct data reporting errors. Data verification of reported analytical results began with automated QC checks that were built into the project-specific EDDs.

In addition to these automated checks, more detailed manual data verification efforts were performed for 100 percent of the all analytical results uploaded to the project database. This data verification process followed project-specific SOP EPA-LIBBY-09, *TEM Data Review and Data Entry Verification* and EPA-LIBBY-11, *FSDS Data Review and Data Entry Verification* (Appendix A), developed to ensure TEM and field sample information in the project database was accurate and reliable. If issues were identified during the data verification, the frequency of verification were increased as appropriate.

Tetra Tech's Analytical Coordinator documented issues identified on a verification tracking spreadsheet and provide the spreadsheet to the ESAT project data manager for resolution. It was the responsibility of the Tetra Tech Database Manager to coordinate with the Field Team Leader and/or Laboratory Coordinator to resolve any project database corrections and address any recommended field or laboratory procedural charges form the data verifier. The Tetra Tech Database Manager was also responsible for electronically tracking in the project database which data had been verified and who performed the verification.

#### *7.7.2.2 Data Validation*

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

Data validation was performed in basic accordance with the *National Functional Guidelines for Asbestos Data Review* (EPA 2011) and the Site-wide Quality Assurance Reference Document for the Libby Asbestos Superfund Site Libby, Montana (QARD) (USACE 2013) and includes an assessment of the following:

- Internal and external field audit/surveillance reports
- Field record of modifications (ROM)
- Field QC sample results

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

### 7.7.3 STATEMENT ON DATA QUALITY

The objective of the indoor ABS investigation was to obtain data necessary to complete an HHRA. The air data needed to complete the HHRA—identified in the HHRA work plan for OU7 (Tetra Tech 2011a)—include ABS data for indoor air. To fulfill the indoor ABS sample needs, Tetra Tech prepared the *Indoor Activity-Based Sampling, Sampling and Analysis Plan for Operable Unit 7 of the Libby Asbestos Superfund Site* (Tetra Tech 2012a), which describes procedures and methodologies for conducting indoor air ABS.

To address indoor ABS objectives, Tetra Tech collected and analyzed passive and active scenario indoor ABS air samples for LA. The results were used in the HHRA. To ensure accuracy and reliability, indoor ABS field data and analytical results were reviewed and verified in accordance with the procedures specified in the SAP (Tetra Tech 2012a). Data quality appears to be adequate for supporting exposure scenario and risk assessment calculations.

### 7.7.4 AUDITS, CORRECTIVE ACTIONS, AND QUALITY ASSURANCE REPORTS

Field audits were an integral part of Tetra Tech's field operations for the duration of the project. Field audits and corrective actions were the responsibility of the DEQ, EPA and the Tetra Tech QA Manager. The field audit forms were kept in the DEQ Troy Information Center for the duration of the project.

#### *7.7.4.1 Field Inspections and Sampling Procedures Audits*

The Tetra Tech QA Manager was responsible for audits of field activities and sampling procedures. Audits were conducted every two weeks. Audits consisted of the QA Manager or his designee

attending a sampling event and observing the field teams' activities. The field teams were not warned of the audit. The auditor compared the field teams' activities with the protocols in SAP. After the audit, the auditor provided the completed audit form to the DEQ and EPA project managers.

#### *7.7.4.2 Corrective Action Procedures*

The QA auditor provided immediate verbal feedback to the ABS field team, as needed, to ensure that discrepancies were corrected as quickly as possible. The Field Team Leader and QA Manager reviewed the report with the ABS field team within 48 hours of the audit to correct any deviations or deficiencies.

If any deviations or deficiencies were noted, the field team was audited again within one week of the original audit to ensure that any deficiencies have been corrected. If a field team member was rotated off the project after deviations or deficiencies were noted, the field team members were audited again within one week of returning to Troy.

## 7.8 DEVIATIONS FROM THE INDOOR ACTIVITY-BASED SAMPLING AND ANALYSIS PLAN

Initially, the air sample flow rate was set at 5.0 liters per minute (LPM) to obtain an air volume of 1,200 liters during each indoor ABS scenario. However, during the first two days of field work, quick turnaround analytical results revealed overloaded sample filters. Tetra Tech then modified the sample protocol by reducing the flow rate to 4.0 LPM, resulting in an air volume of 1,000 liters. The reduced flow rate and total air volume provided better analytical results.

# 8.0 BACKGROUND SOIL STUDY

It has been theorized that LA from Vermiculite Mountain may have been dispersed by glacial processes during the Pleistocene period about 16,000 years ago (Langer et al. 2010), and that such dispersal resulted in LA in naturally-occurring soils in the vicinity of the Libby Asbestos Superfund Site, including OU7 and the surrounding area. Therefore, a background soil study was conducted in OU7 in 2012 to evaluate naturally-occurring, or background, concentrations and distribution of LA and potentially other forms of asbestos in Troy Valley soils, and to characterize the potential for LA fibers to be released to the environment from soils and country rock not associated with former mining operations at OU3, the former W.R. Grace vermiculite mine. Establishing background levels of LA and its unique mineralogical characteristics in soils in and around OU7 was critical to establishing baseline LA conditions for OU7.

The background soil study involved collecting soil samples from areas of naturally-deposited glacial sediments in and around OU7, as well as air sampling to assess the potential for activity-based exposure to background LA. The complete background soil study report appears as Attachment 2 to this report. The results of the study are summarized in Section 10.6.

# 9.0 ADDITIONAL REMOVAL ACTIVITIES

The following sections summarize removal activities done in response to the discovery of VV and/or LA source materials along Callahan Creek, on a portion of the BNSF ROW, and at OU7 parcels with considerable vermiculite contamination.

## 9.1 CALLAHAN CREEK REMEDIATION

As part of the overall investigations for the Libby Asbestos Superfund Site, CDM performed a visual site inspection on a one-mile stretch of Callahan Creek on April 30, 2008, for the presence of LA-containing syenite riprap (CDM 2008a). Callahan Creek is located south of downtown Troy and flows from west-southwest to east-northeast into the Kootenai River (Figure 1-3). The upstream boundary of the inspection was the western residential property on Callahan Creek Cut Off Road/St. Regis Haul Road and the downstream boundary was the creek outlet into the Kootenai River. Syenite was observed on a 100-foot stretch on the north bank of the creek immediately west of Highway 2. To remediate the potential impacts of the syenite, 330 cubic yards of contaminated soil and riprap were removed from the bank of the creek. After the removal, three inches of shotcrete was applied to the bank to encapsulate the residual syenite and to prevent erosion. In addition, thirty inches of Class III riprap was then placed over the shotcrete to further stabilize the bank from erosion. The removal began on August 25, 2008 and was finished by November 20, 2008 (CDM 2008a).

## 9.2 BURLINGTON NORTHERN SANTA FE RAILROAD RIGHT-OF-WAY REMOVAL

During a TAPE inspection of an OU7 parcel adjacent to the BNSF ROW on June 4, 2008, Tetra Tech observed a stockpile and concrete foundation on the BNSF ROW containing VV. Although the BNSF ROW is part of OU6, the presence of VV was deemed critical due to the amount of contamination and evidence of frequent visitation to the site. EPA recommended that the foundation be sealed to prevent public access and that the stockpile be removed. Tetra Tech received approval from BNSF on June 5, 2008 to remove the stockpile from the property. Removal activities were performed in June 2008, with a total of 17 bags of asbestos-contaminated material being removed from the site. BNSF personnel sealed the concrete foundation on July 3, 2008. Tetra Tech documented the removal of the contaminated stockpile in an ERS report.

## 9.3 ENVIRONMENTAL RESOURCE SPECIALIST ACTIVITIES

In some instances where a considerable amount of vermiculite was found during a TAPE or other inspection activity, and it was determined that the property required immediate attention to reduce exposure from material continually leaking into the living space and/or from exposure to heavily contaminated soil, an ERS response action was conducted to temporarily isolate the potential LA-containing material from residents or workers.

A total of 43 ERS response actions were conducted in OU7 between 2007 and 2013. A note indicating that an ERS response action had been conducted at the property was entered into the OU7 Scribe Database.

# 10.0 FIELD RESULTS AND NATURE AND EXTENT OF CONTAMINATION

This section summarizes all TAPE, RDI, AAS, ABS, and background soil study implementation and results at OU7. The purpose of these efforts was to gather sufficient and reliable data to characterize the nature and extent of LA in OU7 and assess the associated human health risks. In addition, TAPE data were used to identify parcels meeting EPA removal action criteria (Attachment 1).

TAPE implementation and findings are summarized in Section 10.1. Section 10.2 summarizes TAPE results, the process of selecting parcels for removal action, and the resulting removal actions and confirmation sampling. AAS implementation and findings are summarized in Section 10.3. Outdoor ABS implementation and findings are summarized in Section 10.4. Indoor ABS implementation and findings are summarized in Section 10.5. Section 10.6 briefly describes the background soil study and refers to the report, which is included as Attachment 2.

## 10.1 TROY ASBESTOS PROPERTY EVALUATION IMPLEMENTATION

The following subsections present TAPE implementation summaries by year. Between 2007 and 2013, a total of 1,305 TAPE inspections were conducted. This includes 81 parcels outside the OU7 boundary as well as 306 road and alley parcels. The inspections outside the OU7 boundary were performed on a case-by-case basis due to unique circumstances such as owner transport of buildings or vermiculite from Libby to the parcel, or according to whether the owner had knowledge of such an event. All road and alley parcels were inspected during the 2009 field season, and the results are discussed separately.

Between 2007 and 2013, a total of 43 ERS actions were completed, including in and around OU7. ERS actions are summarized below, according to the year in which they were conducted.

As discussed in Section 4.4.3, the TAPE Scribe Database is used to store, manage, and retrieve TAPE data. The data presented in the sections below have been extracted from this database.

### 10.1.1 2007 TAPE Implementation

TAPE implementation for 2007 is presented below, organized into the following subsections: site access, dust sampling, attic and interior inspections, soil sampling, and ERS response activities.

#### *10.1.1.1 Site Access*

Tetra Tech initiated the 2007 TAPE field investigation process by mailing access agreements and cover letters to all property owners listed in the OU7 parcel geodatabase. At the time of the 2007 access agreement mailing, there were 1,092 mapped parcels in OU7 that matched ownership records in the CAMA tax database (Tetra Tech 2008a). Tetra Tech was able to ascertain valid and complete mailing addresses for 1,078 properties and sent an access agreement and cover letter to the owner of each. The cover letter explained the objectives of the TAPE, requested access to the property, and provided contact information for the DEQ Project Manager and the DEQ Troy Information Center.

Of the 1,078 property owners to whom access agreements were mailed, 586 returned a signed agreement granting access to their property. Eight property owners returned a signed access agreement with 'limited' access and 12 property owners denied access to their parcels. The reasons for limited access varied, and included such specifications as a 6-month access period only, one-time visit only, exterior inspection only, or secondary building inspection only. Prior to the development of TOAD, access information was tracked on the Troy Call Log through an EPA software program called the Web Emergency Operations Center. A description of the Web Emergency Operations Center software can be found in the Troy Data Management Plan Version 1.0 (Tetra Tech 2008a).

#### *10.1.1.2 Dust Sampling*

A total of 536 parcels underwent a TAPE inspection in 2007. The inspections included 508 primary and 730 secondary buildings. Dust samples were collected from primary buildings and from secondary buildings without dirt floors. A total of 1,211 dust samples (not including field QC samples) were collected. Of these, 678 were collected from primary buildings and 533 were collected from secondary buildings.

The original dust sampling protocol outlined in the TAPE work plan is based on project-specific SOP EPA-LIBBY-2012-10 (Appendix A), which specified 30 dust aliquots for each dust sample collected. During the test parcel sampling during training week, it was determined that 30 aliquots caused cassette overloading and pump failure. Record of modification TFO-00001 was drafted to address this issue. It adjusted the TAPE dust sampling protocol from 30 aliquots to 10 aliquots and specified the target areas from which the aliquots had to come (4 accessible, 4 infrequent, and 2 inaccessible). A copy of this record of modification is included in Appendix B.

In July 2007, TFO-00003 (Appendix B) initiated dust scheme prioritization sampling, which outlined a priority for sub-sample locations within each target area. For example, within an accessible area, the field team would prioritize collecting a dust aliquot from 'flooring at the main entrance used by occupants' over a dust aliquot from the 'kitchen surface'. The dust prioritization scheme and the sub-sample categories for each target area are outlined in a project-specific SOP EPA-LIBBY-2012-10 (Appendix A).

In August 2007, TFO-00005, which specified that dust samples only be collected from parcels with a 'dust trigger' (i.e., observed visible vermiculite in interior or exterior areas, or if the resident was a former miner or had an asbestos-related disease) be collected. The necessity of dust samples as a means of determining a removal action was under evaluation, and TFO-00005 was implemented on August 1, 2007, to assist with that evaluation. TFO-00005 reduced the number of dust samples and allowed investigators to compare dust analytical results to visual (dust trigger) or anecdotal (interview response) evidence. However, it was later decided that dust sampling should occur in all buildings to determine if a removal action was necessary. TFO-00006 was prepared on August 23, 2007, to terminate TFO-00005 and require that dust sampling occur in all buildings. Copies of these TFOs can be found in Appendix B.

#### *10.1.1.3 Attic and Interior Inspections*

As part of the TAPE inspection process, the living areas and attics of primary and secondary buildings were inspected for VV and VCI. Attic inspections were not done in buildings with inaccessible attics.

As stated above, 508 primary buildings and 730 secondary buildings were inspected in 2007. Of these, 111 primary buildings and 442 secondary buildings did not have attics, and the attics of 25 primary buildings and 16 secondary buildings were inaccessible. The remaining buildings were inspected for the presence or absence of attic insulation and the type of insulation. Fifty-six properties had VCI in an attic. Twenty eight properties had VV in an indoor living space.

#### *10.1.1.4 Soil Sampling*

The TAPE soil sampling protocol was modified at the beginning of TAPE activities on May 8, 2007, through TFO-00002, to set the maximum number of visual point inspections for an individual UA at 30. The modification required visual point inspections be evenly distributed throughout the UA and

corresponded to the sample aliquot locations (Appendix B). This modification altered the protocol outlined in the project-specific SOP CDM-LIBBY-16 (Appendix A), which specified that visual point inspections be performed at a minimum of one-point inspection per 100 square feet for UAs greater than 500 square feet in size. This visual point inspection protocol was not needed for the TAPE screening level survey and 30 aliquots spread over the entire UA was deemed sufficient for detecting VV if it was present. Follow up investigations would identify the exact extent of the VV prior to any removal action.

Tetra Tech identified and visually inspected for VV the following numbers of UAs in 2007:

- Common Use Areas = 502
- Limited Use Areas = 145
- Specific Use Areas = 1,146
- Non-use Areas = 194

In 2007, soil samples were not collected from SUAs with VV. Although not all vermiculite contains asbestos, historical sampling and analytical results from OU4 showed a strong correlation between VV in SUAs and LA contamination. Given the similarities between OU4 and OU7, it was hypothesized that VV in SUAs in OU7 would contain LA and that it would be cost effective to forego the collection of soil samples from SUAs with VV, and instead, use VV as a removal action criterion. Tetra Tech performed a 5-point minimum, 30-point maximum, visual point inspection of SUAs with VV and classified the amount of VV as none, low, intermediate, or high as described in SOP CDM-Libby 06 (Tetra Tech 2011b).

A total of 1,608 soil samples were collected in 2007 from the UAs listed above. In addition, 54 field soil duplicate samples were collected.

Tetra Tech also visually inspected and sampled interior crawlspace areas that were safe and accessible. A total of 316 interior crawlspace and 4 field duplicate soil samples were collected in 2007.

A review of resident interview responses and inspection results following the 2007 field season revealed a high frequency of “unexpanded/potting soil mix, homeowner purchase” responses for the description of the VV observed in SUAs. Many residents informed the field teams that they had purchased the vermiculite/potting mix from the hardware store (DEQ 2009a). Presumably, vermiculite or potting soil containing vermiculite that had been recently purchased from a store would not contain LA. In response

to this observation, TFO-00007 (Appendix B) was instituted on November 15, 2007, and specified collection of soil samples from all SUAs with VV. This modification went into effect at the beginning of the2008 field season.

#### *10.1.1.5 Environmental Resource Specialist Activities*

ERS response actions were performed at properties where TAPE inspections identified known or potential LA-containing source materials in interior living areas or high-use SUAs. The objective of the ERS response actions was to isolate potential LA-containing material from residents or workers until removal activities could be scoped and conducted. ERS response action activities typically included a cleanup phase (i.e., vacuuming vermiculite from interior living areas) followed by isolation measures (e.g., duct taping, caulking, or covering with plastic sheeting openings between non-living [e.g., unfinished attic] and living areas) to prohibit potential LA-containing vermiculite from coming into contact with residents or workers. In some cases, extended ERS responses were conducted. These included providing homeowners with HEPA vacuum cleaners, removing bags of Zonolite (i.e., vermiculite attic insulation) from attics or storage areas, and providing advice during remodeling activities.

Eleven ERS response actions were conducted in 2007, including one property outside the OU7 boundary. Each ERS response action was documented in an ERS report.

### 10.1.2 2008 TAPE Implementation

TAPE implementation for 2008 are presented below, organized into the following subsections: site access, dust sampling, attic and interior inspection, VV re-inspections and soil sampling, and ERS response actions.

#### *10.1.2.1 Site Access*

Tetra Tech initiated the 2008 TAPE field investigation process by mailing access agreements to 538 property owners in OU7. This number includes property owners that received an access agreement in 2007, but did not respond, as well as property owners not previously contacted. In addition, property owners who returned a signed access agreement in 2007, but did not get scheduled for a TAPE inspection in 2007, were contacted at the beginning of the 2008 field season for scheduling.

Of the parcel owners to whom access agreement were mailed in 2008, 391 returned a signed agreement granting access to their property. Eight property owners returned a signed access agreement with 'limited' access and 24 property owners denied access to their parcels.

#### *10.1.2.2 Dust Sampling*

A total of 261 parcels were inspected in 2008. The inspections included 226 primary buildings and 349 secondary buildings. A total of 573 dust samples were collected (not including duplicate or field blank samples). Of these, 313 were collected from primary buildings and 260 were collected from secondary buildings.

The comparison of inspection results for VV in interior living areas to corresponding dust analytical results revealed that none of the parcels with VV in interior living areas had corresponding dust results above the 5,000 s/cm$^2$ removal action level. The DEQ concluded that dust analytical results did not contribute meaningful information for removal action decision making. For this reason, TFO-00012, which specified cessation of dust sampling, was drafted in December 2008 and implemented at the start of the 2009 field season. A copy of TFO-00012 is provided in Appendix B.

#### *10.1.2.3 Attic and Interior Inspections*

Visual inspection of interior living areas and accessible attic spaces continued in 2008. As stated above, 226 primary and 349 secondary buildings underwent a TAPE inspection in 2008. Eighty-two primary buildings and 307 secondary buildings did not have attics, and the attics of 13 primary buildings and 10 secondary buildings were inaccessible. The remaining buildings were inspected for the presence or absence of attic insulation and the type of insulation. Twenty-one of the parcels inspected in 2008 had VCI in a building attic and 20 parcels had VV in an indoor living space.

#### *10.1.2.4 Visible Vermiculite Re-Inspections and Soil Sampling*

At the beginning of the 2008 field season, Tetra Tech revisited all UAs identified in 2007 to confirm, semi-quantify, and describe VV in exterior soils and collect soil samples. The reason for the re-inspections was to address the following: (1) poor correlation between soil analytical results and the corresponding VV observations , (2) the need for soil samples from all SUAs as specified in TFO-00007, and (3) the need for an accurate description of the VV that had been reported as 'store purchased potting soil,' to confirm the relationship between vermiculite observed in 'potting soil' and associated

analytical results (Tetra Tech 2008b). TFO-00008 was drafted and specified that Tetra Tech revisit 462 parcels to perform VV re-inspections at 1,743 UAs. It also specified that Tetra Tech revisit each SUA inspected in 2007 where VV was identified and collect a soil sample. Detailed information on how the parcels and UAs were selected, the sampling methodology, and the data management for the VV re-inspections is provided in TFO-00008 (Appendix B).

To ensure accurate identification and quantification of VV, nine Tetra Tech field personnel were trained by experienced CDM field personnel on the recognition of VV in exterior soils. Subsequently, Tetra Tech placed at least one of the additionally trained individuals on each TAPE inspection team during 2008 to ensure that VV was accurately identified.

Between May 20 and July 24, 2008, Tetra Tech re-inspected UAs on 101 parcels for the identification and possible quantification of VV in exterior soils. A total of 527 UAs were re-inspected; the type and number of UAs are as follows: 353 SUAs, 132 CUAs, 41 LUAs, and 1 NUA. As part of the requirements for TFO-00008, CDM conducted audits of Tetra Tech's VV re-inspection process. The first audit was completed in July 2008 on eight parcels where Tetra Tech had conducted VV re-inspection activities. CDM independently assessed VV at each of the parcels and prepared a technical memorandum detailing the findings (CDM 2008b). The identification and quantification of VV by both parties was comparable, and in addition, when Tetra Tech compared the re-inspection results to the original 2007 visual estimations, there was very little difference. CDM conducted two more assessments of VV in August and November 2008 on 28 additional parcels and prepared a revision to the original technical memorandum (CDM 2009b). There were slight variations between Tetra Tech and CDM's semi-quantification of VV, which were attributable to several factors as explained in their technical memorandum (CDM 2009b). The results of the audits demonstrated that VV was being adequately identified during the TAPEs. As a result, Tetra Tech discontinued the VV re-inspections.

The second objective of TFO-00008, to sample all 2007 SUAs with VV, was satisfied. Tetra Tech re-inspected and sampled 131 of the 144 SUAs outlined in TFO-00008. Not all property owners allowed access to their parcels for re-inspection, some of the SUAs identified in 2007 did not exist when field crews went back in 2008 to re-inspect (i.e. flowerpots), and a few of the SUAs could not be sampled due to the material used in the SUAs (i.e. washed gravel).

In December 2008, TFO-00010 was drafted, specifying cessation of the VV re-inspection requirement set forth in TFO-00008. Copies of both TFOs are included in Appendix B.

#### *10.1.2.5 Soil Sampling*

As part of the TAPE activities in 2008, Tetra Tech identified and visually inspected the 257 CUAs, 83 LUAs, 655 SUAs, and 103 NUAs. A total of 1,000 soil samples were collected in 2008 from these UAs (not including field QC samples). Tetra Tech also visually inspected and sampled safe and accessible interior crawlspace areas. A total of 95 interior soil samples (not including field QC samples) were collected in 2008.

TFO-00009 was drafted in June 2009. It specified that Tetra Tech collect split soil samples from UAs with sufficient soil. The protocol for split samples involved collecting twice the volume of soil, homogenizing within a single bowl, filling two separate bags, and giving each split sample an unique TT number (to be referenced to each other in the TAPE Scribe Database). The initiation of split sample collection was done so the laboratories analyzing the soil samples would have sufficient volume for an intra-lab QA study. The practice of collecting split soil samples was ceased on June 13, 2008 per TFO-00011 because the laboratories recognized that Tetra Tech could provide additional soil volume in one bag to be split once the sample arrived at the laboratory. TFO-00011 eliminated the collection of field split samples and specified an increase in soil volume by 50 percent (Appendix B).

#### *10.1.2.6 Environmental Resource Specialist Activities*

In 2008, ERS response actions were conducted at 12 parcels, including four parcels outside the OU7 boundary. Three of the four parcels outside of the OU7 boundary were south of the OU7 boundary and one was along the BNSF railroad tracks (OU6) in Troy. The 2008 ERS response actions involved temporarily isolating potential LA-containing material from residents or workers.

### 10.1.3 2009 TAPE Implementation

TAPE implementation in 2009 is summarized below, organized into the following subsections: site access, attic and interior inspections, soil sampling, road and alley sampling, aggressive attic entry and attic revisits, and ERS response actions. Each topic is presented in the subsections below.

#### *10.1.3.1 Site Access*

Tetra Tech initiated the 2009 TAPE field investigation process by mailing access agreements to 313 property owners in OU7. This number included owners that were mailed an access agreement in 2007 or 2008, but did not respond, as well as new property owners. Property owners who returned a signed access agreement, but did not get scheduled for a TAPE inspection in 2007 or 2008, were contacted by at the beginning of the 2009 field season for scheduling.

Of the 313 parcel owners to whom an access agreement was mailed in 2009, 180 returned a signed agreement granting access to their property. Twelve property owners returned a signed access agreement with 'limited' access and 18 property owners denied access to their parcels.

In addition to the access agreements received from the annual mailings, signed access agreements were also obtained from walk-ins to the DEQ Troy Information Office and from door-to-door solicitation of owners whose addresses were listed incorrectly (or were unknown) in the CAMA database. TOAD was used to track changes to parcel status.

#### *10.1.3.2 Attic and Interior Inspection*

The visual inspection of interior living areas and accessible attics continued in 2009. Tetra Tech inspected 436 properties in 2009, encompassing 120 primary buildings and 216 secondary buildings. Of these, 40 primary and 178 secondary buildings did not have attics. The attics of 12 primary buildings and 13 secondary buildings were inaccessible. The remaining attics were accessed and inspected for the presence or absence of attic insulation and the type of insulation. Five of the parcels initially inspected in 2009 had VCI in a building attic. Field teams also identified five properties with VV in an indoor living space.

#### *10.1.3.3 Aggressive Attic Entry and Attic Revisits*

To address the possibility that VCI was not being identified in attics due to limited access or due to situations where the inspector's view of the entire attic was obstructed, a modification to the work plan was drafted that allowed for more aggressive attic entry procedures. TFO-00013 (Appendix B) was approved on May 27, 2009, and specified that aggressive attic inspections would be conducted to adequately identify VCI, and that these procedures would be implemented, when feasible, during future

TAPEs and re-visits. Tetra Tech prepared a project-specific SOP entitled *TAPE Aggressive Attic Inspection Activities to Verify Presence or Absence of VCI*, which was submitted along with TFO-00013.

Tetra Tech queried the TAPE Scribe Database at the beginning of the 2009 field season for all primary and secondary buildings from 2007 and 2008 that had "no access" documented for the attic and for those buildings that were listed as "unknown" for the question "Is there VCI in the attic?." If the building was built after 1980, it was removed from the list of potential attics to revisit, because the construction year made it unlikely that the attic would contain VCI. Tetra Tech then contacted property owners to request an attic revisit, with the intent to perform an aggressive attic entry if necessary. The Scribe Database Administrator amended PocketScribe so that in addition to "no access", the field teams could provide clarification if possible: (1) No Access – Physical Barrier, (2) No Access – Entry Denied, and (3) No Access – No Vermiculite Insulation Per Owner. Tetra Tech identified eight buildings for a possible aggressive attic entry revisit.

In total, 30 parcels and 38 buildings were revisited in 2009 to verify attic access and perform a re-inspection if necessary. Several attics were indeed inaccessible, and were updated in the TAPE Scribe Database with one of the new descriptions. The majority of the attics could not be accessed due to a physical barrier. Some parcel owners refused to allow an attic revisit and other owners specified the attics did not contain VCI, despite the inability to access the attic.

#### *10.1.3.4 Soil Sampling*

In 2009, Tetra Tech identified and visually inspected the following types and numbers of UAs:

- Common Use Areas = 125
- Limited Use Areas = 153
- Specific Use Areas = 323
- Non-Use Areas = 47

A total of 581 soil samples and 30 field duplicate samples were collected in 2009 from the UAs listed above.

Tetra Tech also visually inspected and sampled interior safe and accessible crawlspace areas. A total of 75 interior soil samples were collected. In addition, two field duplicate soil samples were collected from interior crawlspaces.

#### *10.1.3.5 Road and Alley Sampling*

TAPE inspection of road and alley parcels in OU7 was initiated in 2009 in accordance with record of modification TFO-00014. Tetra Tech prepared the *Protocol Modification to the TAPE Project Work Plan, Inspection and Sampling of Roads and Alleys for Visible Vermiculite* to guide these inspections. It outlined the procedures and techniques to be used for visual point inspection and soil sampling of public road and alley parcels. Tetra Tech identified 316 road and alley parcels within OU7 and assigned each road and alley parcel a unique, 6-digit AD number starting with a 204 series (i.e. AD-204XXX). The sampling method was similar to soil sampling protocols used during TAPE inspections of residential and commercial properties in OU7. A copy of TFO-00014 and Tetra Tech's protocol modification for road and alley inspections are provided in Appendix B.

Tetra Tech inspected and sampled (as appropriate) all accessible road and alley parcels within OU7 during 2009. A total of 302 road and alley parcels were inspected for VV and 271 soil samples (not including field QC samples) were collected. The field team was unable to complete inspection of 14 road and alley parcels because they were inaccessible (i.e., private gate) or because the property owner refused access.

Out of 302 road and alley parcels inspected, VV was identified on 20. Visual inspection results for these parcels were compared to the corresponding analytical data and other lines of evidence to determine if removal action was warranted. Two of the inspected road and alley parcels abut residential parcels with VV and were eligible for removal action. The VV observed on the road and alley parcels was attributed to spillover from neighboring residential parcels with vermiculite. It was determined that these road and alley parcels would be addressed along with the residential parcels when removal actions were conducted at the residences. The visual inspection and analytical results from road and alley sampling suggest that LA-contaminated materials were not used in road building in OU7. A summary of road and alley soil analytical results is presented in Section 10.2.1.

#### *10.1.3.6 Environmental Resource Specialist Activities*

ERS response action activities were conducted at 11 properties in 2009. The 2009 ERS response actions involved temporarily isolating potential LA-containing material from residents or workers.

### 10.1.4 2010 TAPE Implementation

TAPE implementation in 2010 is summarized below, organized into the following subsections: site access, attic and interior inspections, soil sampling, and ERS response actions. Each topic is presented in the subsections below.

#### *10.1.4.1 Site Access*

Tetra Tech did not conduct a mass mailing of access agreements to OU7 residents prior to the 2010 field season because the majority of properties had already undergone a TAPE inspection, and with the level of public outreach within OU7, it was determined that interested property owners would contact the DEQ Troy Information Center directly if interested in an inspection. Property owners who had returned a signed access agreement previously but did not get scheduled for a TAPE inspection in 2009 were contacted at the beginning of the 2010 field season for scheduling.

As of December 31, 2010, the access status for OU7 parcels was the following:

Granted = 952

Limited = 10

Denied = 27

#### *10.1.4.2 Attic and Interior Inspection*

The visual inspection of interior living areas and attic spaces (when accessible) continued in 2010. Tetra Tech inspected 33 properties in 2010, encompassing 24 primary buildings and 25 secondary buildings. Of these, 24 buildings did not have attics. The attic of one building was inaccessible. The remaining attics were accessed and inspected for the presence or absence of attic insulation and the type of insulation. Five of the parcels inspected in 2010 had VCI in a building attic. Field teams also identified six properties with VV in an indoor living space.

#### *10.1.4.3 Soil Sampling*

Tetra Tech identified and visually inspected the following use areas during the 2010 field season:

- Common Use Areas = 27
- Limited Use Areas = 23
- Specific Use Areas = 28
- Non-use Areas = 15

A total of 234 soil samples (not including field QC samples) were collected in 2010 from the use areas listed above. Tetra Tech also visually inspected and sampled interior crawlspace areas (if safe and accessible). A total of 34 interior soil samples were collected in 2010.

#### *10.1.4.4 Environmental Resource Specialist Activities*

ERS response actions were conducted at 3 properties in 2010; all three are located outside the OU7 boundary. The 2010 ERS response actions involved temporarily isolating potential LA-containing material from residents or workers.

### 10.1.5 2011 TAPE Implementation

TAPE implementation in 2011 is summarized below, organized into the following subsections: site access, attic and interior inspections, soil sampling, and ERS response actions. Each topic is presented in the subsections below.

Tetra Tech conducted TAPE inspections of 35 parcels in 2011. Of these, 27 were within the OU7 boundary and 8 were outside the OU7 boundary (Figure 4-1). The out-of-OU7 boundary TAPE inspections were performed case-by-case because of unique circumstances such as owner transport of buildings or vermiculite from Libby to the parcel, or if the owner had knowledge of such an event. For the purposes of the RI, these parcels are considered part of OU7 and are included in the TAPE inspection count for calendar year 2011.

#### *10.1.5.1 Site Access*

Tetra Tech did not conduct a mass mailing of access agreements to OU7 residents in 2011 because most properties in OU7 had undergone a TAPE inspection previously, and considering the level of public

outreach in OU7, it was determined that interested property owners would contact the DEQ Troy Information Center directly if interested in an inspection. Property owners who had returned signed access agreements previously, but had not undergone a TAPE in 2010, were contacted at the beginning of the 2011 field season for scheduling.

The access status of all OU7 parcels as of December 31, 2011, was as follows:

Granted = 984

Limited = 10

Denied = 138

#### *10.1.5.2 Attic and Interior Inspections*

Visual inspections of interior living areas and accessible attic spaces continued in 2011. Tetra Tech inspected 35 parcels in 2011, which included 35 primary buildings and 72 secondary buildings. Of these buildings, 23 buildings did not have attics, and 11 buildings had inaccessible attics. The remaining attics were inspected for presence of and type of insulation. One building inspected in 2011 had VCI in the attic. Field teams also identified two properties with VV in an indoor living space.

#### *10.1.5.3 Soil Sampling*

Tetra Tech identified and visually inspected the following types and numbers of UAs in 2011:

CUAs = 27

LUAs = 30

SUAs = 31

NUAs = 19

A total of 208 soil samples (including exterior, interior, and co-located samples) were collected in 2011. Of these, 9 were co-located exterior samples and 11 were interior soil (i.e., from crawl spaces) samples (including co-located samples).

#### *10.1.5.4 Environmental Resource Specialist Activities*

ERS response actions were conducted at four parcels in 2011; two are located outside the OU7 boundary and two are located inside the boundary. The ERS response actions involved temporarily isolating potential LA-containing material from residents or workers.

Additionally, five minor ERS responses were completed by Tetra Tech in 2011. These responses did not require preparation of formal statements of work or involvement by Project Resources, Inc. (PRI) or the removal contractors. Electronic documentation records of these minor ERS response were placed in the electronic archive folders for the properties.

### 10.1.6 2012 TAPE Implementation

TAPE implementation in 2012 is summarized below, organized into the following subsections: site access, attic and interior inspections, soil sampling, and ERS response actions.

Tetra Tech conducted 15 TAPE inspections in 2012 (Figure 4-1). Of these, eight were on properties within the OU7 boundary and 7 were outside the OU7 boundary. The out-of-boundary TAPEs were performed case-by-case because of unique circumstances such as owner transport of buildings or vermiculite from Libby to the parcel, or if the owner had knowledge of such an event. For the purpose of the RI, these parcels are considered part of OU7 and are included in the TAPE inspection counts for 2012.

#### *10.1.6.1 Site Access*

Tetra Tech did not conduct a mass mailing of access agreements to OU7 residents in 2012 because most properties in OU7 had undergone a TAPE inspection previously, and, considering the level of public outreach in OU7, it was determined that interested property owners would contact the DEQ Troy Information Center directly if interested in an inspection. Property owners who had returned signed access agreements previously, but had not undergone a TAPE, were contacted at the beginning of the 2012 field season for scheduling.

#### *10.1.6.2 Attic and Interior Inspections*

Six of the 15 properties inspected in 2012 had buildings with attics, one of which was inaccessible. The 6 attics were inspected for presence of and type of insulation. No VCI was observed in any of the attics inspected in 2012. Field teams identified one property with VV in an indoor living space.

#### *10.1.6.3 Soil Sampling*

A total of 109 soil samples (including exterior, interior, and co-located samples) were collected in 2012. Of these, three were co-located exterior samples and 11 were interior soil (i.e., from crawl spaces) samples (including co-located samples).

#### *10.1.6.4 Environmental Resource Specialist Activities*

One ERS response action was conducted in 2012. The ERS response action involved temporarily isolating potential LA-containing material from residents or workers.

### 10.1.7 2013 TAPE Implementation

TAPE implementation in 2013 is summarized below, organized into the following subsections: site access, attic and interior inspections, soil sampling, and ERS response actions.

Tetra Tech conducted four TAPE inspections in 2013 (Figure 4-1). Three were on parcels located outside the OU7 boundary and one was on an OU7 parcel. The out-of-boundary TAPEs were performed case-by-case because of unique circumstances such as owner transport of buildings or vermiculite from Libby to the parcel, or if the owner had knowledge of such an event. For the purpose of the RI, these parcels are considered part of OU7 and are included in the TAPE inspection counts for 2013.

#### *10.1.7.1 Site Access*

Tetra Tech did not conduct a mass mailing of access agreements to OU7 residents in 2012 because most properties in OU7 had undergone a TAPE inspection previously, and considering the level of public outreach in OU7, it was determined that interested property owners would contact the DEQ Troy Information Center directly if interested in an inspection. Property owners who had returned signed access agreements previously, but had not undergone a TAPE, were contacted at the beginning of the 2012 field season for scheduling.

#### *10.1.7.2 Attic and Interior Inspections*

Tetra Tech inspected four parcels in 2013. Of these buildings inspected, three did not have attics, and one had an inaccessible attic. The remaining attics were inspected for presence of and type of insulation. One building inspected in 2013 had VCI in the attic. Field teams also identified one property with VV in an indoor living space.

#### *10.1.7.3 Soil Sampling*

A total of 61 soil samples (including exterior, interior, and co-located samples) were collected in 2013. Of these, none were co-located exterior samples and nine were interior soil (i.e., from crawl spaces) samples (including co-located samples).

#### *10.1.7.4 Environmental Resource Specialist Activities*

One ERS response action was conducted in 2013. The ERS response action involved temporarily isolating potential LA-containing material from residents or workers.

## 10.2 LIBBY AMPHIBOLE ASBESTOS CONTAMINATION IN OU7, SELECTION OF PARCELS FOR REMOVAL ACTION, AND POST-REMOVAL CLEARANCE AND CONFIRMATION SAMPLING

This section presents TAPE analytical results and compares them to criteria specified in the EPA and DEQ documents guiding removal action decisions in OU7—the EPA *Libby Asbestos Site Residential/Commercial Cleanup Action Level and Clearance Criteria Technical Memorandum* (EPA 2003), *Libby Asbestos Site Troy OU7 Residential/Commercial Cleanup Criteria Specific Use Area Visible Vermiculite Action Level Technical Memorandum* (DEQ 2009a), and the *Libby Asbestos Site Troy OU7 Removal Parcel Status* (DEQ 2009b) memorandum.

### 10.2.1 TAPE Analytical Results

The following subsections describe the analytical results from dust and soil sampling conducted during the TAPE.

**Dust Samples**

Indoor dust samples were collected in 2007 and 2008. Table 10-1 summarizes the analytical results. It does not include results for QC samples (field blanks or field duplicates).

A total of 1,211 dust samples were collected from primary and secondary buildings (without dirt floors) in 2007. Four of these samples exceeded the EPA removal action level for indoor dust; an LA concentration greater than 5,000 s/cm$^2$ (EPA 2003). One hundred and eighty-five samples had a detected LA concentration below 5,000 s/cm$^2$. LA was not detected in 1,022 of the 2007 dust samples.

A total of 573 dust samples were collected from primary and secondary buildings (without dirt floors) in 2008. Of these, results for two of the samples exceeded the EPA removal action level for indoor dust; an LA concentration greater than 5,000 s/cm$^2$ (EPA 2003). Ninety-seven samples had a detected LA concentration below 5,000 c/cm$^2$, and LA was not detected in 474 of the 2008 samples.

**TABLE 10-1: TAPE DUST SAMPLE ANALYTICAL RESULTS**

| Result | 2007 | 2008 |
|---|---|---|
| > 5,000 s/cm$^2$ | 4 | 2 |
| < 5,000 s/cm$^2$ | 185 | 97 |
| Non-detect | 1,022 | 474 |
| Total | 1,211 | 573 |

Notes:
> Greater than
< Less than
s/cm$^2$ Structures per square centimeter

**Exterior, Interior, and Co-Located Soil Samples**

Exterior soil samples were collected from UAs at all TAPE inspected properties beginning in 2007. In addition, Tetra Tech field crews collected indoor soil samples from safely accessible crawlspaces and dirt floors in primary and secondary buildings, and co-located (duplicate) soil samples. Soil analytical results for all field soil samples, excluding road and alley samples, are presented in Table 10-2 by year and assigned bin category. This table does not include results for QC samples (equipment blanks or co-located samples).

The bin categories for LA in soil are:

Bin A = Non-detect

Bin B1 = Trace LA (less than 0.2 percent)

Bin B2 = Between 0.2 and 1 percent LA

Bin C = LA greater than or equal to 1 percent by weight (EPA action level for removal)

It should be noted that no exterior or interior soil samples collected as part of the TAPE were assigned to Bin C.

**TABLE 10-2: TAPE SOIL SAMPLE ANALYTICAL RESULTS**

| Bin | Number of Samples | | | | | | |
|---|---|---|---|---|---|---|---|
| | 2007 | 2008 | 2009 | 2010 | 2011 | 2012 | 2013 |
| A | 1,274 | 894 | 426 | 227 | 146 | 91 | 51 |
| B1 | 637 | 327 | 228 | 49 | 62 | 15 | 10 |
| B2 | 13 | 5 | 2 | 0 | 0 | 0 | 0 |
| C | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Total | 1,924 | 1,226 | 656 | 276 | 208 | 106 | 61 |

Note:
Results include exterior, interior, and co-located soil samples.

**Road and Alley Soil Samples**

The analytical results for soil samples collected from road and alley parcels in 2009 are presented in Table 10-3 below. This table does not include results from QC samples such as equipment blanks or co-located samples.

**TABLE 10-3: TAPE ROAD AND ALLEY SOIL SAMPLE ANALYTICAL RESULTS**

| Bin | Number of Parcels |
|---|---|
| A | 197 |
| B1 | 74 |
| B2 | 0 |
| C | 0 |
| Total | 271 |

### 10.2.2 Comparison of TAPE Results to EPA Removal Action Levels

Two objectives of the TAPEs were to (1) identify parcels that met EPA removal action criteria (i.e., LA contamination at levels that pose potentially unacceptable health risks), and (2) provide reliable data on LA contamination for RDIs and removal activities that would minimize unacceptable health risks by removing the contamination. EPA established removal action levels that allowed investigators to determine whether a removal action was warranted (EPA 2003). The need for a removal action was assessed by determining contaminant concentrations in one or more of the following areas: (1) attic or interior walls (attics/walls), (2) indoor living space (interiors), or (3) outdoor soils (soils). A cumulative summary of TAPE results that met EPA removal action levels appears below:

**Attic/Walls**

- Total number of parcels with visual confirmation of VCI: 2007 through 2013 = 75

**Interiors**

- Total number of parcels with visual confirmation of VV in an indoor living space: 2007 through 2013 = 62
- Total number of indoor dust samples with an LA concentration exceeding 5,000 s/cm$^2$: 2007 through 2009 = 6

**Soils**

- Total number of SUAs with visual confirmation of VV: 2007 through 2013 = 292
- Total number of exterior soil samples with an LA concentration greater than or equal to 1 percent (i.e., Bin C): 2007 through 2013 = 0

### 10.2.3 DEQ Criterion for Visible Vermiculite in Specific Use Areas

As discussed in Section 10.1, the TAPE soil sampling protocol was originally based on procedures established for OU4, but with some modifications and updated data collection procedures. A strong correlation between VV in soil and the presence of LA fibers in soil samples had been observed in OU4; therefore, in 2007, soil samples were not collected from SUAs containing VV. However, at the end of the 2007 field season, it was noted that a relatively high number of property owners had informed Tetra Tech field teams that the vermiculite in their SUA had been purchased at a local hardware store (DEQ 2009a). Based on this information, DEQ modified the TAPE work plan (TFO-00007 [Appendix B]) to include soil sampling at SUAs with VV starting in 2008.

Of 392 soil samples collected in 2008 from SUAs with VV, only 3 contained LA fibers above 0.2 percent (Bin B2). There were no Bin C results from SUAs with VV (or for any OU7 soil sample). From this information, DEQ recommended that field crews continue to collect soil samples from all OU7 UAs, that the semi-quantification of VV in all UAs continue, and that the EPA action level of "visual confirmation of vermiculite or other vermiculite mine based materials" in SUAs in OU7 would indicate the need for further review of additional lines of evidence pertaining to the property to determine if the vermiculite noted could be from the W.R. Grace mine (DEQ 2009a). The DEQ suggested several lines of evidence, in addition to the presence of VV in an SUA, be considered for removal action decisions. These are discussed in Section 10.2.4.

### 10.2.4 Parcel Selection for Removal Action

Two of the primary objectives of the TAPE inspections were to identify parcels that met EPA removal action criteria and to provide reliable data on LA contamination in support of RDIs and removal activities. The overall goal was to address parcels with LA contamination at levels that present a sufficient health risk and minimize that risk by completing a removal action.

In addition to the EPA removal action criteria presented in the previous section, OU7 properties where evaluated using additional lines of evidence as described below. Background information regarding this process is provided in the *Final Libby Asbestos Site Troy Operable Unit 07 Residential/Commercial Cleanup Criteria, Specific Use Area Visible Vermiculite Action Level Technical Memorandum* (Attachment 1). In November 2009, DEQ evaluated OU7 parcels that had undergone a TAPE against EPA removal action levels and the additional lines of evidence summarized below (Attachment 1) to generate a list of OU7 properties where RDIs and removal actions were warranted. The EPA removal action levels and DEQ additional lines of evidence are:

**<u>EPA Removal Action Levels:</u>**

- VV in a living space = Yes
- VV in an attic = Yes
- VV in an SUA = Yes
- Field soil sample (interior or exterior) with an analytical result = Bin B2 or Bin C
- Dust concentration greater than or equal to 5,000 s/cm$^2$.

**DEQ Additional Lines of Evidence:**

- Any knowledge of former miners, close relatives of miners, or any highly exposed persons living or visiting the building? = Yes
- Has a resident, past or present, been diagnosed with an asbestos-related disease? = Yes
- To the best of your knowledge, was vermiculite from the mine used in, or around, your home? = Yes
- Has the resident/business purchased any Libby vermiculite materials from W.R. Grace in the past? = Yes
- Has the property been used for a for-profit enterprise of distributing, treating, storing, or disposing of Libby vermiculite? = Yes
- Are Libby vermiculite additives in any of the building materials? = Yes

Each parcel was reviewed against the above criteria to determine whether a removal action was necessary. This included review of the analytical results, electronic data archive records from visual inspections at the parcel (logbook, property sketch, photographs, and ERS reports), and all pertinent data from the OU7 Scribe Database (DEQ 2009b).

In 2012, EPA and DEQ revisited the removal criteria variations between OU4 and OU7. As a result, some OU7 properties that were on the "pending" list were revisited and placed on the removal list.

Table 10-4 summarizes the number of parcels meeting the EPA removal action levels used as OU4 removal criteria and the number of parcels meeting these criteria after parcel review and comparison to additional OU7 lines of evidence (DEQ 2009a, 2009b). The number of parcels presented in Table 10-4 does not directly compare to the number of OU7 parcels identified for removal due to several parcels satisfying more than one EPA removal criterion (i.e., a parcel with both VCI in the attic and a dust sample result greater than 5,000 s/cm$^2$).

**TABLE 10-4: PARCELS ELIGIBLE FOR REMOVAL ACTION**

| Total Number of Parcels | Number of Inspected Parcels | EPA 2003 Removal Action Levels | Parcels Meeting EPA 2003 Criteria | Parcels Meeting EPA Removal Criteria as Amended for OU7 |
|---|---|---|---|---|
| 1,578 | 1,239 | Visual confirmation of vermiculite in attic | 88 | 88 |
| | | Visual confirmation of vermiculite in living area | 62 | 62 |
| | | Concentration of LA in an indoor dust sample > 5,000 s/cm$^2$ | 6 | 6 |
| | | Concentration of LA in a soil sample ≥ 1 percent LA (Bin C) | 0 | 0 |
| | | Visual confirmation of vermiculite in SUA | 292 | 21 |

Notes:
> greater than
≥ greater than or equal to
LA Libby Amphibole asbestos
SUA Specific use area
s/cm$^2$ structures per square centimeter

An RDI was conducted at each property before removal activities were initiated. The primary objective of the RDI was to collect the additional data necessary to design removal activities based on the presence of LA and/or source of LA identified during the TAPE inspection. The RDIs were designed to fill remaining data gaps at these properties by further delineating the extent of LA. RDI and removal activities were initiated in OU7 in the spring of 2010.

### 10.2.5 Parcels Identified and Removal Actions

Tetra Tech coordinated RDI efforts with the property owners. The objective of the RDI process was to collect additional data such that remaining data gaps on the presence of LA and/or source of LA from TAPE inspections could be addressed and removal activities could be performed. A RDI was conducted at each participating property (some homeowners denied or deferred before removal activities were initiated). The exterior portion of the RDI inspection was performed by Tetra Tech and the interior portion of the RDI inspection was performed by a USACE contractor. Tetra Tech reviewed field and analytical inspection data prior to providing results of the RDI to the USACE contractor responsible for preparing design drawings and performing removal activities. Once design drawings had been prepared, Tetra Tech again reviewed the RDI results to ensure the removal was based on accurate information. Removal activities then commenced and were performed by the USACE contractor.

Table 10-5 below summarizes removal action information since project inception. Figure 10-1 depicts the parcels where removal activities have occurred since project inception.

**TABLE 10-5: SUMMARY OF OU7 REMOVAL ACTIVITIES**

| Type of Removal Action | Completed in 2008 | Completed in 2009 | Completed in 2010 | Completed in 2011 | Completed in 2012 | Completed in 2013 | Total Completed |
|---|---|---|---|---|---|---|---|
| Interior Only | 1 | 1 | 55 | 5 | 1 | 1 | 64 |
| Exterior Only | 0 | 0 | 12 | 0 | 0 | 0 | 12 |
| Combination | 6 | 5 | 17 | 1 | 0 | 0 | 29 |
| TOTAL | 7 | 6 | 84 | 6 | 1 | 1 | 105 |

The USACE contractor that performed the removal activities maintained detailed records of removal work at each property, such as the volume of interior material (i.e., insulation) removed and the volume of soil removed.

### 10.2.6 Post-Removal Clearance and Confirmation Sampling

Following completion of a removal action at a property, clearance air samples or confirmation soil samples, as appropriate, were collected and analyzed to determine whether the removal activities had been effective. Each sample type is described below.

**Clearance Air Samples**

If the removal involved vermiculite insulation, LA-contaminated interior dust or soil floors, or LA-contaminated building materials, clearance air samples were collected following removal activities to determine if interior LA contamination levels had been reduced to project-specific action levels.

Clearance air samples were collected from living spaces and non-living spaces (e.g., attics) where LA-contaminated media had been removed. Secondary structures were sampled in accordance with building-type designation as described in the response action work plan (RAWP) (PRI 2011). After the contractor had removed the contaminated material, a third-party independent contractor (TPIC) inspected the area to determine if clearance air sampling could commence. If sample results did not meet project-specific action levels, additional cleaning was performed and clearance samples were re-collected. Once the action levels had been met, the area was designated as adequately cleaned and restoration activities began.

Prior to collecting clearance air samples, a TPIC field member determined whether the area being sampled was considered a living space or an attic space to compare the data obtained to the project-specific action levels specified for these two area types. Five clearance samples were collected within

each area where a removal action had occurred. Each clearance air sample was collected and analyzed in accordance with TEM AHERA sampling guidance (EPA 1987), with applicable project-specific laboratory modifications. Clearance levels are discussed in detail in *the Libby Asbestos Site Residential/Commercial Clean Up Action Level and Clearance Criteria Technical Memorandum, Draft Final* (U.S. EPA 2003)

**Confirmation Soil Samples**

If a property required removal of vermiculite-containing or LA-contaminated soil, confirmation soil samples were collected following removal activities to determine if contaminated soils had been removed to project-specific clearance criteria.

Following excavation of contaminated soils within the removal area and before confirmation soil sampling, the excavated area and sidewalls were visually inspected for high concentrations of vermiculite. Because presence of high levels of vermiculite is a likely indicator of LA, further excavation may have been required prior to collecting confirmation soil samples. Once an excavation had been cleared through a visual inspection, a confirmation soil sample was collected for analysis and any remaining observed vermiculite within the sample area was documented during that sampling to determine if cleanup goals had been achieved.

Each confirmation soil sample was collected as a 30-point composite surface soil sample to characterize an area where contaminated soil had been removed and to document any remaining vermiculite. Each sample aliquot was collected from 0 to 2 inches below the surface of the completed excavation and consisted of nearly equal portions of soil from 30 locations within the delineated sample area. It was up to the discretion of the TPIC to decide the number of samples required to characterize the excavated area. However, to maintain consistency among the sampling teams, at least one composite sample was collected for every 2,500 square feet of excavation area.

Individual confirmation soil samples may have included composite points from different UAs (e.g., yard and flowerbed, yard and garden) as long as all areas had been excavated to design depth and had passed visual inspection. If excavation advanced more than 10 feet beyond a computed boundary to a neighboring property with a different address, soil clearance samples were collected in association with the different address with a separate FSDS, red-line drawing, and project completion checklist form. All confirmation soil samples were analyzed by PLM.

**Soil Sampling for Areas Not Excavated to Design Depth**

Excavation along or adjacent to foundations, curbs and roads, sidewalks, and around trees presented challenges to the removal contractor—additional potential hazards included structure failure, slope failure, and falling trees. Therefore, excavations in these areas may not have been advanced to the design depth specified in the site-specific removal work plan. In addition, these areas may have been sampled separately or in combination with other similar areas, as necessary. Although no additional excavation may have been feasible, samples were collected for documentation. Compositing samples collected at multiple areas that were not excavated to design depth into one sample was deemed acceptable in some instances. However, to be more representative of the areas not excavated to design depth, samples collected in these areas were not combined with sample aliquots from areas excavated to design depth.

Excavation around trees was completed in accordance with the RAWP (PRI 2011) and the site-specific removal work plan to the extent possible without impacting the integrity of the root system. If sampling was not feasible due to root congestion, a visual inspection, as outlined in project-specific SOP CDM-LIBBY-16 (Appendix A), was performed, and quantities of vermiculite were documented as low or intermediate. If high concentrations of vermiculite were present, TPIC obtained approval from the government representative to allow excavation to continue.

**Soil Sampling Under Structures**

If a structure (e.g., shed, deck, etc.) was moved during excavation and the footprint of the structure was less than 2,500 square feet, composite points of soil from the original structure's location may have been combined with composite points of soil from the surrounding area to a maximum of 2,500 square feet of the combined areas. If a structure was not moved during excavation, a separate discreet soil sample was collected in the footprint of the structure, and was not combined with samples from the surrounding excavation area**.**

## 10.3 OUTDOOR AMBIENT AIR STUDY RESULTS

The outdoor AAS monitoring program implemented by Tetra Tech is based on the Remedial Investigation Work Plan, Outdoor Ambient Air Study (Tetra Tech 2009a) and includes monitoring of ambient air in four distinct “air zones” across OU7. Tetra Tech established monitoring station locations

within four air zones to evaluate upwind, downwind and residential exposure scenarios (Figure 5-1). The AAS monitoring activities and schedule, and analytical results organized by year, are summarized below.

### 10.3.1 Monitoring Activities and Schedule

Three years of AAS monitoring began with Sample Period 1 on October 30, 2009, and continued through June 21, 2013 (Sample Period 101). The following sections provide a summary by year of AAS monitoring activities and schedule.

#### *10.3.1.1 Year 1 Activities and Schedule*

Year 1 monitoring was initiated on October 30, 2009. Initial field activities such as selection of the seven site monitoring stations and assembly and installation of monitoring equipment are described in the Remedial Investigation Work Plan, Outdoor Ambient Air Study (Tetra Tech 2009a). Figure 5-1 shows the Year 1 monitoring station locations, Table 10-6 shows sample periods, and Table 10-7 provides the general and detailed locations and rationale for the Year 1 station locations.

Year 1 ambient air sampling consisted of 36 five-day sampling periods generally separated by five off days between each period. Between some sampling periods, the five days were modified by one or two days to adjust for weather or scheduling issues, however, the overall sampling schedule was not impacted. Year 1 sampling began with period 1 on October 30, 2009 and ended with period 36 on October 24, 2010.

Throughout Year 1, field data and observations were recorded daily on FSDSs. A detailed summary of Year 1 activities and schedule are provided (by quarter) in the following technical memoranda.

- *Final First Quarter Memorandum, Outdoor Ambient Air Study, Operable Unit Number 7 of the Libby Asbestos Superfund Site*, dated February 2010 (Tetra Tech 2010c)
- *Final Quarter 2 and Quarter 3 Memorandum, Outdoor Ambient Air Study, Operable Unit Number 7 of the Libby Asbestos Superfund Site*, dated September 2010 (Tetra Tech 2010d)
- *Final Quarter 4 Memorandum, Outdoor Ambient Air Study, Operable Unit Number 7 of the Libby Asbestos Superfund Site*, dated December 2010 (Tetra Tech 2010e)

#### *10.3.1.2 Year 2 Activities and Schedule*

Year 2 monitoring was initiated on November 10, 2010. At the start of Year 2 (quarter 5), six of the seven monitoring stations were moved from their Year 1 locations to new locations in order to collect further data in support of the OU7 human health risk assessment. Figure 5-1 shows the Year 2 monitoring station locations. Table 10-6 shows sample periods and Table 10-7 provides the general and detailed locations and rationale for the Year 2 station locations.

**TABLE 10-6: AMBIENT AIR PROGRAM SAMPLE PERIODS**

| QUARTER 1 SAMPLE PERIODS | |
|---|---|
| **Sample Period 1** | October 30, 2009 to November 3, 2009 |
| **Sample Period 2** | November 9, 2009 to November 13, 2009 |
| **Sample Period 3** | November 19,2009 to November 23, 2009 |
| **Sample Period 4** | November 29, 2009 to December 3, 2009 |
| **Sample Period 5** | December 9, 2009 to December, 13, 2009 |
| **Sample Period 6** | December 19, 2009 to December 23, 2009 |
| **Sample Period 7** | December 29, 2009 to January 2, 2010 |
| **Sample Period 8** | January 8, 2010 to January 12, 2010 |
| **Sample Period 9** | January 18, 2010 to January 22, 2010 |
| **QUARTER 2 SAMPLE PERIODS** | |
| **Sample Period 10** | January 28, 2010 through February 1, 2010 |
| **Sample Period 11** | February 7, 2010 through February 11, 2010 |
| **Sample Period 12** | February 17, 2010 through February 21, 2010 |
| **Sample Period 13** | March 6, 2010 through March 10, 2010 |
| **Sample Period 14** | March 16, 2010 through March 20, 2010 |
| **Sample Period 15** | March 28, 2010 through April 1, 2010 |
| **Sample Period 16** | April 7, 2010 through April 11, 2010 |
| **Sample Period 17** | April 17, 2010 through April 21, 2010 |
| **Sample Period 18** | April 27, 2010 through May 1, 2010 |
| **QUARTER 3 SAMPLE PERIODS** | |
| **Sample Period 19** | May 7, 2010 through May 11, 2010 |
| **Sample Period 20** | May 17, 2010 through May 21, 2010 |
| **Sample Period 21** | May 27, 2010 through May 31, 2010 |
| **Sample Period 22** | June 6, 2010 through June 10, 2010 |
| **Sample Period 23** | June 16, 2010 through June 20, 2010 |
| **Sample Period 24** | June 26, 2010 through June 30, 2010 |
| **Sample Period 25** | July 6, 2010 through July 10, 2010 |
| **Sample Period 26** | July 16, 2010 through July 20, 2010 |
| **Sample Period 27** | July 26, 2010 through July 30, 2010 |
| **QUARTER 4 SAMPLE PERIODS** | |
| **Sample Period 28** | August 5, 2010 through August 9, 2010 |
| **Sample Period 29** | August 15, 2010 through August 19, 2010 |
| **Sample Period 30** | August 25, 2010 through August 29, 2010 |
| **Sample Period 31** | September 4, 2010 through September 8, 2010 |
| **Sample Period 32** | September 14, 2010 through September 18, 2010 |

## TABLE 10-6: AMBIENT AIR PROGRAM SAMPLE PERIODS (Cont.)

| | |
|---|---|
| **Sample Period 33** | September 24, 2010 through September 28, 2010 |
| **Sample Period 34** | October 4, 2010 through October 8, 2010 |
| **Sample Period 35** | October 14, 2010 through October 18, 2010 |
| **Sample Period 36** | October 24, 2010 through October 28, 2010 |
| **QUARTER 5 SAMPLE PERIODS** | |
| **Sample Period 37** | November 10, 2010 through November 14, 2010 |
| **Sample Period 38** | November 20, 2010 through November 24, 2010 |
| **Sample Period 39** | December 1, 2010 through December 5, 2010 |
| **Sample Period 40** | December 10, 2010 through December 14, 2010 |
| **Sample Period 41** | December 20, 2010 through December 24, 2010 |
| **Sample Period 42** | December 30, 2010 through January 3, 2011 |
| **Sample Period 43** | January 9, 2011 through January 13, 2011 |
| **Sample Period 44** | January 19, 2011 through January 23, 2011 |
| **Sample Period 45** | January 29, 2011 through February 2, 2011 |
| **QUARTER 6 SAMPLE PERIODS** | |
| **Sample Period 46** | February 9, 2011 through February 13, 2011 |
| **Sample Period 47** | February 19, 2011 through February 23, 2011 |
| **Sample Period 48** | March 1, 2011 through March 5, 2011 |
| **Sample Period 49** | March 11, 2011 through March 15, 2011 |
| **Sample Period 50** | March 21, 2011 through March 25, 2011 |
| **Sample Period 51** | April 1, 2011 through April 5, 2011 |
| **Sample Period 52** | April 10, 2011 through April 14, 2011 |
| **Sample Period 53** | April 21, 2011 through April 25, 2011 |
| **Sample Period 54** | April 30, 2011 through May 4, 2011 |
| **QUARTER 7 SAMPLE PERIODS** | |
| **Sample Period 55** | May 10, 2011 through May 14, 2011 |
| **Sample Period 56** | May 20, 2011 through May 24, 2011 |
| **Sample Period 57** | May 30, 2011 through June 4, 2011 |
| **Sample Period 58** | June 9, 2011 through June 13, 2011 |
| **Sample Period 59** | June 19, 2011 through June 23, 2011 |
| **Sample Period 60** | June 29, 2011 through July 3, 2011 |
| **Sample Period 61** | July 9, 2011 through July 13, 2011 |
| **Sample Period 62** | July 19, 2011 through July 23, 2011 |
| **Sample Period 63** | July 29, 2011 through August 2, 2011 |
| **QUARTER 8 SAMPLE PERIODS** | |
| **Sample Period 64** | August 7, 2011 through August 11, 2011 |
| **Sample Period 65** | August 16, 2011 through August 20, 2011 |
| **Sample Period 66** | August 26, 2011 through August 30, 2011 |
| **Sample Period 67** | September 5, 2011 through September, 9, 2011 |
| **Sample Period 68** | September 15, 2011 through September 19, 2011 |
| **Sample Period 69** | September 25, 2011 through September 29, 2011 |
| **Sample Period 70** | October 6, 2011 through October 10, 2011 |
| **Sample Period 71** | October 15, 2011 through October 19, 2011 |
| **Sample Period 72** | October 25, 2011 through October 29, 2011 |

## TABLE 10-6: AMBIENT AIR PROGRAM SAMPLE PERIODS (Cont.)

| QUARTER 9 SAMPLE PERIODS | |
|---|---|
| **Sample Period 73** | May 10, 2012 through May 14, 2012 |
| **Sample Period 74** | May 20, 2012 through May 24, 2012 |
| **Sample Period 75** | May 30, 2012 through June 3, 2012 |
| **Sample Period 76** | June 9, 2012 through June 13, 2012 |
| **Sample Period 77** | June 19, 2012 through June 23, 2012 |
| **Sample Period 78** | June 29, 2012 through July 3, 2012 |
| **Sample Period 79** | July 9, 2012 through July 13, 2012 |
| **Sample Period 80** | July19, 2012 through July 23, 2012 |
| **Sample Period 81** | July29, 2012 through August 2, 2012 |
| **QUARTER 10 SAMPLE PERIODS** | |
| **Sample Period 82** | August 8, 2012 through August 12, 2012 |
| **Sample Period 83** | August 18, 2012 through August 22, 2012 |
| **Sample Period 84** | August 28, 2012 through September 1, 2012 |
| **Sample Period 85** | September 7, 2012 through September 11, 2012 |
| **Sample Period 86** | September 17, 2012 through September 21, 2012 |
| **Sample Period 87** | September 27, 2012 through October 1, 2012 |
| **Sample Period 88** | October 7, 2012 through October 11, 2012 |
| **Sample Period 89** | October 17, 2012 through October 21, 2012 |
| **Sample Period 90** | October 27, 2012 through October 31, 2012 |
| **QUARTER 11 SAMPLE PERIODS** | |
| **Sample Period 91** | November 7, 2012 through November 11, 2012 |
| **Sample Period 92** | November 17, 2012 through November 21, 2012 |
| **Sample Period 93** | November 28, 2012 through December 2, 2012 |
| **Sample Period 94** | December 7, 2012 through December 11, 2012 |
| **Sample Period 95** | December 16, 2012 through December 20, 2012 |
| **Sample Period 96** | December 23, 2012 through December 27, 2012 |
| **QUARTER 12 SAMPLE PERIODS** | |
| **Sample Period 97** | February 18, 2013 through February 22, 2013 |
| **Sample Period 98** | March 15, 2013 through March 19, 2013 |
| **Sample Period 99** | April 8, 2013 through April 12, 2013 |
| **Sample Period 100** | May 24, 2013 through May 28, 2013 |
| **Sample Period 101** | June 17, 2013 through June 21, 2013 |

## TABLE 10-7: AMBIENT AIR PROGRAM SAMPLE STATIONS

| AMBIENT AIR PROGRAM YEAR 1 (2009-2010) | | | |
|---|---|---|---|
| **Station Number** | **General Location[a]** | **Detailed Location** | **Purpose** |
| T1 | Community exposure site and upper-middle bounds of OU7, located at the small community area NE of the Kootenai River | Residential Property at North River Road | This site is used to evaluate LA concentrations at the small community and the upper-middle bounds of OU7 |
| T2 | Upwind/downwind site near the NW border of OU7 | Fire Station at Forest Drive at Vacation Rd in Kootenai Vista | This site is used to evaluate LA concentrations at the northern boundary of OU7 |
| T3 | City of Troy northern site | Water Treatment Station at North end of Roosevelt Park | This site is used to evaluate LA concentrations north of the Troy community |
| T4 | City of Troy population exposure site | DEQ Troy Info. Center at 303 Third Street | This site is used to evaluate LA concentrations in the Troy community (specifically in the population center). |
| T4QC | City of Troy population exposure site | DEQ Troy Info. Center at 303 Third Street | Co-located sample station of T4 |
| T5 | City of Troy southern site | County Shops at Hwy. 2 at Sunset Rd. | This site is used to evaluate LA concentration south of the Troy community |
| T6 | Upwind/downwind site near the SW boarder of OU7 | Water Tower at Iron Creek Rd. ¾ mile south of Hwy 2 | This site is used to evaluate LA concentrations at the southwestern boundary of the OU and confirm if any LA is entering or leaving OU7 |
| T7 | Upwind/downwind site near the SE boarder of OU7 | Residential Property at Hummingbird Way at Bighorn Way in Wilderness Plateau | This site is used to evaluate LA concentrations at the southeastern boundary of OU7 |

| AMBIENT AIR PROGRAM YEAR 2 (2010-2011) | | |
|---|---|---|
| **Station Number** | **Location[a]** | **Purpose** |
| T11 | Community exposure site and middle portion of OU7, located at the small community area NE of the Kootenai River | This site is used to evaluate LA concentrations at the small community area and the northern boundary of OU7 |
| T12 | Upwind and downwind site near the NW border of OU7 | This site is used to evaluate LA concentrations at the northernmost boundary of OU7 and confirm if any LA is entering or leaving OU7 |
| T13 | City of Troy northern site | This site is used to evaluate LA concentrations north of the Troy community |
| T14 | City of Troy population exposure site | This site is used to evaluate LA concentrations in the Troy community (specifically in the population center). |
| T15 | City of Troy southern site | This site is used to evaluate LA concentration south of the Troy community |
| T16 | SW upwind and downwind site | This site is used to evaluate LA concentrations at the southwestern boundary of the OU and confirm if any LA is entering or leaving OU7 |
| T17 | SE upwind and downwind site | This site is used to evaluate LA concentrations at the southeastern boundary of the OU and confirm if any LA is entering or leaving OU7 |
| TXXQC | Rotating co-located sampling station to each of the seven sampling locations | Co-located sampling station to evaluate analytical variability at each of the seven station locations |

## TABLE 10-7: AMBIENT AIR PROGRAM SAMPLE STATIONS (Cont.)

| AMBIENT AIR PROGRAM YEAR 3 (2012-2013) | | |
|---|---|---|
| **Station Number** | **Location[a]** | **Purpose** |
| T21 | Upwind and downwind site near the northern border of OU7 | This site is used to evaluate LA concentrations at the small community area near the northernmost boundary of OU7 and confirm if any LA is entering or leaving OU7 through Air Zone 1 |
| T22 | City of Troy population exposure site | This site is used to evaluate LA concentrations in the Troy community (specifically in the population center) of Air Zone 2 |
| T23 | City of Troy southern site | This site is used to evaluate LA concentration south of the Troy community in Air Zone 3 |
| T24 | SE upwind and downwind site | This site is used to evaluate LA concentrations at the southeastern boundary of OU7 and confirm if any LA is entering or leaving OU7through Air Zone 4 |
| TXXQC | Rotating co-located sampling station to each of the four sampling locations | Co-located sampling station to evaluate analytical variability at each of the four station locations |

Notes:

a Predominant winds in the area blow from the southeast and northwest. Stations on the southeast and northwest boundaries of OU7 act as upwind and downwind receptors depending on wind direction.

| | | | |
|---|---|---|---|
| LA | Libby Amphibole | SE | Southeast |
| NE | Northeast | SW | Southwest |
| NW | Northwest | OU7 | Operable Unit 7 |

Year 2 ambient air sampling consisted of 36 five-day sampling periods generally separated by five off days between each period. Between some sampling periods, the five days were modified by one or two days to adjust for weather or scheduling issues, however, the overall sampling schedule was not impacted. Year 2 sampling began with period 37 on November 10, 2010 and ended with period 72 on October 25, 2011.

Throughout Year 2, field data and observations were recorded daily on FSDSs. A detailed summary of Year 2 activities and schedule are provided (by quarter) in the following technical memoranda.

- *Final Quarter 5 Memorandum, Outdoor Ambient Air Study, Operable Unit Number 7 of the Libby Asbestos Superfund Site*, dated February 2011 (Tetra Tech 2011d)
- *Final Quarter 6 Memorandum, Outdoor Ambient Air Study, Operable Unit Number 7 of the Libby Asbestos Superfund Site*, dated August 2011 (Tetra Tech 2011e)
- *Final Quarter 7 Memorandum, Outdoor Ambient Air Study, Operable Unit Number 7 of the Libby Asbestos Superfund Site*, dated September 2011 (Tetra Tech 2011f)
- *Final Quarter 8 Memorandum, Outdoor Ambient Air Study, Operable Unit Number 7 of the Libby Asbestos Superfund Site*, dated December 2011 (Tetra Tech 2011g)

#### *10.3.1.3 Year 3 Activities and Schedule*

Year 3 ambient air monitoring was initiated on May 10, 2012. At the start of Year 3 (Quarter 9), a total of four monitoring stations (reduced from the seven stations utilized in Year 1 and Year 2) were placed at their new locations to collect further data in support of the OU7 human health risk assessment. The Year 3 station locations were chosen from Year 1 locations. Figure 5-1 shows the Year 3 monitoring station locations, Table 10-6 shows sample periods, and Table 10-7 provides the general and detailed locations and rationale for the Year 3 station locations.

Year 3 sampling began with period 73 on May 10, 2012 and ended with period 101 on June 21, 2013. Table 10-6 provides a summary of sampling dates for periods 73 through 101. Year 3 AAS sampling consisted of 29 five-day sampling periods generally separated by five off days between each period during Quarter 9 and Quarter 10 (sample periods 73 through 90). Between some sampling periods, the 5 days were modified by 1 or 2 days to adjust for weather or scheduling issues; the overall sampling schedule was not impacted. During Quarter 11, only 6 sampling periods occurred (sample periods 91 through 96) as the task order ended and could not be resumed in time to continue sampling with no interruptions. Once the task order was renewed, sampling resumed with Quarter 12 sampling periods 97

through 101 being conducted once per month from February 2013 through June 2013. Sampling was conducted at all four stations during periods 97-100 and was reduced to three of the four stations for period 101.

Throughout year 3, field data and observations were recorded daily on FSDSs. A detailed summary of Year 3 activities and schedule for quarters 9, 10, and 11 are provided in the following technical memoranda. No technical memorandum was prepared for Quarter 12.

- *Final Quarter 9 Memorandum, Outdoor Ambient Air Study, Operable Unit Number 7 of the Libby Asbestos Superfund Site*, dated December 2012 (Tetra Tech 2012c)
- *Final Quarter 10 Memorandum, Outdoor Ambient Air Study, Operable Unit Number 7 of the Libby Asbestos Superfund Site*, dated May 2013 (Tetra Tech 2013a)
- *Final Quarter 11Memorandum, Outdoor Ambient Air Study, Operable Unit Number 7 of the Libby Asbestos Superfund Site*, dated July 2013 (Tetra Tech 2013b)

### 10.3.2 Analytical Results

This section summarizes the AAS analytical data acquired during the entire three years of the AAS program (sampling periods 1 through 101). Detailed discussions of AAS analytical results appear in the applicable AAS program quarterly reports as referenced above and are only briefly summarized herein. Table 10-8 provides a summary of all LA detections and Figure 5-1 provides the AAS station locations for AAS program years 1 through 3.

The complete set of analytical results for AAS sampling periods 1 through 101 are available in the Scribe Database. Data review and data entry verification findings for the AAS are provided in the AAS quarterly reports.

#### *10.3.2.1 Year 1 Analytical Results*

Year 1 of the AAS program included sampling periods 1 through 36. Year 1 results indicated the presence of LA in a total of 18 samples with LA identified at each of the seven station locations and at the stationary co-located station. LA detections included a range of 1 to 5 structures per sample and concentrations ranging from 3.57E-05 to 1.75E-04 structures per cubic centiliter (s/cc).

**TABLE 10-8**
**AMBIENT AIR PROGRAM**
**SUMMARY OF LIBBY AMPHIBOLE DETECTIONS**

| Property ID | Location | Location Comment | Sample No. | Sample Date | Sample Type | COC | LA Detected | No Of LA Structures Counted | LA Concentration (s/cc) | Sampling Period |
|---|---|---|---|---|---|---|---|---|---|---|
| AD-200653 | T4QC | DEQ QC | TA-0001 | 30-Oct-09 | Field Duplicate | TAA0001 | Y | 3 | 1.20E-04 | 1 |
| AD-200920 | T5 | State Hwy Dept/Sewer Lift Station | TA-0003 | 30-Oct-09 | Field Sample | TAA0001 | Y | 5 | 1.75E-04 | 1 |
| AD-200653 | T4 | DEQ | TA-0004 | 30-Oct-09 | Field Sample | TAA0001 | Y | 4 | 1.56E-04 | 1 |
| AD-201580 | T2 | Kootenai Vista Truck Barn #2 TRFD | TA-0078 | 18-Jan-10 | Field Sample | TAA0009 | Y | 1 | 3.77E-05 | 9 |
| AD-201580 | T2 | Kootenai Vista Truck Barn #2 TRFD | TA-0087 | 28-Jan-10 | Field Sample | TAA0010 | Y | 1 | 3.81E-05 | 10 |
| AD-200653 | T4QC | DEQ QC | TA-0099 | 07-Feb-10 | Field Duplicate | TAA0011 | Y | 4 | 1.58E-04 | 11 |
| AD-200809 | T1 | Brown Rental | TA-0104 | 17-Feb-10 | Field Sample | TAA0012 | Y | 2 | 7.37E-05 | 12 |
| AD-201580 | T2 | Kootenai Vista Truck Barn #2 TRFD | TA-0105 | 17-Feb-10 | Field Sample | TAA0012 | Y | 1 | 3.77E-05 | 12 |
| AD-200335 | T3 | City Park Shop | TA-0106 | 17-Feb-10 | Field Sample | TAA0012 | Y | 1 | 3.68E-05 | 12 |
| AD-201138 | T7 | Jordan Residence | TA-0110 | 17-Feb-10 | Field Sample | TAA0012 | Y | 1 | 3.63E-05 | 12 |
| AD-200653 | T4 | DEQ | TA-0115 | 06-Mar-10 | Field Sample | TAA0013 | Y | 1 | 3.74E-05 | 13 |
| AD-200653 | T4QC | DEQ QC | TA-0125 | 16-Mar-10 | Field Duplicate | TAA0014 | Y | 2 | 7.15E-05 | 14 |
| AD-201535 | T6 | Iron Creek Road Water Tower | TA-0127 | 16-Mar-10 | Field Sample | TAA0014 | Y | 1 | 3.57E-05 | 14 |
| AD-201138 | T7 | Jordan Residence | TA-0128 | 16-Mar-10 | Field Sample | TAA0014 | Y | 1 | 3.63E-05 | 14 |
| AD-201580 | T2 | Kootenai Vista Truck Barn #2 TRFD | TA-0131 | 28-Mar-10 | Field Sample | TAA0015 | Y | 1 | 3.96E-05 | 15 |
| AD-200809 | T1 | Brown Rental | TA-0148 | 17-Apr-10 | Field Sample | TAA0017 | Y | 1 | 3.96E-05 | 17 |
| AD-200653 | T4 | DEQ | TA-0160 | 27-Apr-10 | Field Sample | TAA0018 | Y | 1 | 3.96E-05 | 18 |
| AD-200920 | T5 | State Hwy Dept/Sewer Lift Station | TA-0316 | 14-Oct-10 | Field Sample | TAA0035 | Y | 1 | 3.97E-05 | 35 |
| AD-200783 | T11QC | Epps Body Shop QC | TA-20068 | 19-Jan-11 | Field Duplicate | TAA0044 | Y | 2 | 7.76E-05 | 44 |
| AD-201580 | T12 | NW border of OU7 | TA-20151 | 20-Apr-11 | Field Sample | TAA0053 | Y | 1 | 3.97E-05 | 53 |
| AD-201580 | T12 | NW border of OU7 | TA-20204 | 18-Jun-11 | Field Sample | TAA0059 | Y | 1 | 3.90E-05 | 59 |
| AD-200381 | T14 | City of Troy Central Site | TA-20224 | 08-Jul-11 | Field Sample | TAA0061 | Y | 1 | 3.99E-05 | 61 |
| AD-200783 | T11 | Mid-OU7 Community Exposure Site | TA-20275 | 06-Sep-11 | Field Sample | TAA0067 | Y | 1 | 3.92E-05 | 67 |
| AD-201580 | T12 | NW border of OU7 Site | TA-20276 | 06-Sep-11 | Field Sample | TAA0067 | Y | 1 | 3.92E-05 | 67 |
| AD-200381 | T14 | City of Troy Central Site | TA-20279 | 06-Sep-11 | Field Sample | TAA0067 | Y | 1 | 2.67E-04 | 67 |
| AD-200381 | T14 | City of Troy Central Site | TA-20287 | 16-Sep-11 | Field Sample | TAA0068 | Y | 2 | 7.96E-05 | 68 |
| AD-200630 | T15 | City of Troy Southern Site | TA-20297 | 26-Sep-11 | Field Sample | TAA0069 | Y | 1 | 3.85E-05 | 69 |
| AD-201580 | T21QC | Fire Station QC | TA-20333 | 5/10/2012 | Field Duplicate | TAA0073A | Y | 1 | 4.00E-05 | 73 |
| AD-200653 | T22 | DEQ | TA-20342 | 5/30/2012 | Field Sample | TAA0075 | Y | 2 | 7.99E-05 | 75 |
| AD-201535 | T23 | Troy Water Tower | TA-20362 | 6/29/2012 | Field Sample | TAA0078 | Y | 1 | 4.00E-05 | 78 |

**TABLE 10-8**
**AMBIENT AIR PROGRAM**
**SUMMARY OF LIBBY AMPHIBOLE DETECTIONS**

| Property ID | Location | Location Comment | Sample No. | Sample Date | Sample Type | COC | LA Detected | No Of LA Structures Counted | LA Concentration (s/cc) | Sampling Period |
|---|---|---|---|---|---|---|---|---|---|---|
| AD-201580 | T21 | Fire Station | TA-20377 | 7/29/2012 | Field Sample | TAA0081 | Y | 1 | 3.47E-05 | 81 |
| AD-201580 | T21QC | Fire Station QC | TA-20378 | 7/29/2012 | Field Duplicate | TAA0081 | Y | 3 | 1.08E-04 | 81 |
| AD-200653 | T22QC | DEQ QC | TA-20386 | 8/8/2012 | Field Duplicate | TAA0082 | Y | 1 | 3.91E-05 | 82 |
| AD-201580 | T21QC | Fire Station QC | TA-20402 | 9/7/2012 | Field Duplicate | TAA0085 | Y | 1 | 3.97E-05 | 85 |
| AD-200653 | T22 | DEQ | TA-20403 | 9/7/2012 | Field Sample | TAA0085 | Y | 1 | 3.97E-05 | 85 |
| AD-201535 | T23 | Troy Water Tower | TA-20415 | 9/27/2012 | Field Sample | TAA0087 | Y | 1 | 3.99E-05 | 87 |
| AD-201138 | T24 | Jordan Residence | TA-20417[a] | 9/27/2012 | Field Sample | TAA0087 | Y | 1 | 3.99E-05 | 87 |
| AD-200653 | T22 | DEQ | TA-20427 | 10/17/2012 | Field Sample | TAA0089 | Y | 2 | 7.98E-05 | 89 |
| AD-201580 | T21QC | Fire Station QC | TA-20450 | 11/27/2012 | Field Duplicate | TAA0093 | Y | 1 | 3.96E-05 | 93 |
| AD-201580 | T21 | Ambient Air Station - FIRE STATION | TA-20479 | 3/15/2013 | Field Sample | TAA0098 | Y | 2 | 7.98E-05 | 98 |
| AD-200653 | T22 | Ambient Air Station - DEQ | TA-20480 | 3/15/2013 | Field Sample | TAA0098 | Y | 1 | 3.99E-05 | 98 |
| AD-200653 | T22QC | Ambient Air Station - DEQ QC | TA-20481 | 3/15/2013 | Field Duplicate | TAA0098 | Y | 1 | 3.99E-05 | 98 |
| AD-201138 | T24 | Ambient Air Station - Jordan Residence | TA-20483 | 3/15/2013 | Field Sample | TAA0098 | Y | 1 | 3.99E-05 | 98 |

Note:
LA Libby Amphibole

The following presents a summary of Year 1 LA detections by station location.

- Station T1: 2 LA detections - sampling period 12 (7.37E-05 s/cc) and 17 (3.96E-05 s/cc )
- Station T2: 4 LA detections – sampling period 9 (3.77E-05 s/cc), 10 (8.81E-05 s/cc), 12 (3.77E-05), and 15 (3.86E-05 s/cc)
- Station T3: 1 LA detection – sampling period 12 (3.68E-05 s/cc)
- Station T4: 3 LA detections – sampling period 1 (1.56E-04 s/cc), 13 (3.74E-05 s/cc), and 18 (3.96E-05 s/cc)
- Station T5: 2 LA detections – sampling period 1 (1.75E-04 s/cc) and 35 (3.97E-05 s/cc)
- Station T6: 1 LA detection – sampling period 14 (3.57E-05 s/cc)
- Station T7: 2 LA detections – sampling period 12 (3.63E-05 s/cc) and 14 (3.63E-05 s/cc)
- Station 4QC: 3 LA detections – sampling period 1 (1.20E-04 s/cc), 11 (1.58E-04 s/cc), and 14 (7.15E-05 s/cc)

Table 10-8 provides a summary of all LA detections and Figure 5-1 provides the AAS station locations for AAS program years 1-3.

#### *10.3.2.2 Year 2 Analytical Results*

Year 2 of the AAS program included sampling periods 37 through 72. Year 2 results indicated the presence of LA in a total of nine samples with LA identified at four of the seven station locations and one of the mobile co-located stations. LA detections included a range of 1 to 2 structures per sample and concentrations ranging from 3.85E-05 to 2.67E-04 s/cc. The following presents a summary of Year 2 LA detections by station location.

- Station T11: 1 LA detection - sampling period 67 (3.92E-05 s/cc)
- Station T12: 3 LA detections – sampling period 53 (3.97E-05 s/cc), 59 (3.90E-05 s/cc), and 67 (3.92E-05)
- Station T13: No LA detections
- Station T14: 3 LA detections – sampling period 61 (3.99E-05 s/cc), 67 (2.67E-04 s/cc), and 68 (7.96E-05 s/cc)
- Station T15: 1 LA detections – sampling period 69 (3.85E-05 s/cc)
- Station T16: No LA detections
- Station T17: No LA detections
- Station 11QC: 1 LA detection – sampling period 44 (7.76E-05 s/cc)

Table 10-8 provides a summary of all LA detections and Figure 5-1 provides the AAS station locations for AAS program years 1 through 3.

#### *10.3.2.3 Year 3 Analytical Results*

Year 3 of the AAS program included sampling periods 73 through 101. Year 3 results indicated the presence of LA in a total of 16 samples with LA identified at all 4 of the station locations and 2 of the 4 mobile co-located stations. LA detections included a range of 1 to 3 structures per sample and concentrations ranging from 3.47E-05 to 1.08E-04 s/cc. The following presents a summary of Year 3 LA detections by station location.

- Station T21: 2 LA detection - sampling period 81 (3.47E-05 s/cc) and 98 (7.98E-05 s/cc)
- Station T22: 4 LA detections – sampling period 75 (7.99E-05 s/cc), 85 (3.97E-05 s/cc), 89 (7.98E-05 s/cc), and 98 (3.99E-05)
- Station T23: 2 LA detections - sampling period 78 (4.00E-05 s/cc) and 87 (3.99E-05 s/cc)
- Station T24: 2 LA detections - sampling period 87 (3.99E-05 s/cc) and 98 (3.99E-05 s/cc)
- Station T21QC: 4 LA detections – sampling period 73 (4.00E-05 s/cc), 81 (1.08E-04 s/cc), 93 (3.96E-05 s/cc), and 98 (3.99E-05)
- Station T22QC: No LA detections

Table 10-8 provides a summary of all LA detections and Figure 5-1 provides the AAS station locations for AAS program years 1 through 3.

## 10.4 OUTDOOR ACTIVITY-BASED SAMPLING RESULTS

The primary objective of outdoor ABS in OU7 was to obtain data needed to complete an HHRA to assess threats to human health from both residential and community-wide recreational sources of LA in air. Other outdoor scenarios such as outdoor exposures to commercial workers were considered similar enough to those of OU4 that the OU4 ABS commercial worker data were used for both OUs. HHRA findings will be used to prepare an FS, and for remedy selection. Residential properties used in the outdoor ABS scenarios in OU7 fall into two categories as described in the ABS SAP for OU7 (Tetra Tech 2011b):

- Properties for which soil removal is not required based on removal criteria specified in EPA (2003) and DEQ (2009a)

- Properties for which soil removal had been completed

A total of 495 ABS air samples (including field QC samples) were collected in 2011 under the three residential-disturbance scenarios and three community-wide disturbance scenarios described in Section 6.1.2. Two outdoor ABS sampling events were conducted for each scenario, one in wet season conditions and one in dry season conditions.

Detected ABS results for non-removal residential-properties appear in Table 10-9. Detected ABS results for post-removal residential properties appear in Table 10-10. Detected ABS results for community-wide disturbance scenarios appear in Table 10-11. The driving scenario contained the highest number of LA detections, with most detections occurring in the dry season sampling event.

Outdoor ABS results, along with indoor ABS results and outdoor ambient air results, were used in the HHRA.

**TABLE 10-9: DETECTED RESULTS FOR OUTDOOR ABS SCENARIOS AT NON-REMOVAL RESIDENTIAL PROPERTIES**

| Location | Sample ID | Season | Location Type | Location Description | Personnel Task | Scenario Description | Number of Structures | Result (s/cc) |
|---|---|---|---|---|---|---|---|---|
| AD-200438 | TB-00538 | DRY | Specific Use Area | Flowerbed | Digging, Rototilling | Gardening in residential yards | 1 | 2.20E-04 |
| AD-200458 | TB-00296 | WET | Common Use Area | Yard | Raking, Digging, Mowing | Yard work in residential yards | 1 | 2.16E-04 |
| AD-200458 | TB-00300 | WET | Specific Use Area | Garden | Digging, Rototilling | Gardening in residential yards | 1 | 2.16E-04 |
| AD-201096 | TB-00144 | WET | Specific Use Area | Driveway (unpaved) | Digging, Bicycling (child) | Bicycling and playing on unpaved driveways | 2 | 4.37E-04 |
| AD-201096 | TB-00660 | DRY | Common Use Area | Yard | Raking, Digging, Mowing | Yard work in residential yards | 1 | 2.13E-04 |
| AD-201096 | TB-00665 | DRY | Common Use Area | Former Garden | Digging, Rototilling | Gardening in residential yards | 2 | 9.98E-04 |
| AD-201096 | TB-00670 | DRY | Specific Use Area | Driveway (unpaved) | Digging, Bicycling (child) | Bicycling and playing on unpaved driveways | 4 | 8.59E-04 |

Note:

s/cc Structures per cubic centimeter

**TABLE 10-10: DETECTED RESULTS FOR OUTDOOR ABS SCENARIOS AT REMOVAL RESIDENTIAL PROPERTIES**

| Location | Sample ID | Season | Location Type | Location Description | Personnel Task | Scenario Description | Number of Structures | Result (s/cc) |
|---|---|---|---|---|---|---|---|---|
| AD-200594 | TB-00878 | DRY | Common Use Area | Yard | Raking, Digging, Mowing | Yard work in residential yards | 2 | 4.39E-04 |
| AD-200634 | TB-00490 | WET | Specific Use Area | Driveway (unpaved) | Digging, Bicycling (child) | Bicycling and playing on unpaved driveways | 1 | 2.12E-04 |
| AD-200634 | TB-00893 | DRY | Specific Use Area | Driveway (unpaved) | Digging, Bicycling (child) | Bicycling and playing on unpaved driveways | 1 | 2.18E-04 |
| AD-200759 | TB-00955 | DRY | Common Use Area | Yard | Raking, Digging, Mowing | Yard work in residential yards | 1 | 2.14E-04 |
| AD-200759 | TB-00963 | DRY | Specific Use Area | Driveway (unpaved) | Digging, Bicycling (child) | Bicycling and playing on unpaved driveways | 1 | 2.15E-04 |
| AD-200880 | TB-00967 | DRY | Specific Use Area | Lean-To | Digging, Bicycling (child) | Bicycling and playing on unpaved driveways | 1 | 2.15E-04 |

Note:

s/cc Structures per cubic centimeter

**TABLE 10-11: DETECTED RESULTS FOR OUTDOOR ABS COMMUNITY-WIDE SCENARIOS**

| Location | Sample ID | Season | Location Description | Personnel Task | Scenario Description | Number of Structures | Result (s/cc) |
|---|---|---|---|---|---|---|---|
| AD-200335 | TB-00597 | DRY | Community | Recreational sports & Ball field | Recreational activities at parks and school yards | 1 | 2.20E-04 |
| AD-OU7NA | TB-00563 | DRY | Community | Driving | Driving on paved and unpaved roads and alleys | 6 | 1.30E-03 |
| AD-OU7NA | TB-00565 | DRY | Community | Driving | Driving on paved and unpaved roads and alleys | 2 | 4.09E-04 |
| AD-OU7NA | TB-00568 | DRY | Community | Driving | Driving on paved and unpaved roads and alleys | 2 | 4.14E-04 |
| AD-OU7NA | TB-00570 | DRY | Community | Driving | Driving on paved and unpaved roads and alleys | 3 | 6.39E-04 |
| AD-OU7NA | TB-00573 | DRY | Community | Driving | Driving on paved and unpaved roads and alleys | 4 | 8.73E-04 |
| AD-OU7NA | TB-00578 | DRY | Community | Driving | Driving on paved and unpaved roads and alleys | 13 | 2.80E-03 |
| AD-OU7NA | TB-00581 | DRY | Community | Driving | Driving on paved and unpaved roads and alleys | 1 | 2.07E-04 |
| AD-200335 | TB-00414 | WET | Community | Recreational sports & ball field | Recreational activities at parks and school yards | 3 | 6.37E-04 |
| AD-OU7NA | TB-00322 | WET | Community | Bicycling (infant in trailer) | Bicycling on paved and unpaved roads and alleys | 1 | 2.20E-04 |

Note:
s/cc Structures per cubic centimeter

## 10.5 INDOOR ACTIVITY-BASED SAMPLING RESULTS

The primary objective of indoor ABS in OU7 was to obtain data needed to complete the HHRA to assess threats to human health from residential sources of LA in indoor air. Field methods and data quality objectives for indoor air ABS are provided in the indoor ABS SAP for OU7 (Tetra Tech 2012a). HHRA findings are being used to prepare an FS, and for remedy selection. Residential properties in OU7 fall into two categories:

- Properties for which removal was not required based on removal criteria specified in EPA (2003) and DEQ (2009a)
- Properties for which removal had been completed

A total of 138 indoor ABS air samples (including field QC samples) were collected in 2012 and 2013 under the two residential-disturbance scenarios described in Section 7.1.2.

Detected indoor ABS results for non-removal residential properties appear in Table 10-12. Detected indoor ABS results for post-removal residential-properties appear in Table 10-13. As shown on the tables, the active ABS scenario had the highest frequency of detections. Also, total LA detections of non-removal properties (Table 10-12) were less than those properties where indoor removals were conducted (Table 10-13).

**TABLE 10-12: DETECTED RESULTS FOR INDOOR ABS SCENARIOS AT NON-REMOVAL RESIDENTIAL PROPERTIES**

| Location | Sample ID | Season | Location Type | Location Description | Personnel Task | Scenario Description | Number of Structures | Result (s/cc) |
|---|---|---|---|---|---|---|---|---|
| AD-200340 | TB-01038 | WET | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 3 | 1.37E-04 |
| AD-200340 | TB-00908 | DRY | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 1 | 4.56E-05 |
| AD-200340 | TB-01039 | WET | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 1 | 1.92E-05 |
| AD-200436 | TB-01027 | WET | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 5 | 9.63E-05 |
| AD-200436 | TB-00823 | DRY | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 2 | 9.15E-05 |
| AD-200514 | TB-01021 | WET | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 2 | 3.85E-05 |
| AD-200554 | TB-00841 | DRY | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 1 | 7.12E-05 |
| AD-200554 | TB-00982 | WET | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 1 | 4.61E-05 |
| AD-200554 | TB-00842 | DRY | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 2 | 3.84E-05 |
| AD-200632 | TB-00974 | DRY | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 1 | 1.92E-05 |
| AD-200687 | TB-01023 | WET | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 5 | 2.30E-04 |
| AD-200687 | TB-01024 | WET | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 1 | 1.90E-05 |
| AD-200785 | TB-00850 | WET | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 1 | 4.60E-05 |
| AD-200785 | TB-00820 | DRY | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 1 | 4.60E-05 |
| AD-200911 | TB-00833 | DRY | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 4 | 7.70E-05 |
| AD-200911 | TB-00832 | DRY | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 2 | 5.54E-05 |

Note:

s/cc Structures per cubic centimeter

**TABLE 10-13: DETECTED RESULTS FOR INDOOR ABS SCENARIOS AT REMOVAL RESIDENTIAL PROPERTIES**

| Location | Sample ID | Season | Location Type | Location Description | Personnel Task | Scenario Description | Number of Structures | Result (s/cc) |
|---|---|---|---|---|---|---|---|---|
| AD-200005 | TB-00897 | DRY | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 1 | 4.60E-05 |
| AD-200005 | TB-01030 | WET | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 1 | 1.93E-05 |
| AD-200007 | TB-00555 | DRY | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 5 | 2.30E-04 |
| AD-200007 | TB-00556 | DRY | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 6 | 9.53E-05 |
| AD-200007 | TB-01033 | WET | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 1 | 1.92E-05 |
| AD-200265 | TB-01000 | WET | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 2 | 5.54E-05 |
| AD-200342 | TB-00997 | WET | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 1 | 2.79E-05 |
| AD-200342 | TB-00836 | DRY | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 1 | 1.87E-05 |
| AD-200510 | TB-00905 | DRY | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 2 | 8.72E-05 |
| AD-200533 | TB-00970 | DRY | Primary Building | Residential | Vacuuming, Dusting, Simulated Play, Walking | Active | 12 | 2.84E-04 |
| AD-200533 | TB-00971 | DRY | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 3 | 4.81E-05 |
| AD-201150 | TB-00902 | DRY | Primary Building | Residential | Vacuuming, Dusting, General Cleaning, Simulated Play, Walking | Active | 2 | 5.54E-05 |
| AD-201150 | TB-00903 | DRY | Primary Building | Residential | Walking room to room, Sitting, Reading, Watching TV | Passive | 1 | 1.92E-05 |

Note:

s/cc Structures per cubic centimeter

## 10.6 BACKGROUND SOIL STUDY

As discussed in Section 8, a background soil study was performed for OU7 to determine the possible presence of LA in background soils associated with naturally-deposited glacial sediments in and around OU7. The resulting background soil study report is included as Attachment 2 to this RI report. The following conclusions were drawn from the results of the background soils study:

- LA has been detected in background soils in and around OU7 that are not thought to be affected by anthropogenic releases from vermiculite mining and processing activities.
- The types of LA structures in background soils within the Kootenai Valley indicate that a portion of the LA structures observed likely do not originate from the Libby vermiculite ore body.
- The concentration of LA in background soils (≤ 0.02 percent) is well below the detection limit of PLM-VE, but is able to be reliably detected by Fluidized Bed Asbestos Segregator (FBAS) following preparation using the rock flour method and analysis by TEM. FBAS soil preparation method appears to be a more sensitive metric of LA detection in soil than either ABS or field visible vermiculite observations.

The results support the conclusion that there is a non-zero level of LA in soils in and around OU7 that is not attributable to vermiculite mining and processing activities associated with the Libby Mine. Further, these results support the data reported by Adams and others 2010; Langer et al 2010; and Gunter and Sanchez 2009, indicating that low level detections of amphibole fibers in soils within the Kootenai Valley originated from normal geologic, soil building processes unrelated to mining and milling of vermiculite ore from Vermiculite Mountain.

# 11.0 CONTAMINANT FATE AND TRANSPORT

Evaluation of fate and transport of contaminants is based on results of site physical characteristics, source characteristics, and extent of contamination investigations. This section discusses the important factors involved in fate and transport of LA in OU7.

## 11.1 RELEASES TO THE ENVIRONMENT

Asbestos occurs naturally in the environment and may be released to water and air from erosion and the weathering of natural deposits of asbestos-bearing rocks. Asbestos is more likely to be released to the environment when these natural deposits are disturbed, such as during mining operations. Asbestos is released to the environment from the crushing, screening, and milling of ore, the processing of asbestos products, the use of asbestos-containing materials, and the transport and disposal of asbestos-containing wastes (Agency for Toxic Substances and Disease Registry [ATSDR] 2001).

## 11.2 TRANSPORT AND DEPOSITION

Once asbestos fibers enter the environment from either natural or artificial sources, they tend to settle out of the air or water and deposit onto soil and sediment (EPA 1979; Millette 1979). Asbestos fibers can be re-suspended into the air or water following soil and sediment disturbances. The rate at which asbestos particles settle out of the air or water depends on their size. Jaenicke (1980) reported that the residence time for a particle to remain airborne is shortest for the smallest particles (0.001 µm in diameter) and largest particles (100 µm in diameter), and greatest for particles ranging from 0.1 to1 µm in diameter. Fibers in this size range could be transported long distances in air. Thin asbestos fibers could remain airborne for hours or even days before settling (Webber *et al*. 2006).

In water, asbestos fibers may also travel long distances from the point of origin, depending on the surface chemistry and detailed mineralogy of the fiber (EPA 1979). For example, tailings containing asbestos from taconite mining were released into Lake Superior and asbestos fibers were detected in the drinking water of Duluth, Minnesota, about 75 miles away from the point source (EPA 1979). Studies have shown that asbestos concentrations in surface water near natural chrysotile deposits in California are usually highest during or after rain events, which reflects re-entrainment of asbestos from river sediments and watershed erosion (Webber and Covey 1991). At the Libby Asbestos

Superfund Site, surface water monitoring of streams near the mine (OU3) show similar results; LA concentrations in water tend to vary seasonally, with the highest concentrations reported in the spring during periods of runoff. After eroded asbestos-contaminated soils enter the water, asbestos fibers will remain suspended in the water column even after soil particles have settled. Pilot studies to evaluate methods for concentrating LA fibers in soil using water elutriation demonstrated that most soil particles greater than 3 µm in size had settled out after 3 hours, while the asbestos fibers remained in suspension (TechLaw, Inc. 2011). Fiber interaction with natural organic matter may increase their precipitation (ATSDR 2001; EPA 1979). Studies have shown that asbestos fibers in water will adhere to algae, perhaps due to the positive surface charge of the fibers, which results in clumping and increased settling (Webber and Covey 1991). Because there may be a range of asbestos fiber sizes in water, and settling time differs by size, there may be a vertical distribution of asbestos fibers in large bodies of water (Chatfield and Dillon 1983). Even after settling, asbestos fibers in sediment can be re-entrained into water following sediment disturbances.

In soils, asbestos will tend to be retained at or near the surface. Movement of asbestos fibers through soils or from soil to surface water occurs during runoff or erosion. Asbestos fibers in soil are fairly immobile, and fibers less than 2 µm in diameter will tend to move at the same rate as clays (i.e., about 1 to 10 centimeters per 3,000 to 40,000 years) (EPA 1979). Asbestos fibers in soil can become suspended into the air by disturbing the contaminated soil. The releasability of asbestos from soil to air depends upon many factors, including the soil moisture content (drier conditions tend to increase releasability), vegetation coverage and condition (lush plant cover will tend to decrease releasability), and the intensity of the disturbance activity (higher intensity activities will yield higher releases).

Studies have also shown that asbestos fibers may also settle onto the outer surface of trees (bark) and become a reservoir for potential asbestos exposure (Ward *et al.* 2006; 2012). Extensive data on LA levels on the bark surface of trees has been collected in the forested area near the mine site and along the OU4 National Priorities List boundary (EPA 2013b). These data showed that tree bark surface loading levels of asbestos tended to be highest on trees collected closest to the mine (within about 3 to 4 miles), but fibers were also detected on trees located even 13 miles from the mine site (EPA 2013b). Asbestos fibers on the bark surface can become re-suspended to air following bark disturbances, such as during logging operations. Studies have also shown that asbestos fibers can become concentrated in ash following the burning of asbestos-contaminated wood (EPA 2012l; Ward *et al.* 2009), which itself can become a source of potential exposure.

Lastly, asbestos can be suspended into indoor air when source material (insulation, dust, carpets, etc.) is disturbed by cleaning, renovation or other general disruption.

## 11.3 TRANSFORMATION AND DEGRADATION IN THE ENVIRONMENT

Asbestos fibers are non-volatile and insoluble. They are transported and distributed by air and water and tend to persist under typical environmental conditions (ATSDR 2001). In general, asbestos is exceptionally resistant to thermal and chemical degradation. However, the range in the ability of different types of asbestos to persist in the environment varies. For instance, chrysotile asbestos is expected to degrade more readily than amphibole asbestos under certain environmental conditions (e.g., acidic environments) (ATSDR 2001). Possible transformation and degradation for each environmental medium are discussed below.

**Air:** Asbestos particles are not known to undergo any significant transformation or degradation in air (ATSDR 2001).

**Water:** Asbestos fibers are relatively stable in water and are not prone to significant chemical or biological degradation. However, some asbestos fibers may undergo chemical alteration and adsorb additional organic agents. In general, asbestos does not volatilize from water surfaces. In water, at low pH, chrysotile asbestos may undergo some dissolution, as magnesium hydroxide leaches from the outer brucite layer, but amphibole asbestos is expected to persist in aquatic environments virtually unchanged for long periods of time (ATSDR 2001).

**Soil:** In general, asbestos fibers are not known to undergo significant transformation or degradation in soil (ATSDR 2001).

**Bulk Asbestos-Containing Material (ACM):** Bulk AMC is subject to physical degradation through crushing or erosion that can generate fibers that are more mobile than the original material (ATSDR 2001).

# 12.0 RISK ASSESSMENT

An evaluation of potential exposures to, and risks from, LA will be included in the site-wide human health and ecological risk assessments for the Libby Asbestos Superfund Site. The site-wide risk assessments are stand-alone documents that support the feasibility study and ROD. As such, OU-specific risk assessment reports have not been developed. The *Site-Wide Human Health Risk Assessment* will evaluate potential risks to humans from exposures to LA under a variety of different exposure scenarios, including both indoor and outdoor exposure scenarios that may occur at the Site. Potential risks will be evaluated both alone and across multiple exposure scenarios as part of a cumulative exposure assessment. The *Site-Wide Ecological Risk Assessment* will evaluate potential risks to aquatic and terrestrial ecological receptors from exposures to LA that may be present in the environment at the Site.

# 13.0 CONCLUSIONS

This final RI report for OU7 summarizes RI activities and findings pursuant to the TAPE work plan (Tetra Tech 2007), the outdoor AAS work plan (Tetra Tech 2009a), the RDI SAP (Tetra Tech 2010a), the ABS SAP (Tetra Tech 2011b), the indoor ABS SAP (Tetra Tech 2012a), and the soil background study SAP (Tetra Tech 2012b). It does not include an HHRA or ecological risk assessment because they are being prepared separately on a Libby Asbestos Superfund Site-wide basis.

The RI was completed to assess the nature and extent of LA contamination within OU7. The information presented in this final RI report, together with the site-wide HHRA, will be used to prepare an FS. The FS will identify and evaluate remedial alternatives that can be used to remediate areas in OU7 that pose unacceptable risk to human health and the environment. The development of remedial alternatives requires the following:

(1) Identifying federal and state applicable or relevant environmental requirements, criteria, and limitations

(2) Identifying remedial action objectives

(3) Identifying potential treatment, resource recovery, and containment technologies that will satisfy these objectives

(4) Screening the technologies based on their effectiveness, implementability, and cost; and assembling technologies and their associated containment or disposal requirements into alternatives for the contaminated media at the site

Typically, a range of remedial alternatives are developed during the FS process varying primarily in the long-term effectiveness of the selected technology or treatment of the site contamination. Given the unique situation in OU7 where LA is the known contaminant, remedial actions to address the LA will likely be limited to removal/disposal options. Once sufficient data are available, remedial alternatives will be evaluated in detail with respect to evaluation criteria that DEQ has developed to address the statutory requirements. The results of the detailed analysis will be summarized and presented so that an appropriate remedy consistent with federal, state, and local requirements can be selected.

In general, the results of the FS will then lead to the selection of a preferred remedy or remedies, development of a proposed plan, public comment, preparation of a ROD, development of the remedial design, and implementation of the remedial action.

The information presented in this final RI report indicates that LA from the Libby mine is present in OU7 as a result of mining activities, inadvertent transport and dispersal from the Libby mine by residents within OU7, commercial use of vermiculite from the Libby mine, and natural processes. Evaluation of the health effects to residents and ecological receptors within OU7 is the subjects of a separate report, which, together with this RI report, will provide the necessary information to formulate remedial alternatives for OU7.

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# FIGURES

**LEGEND**

- OPERABLE UNITS
- LINCOLN COUNTY
- TOWNS
- STATE HIGHWAY
- US HIGHWAY
- RAILROAD
- WATER

N

0 5 10 15 20 Miles

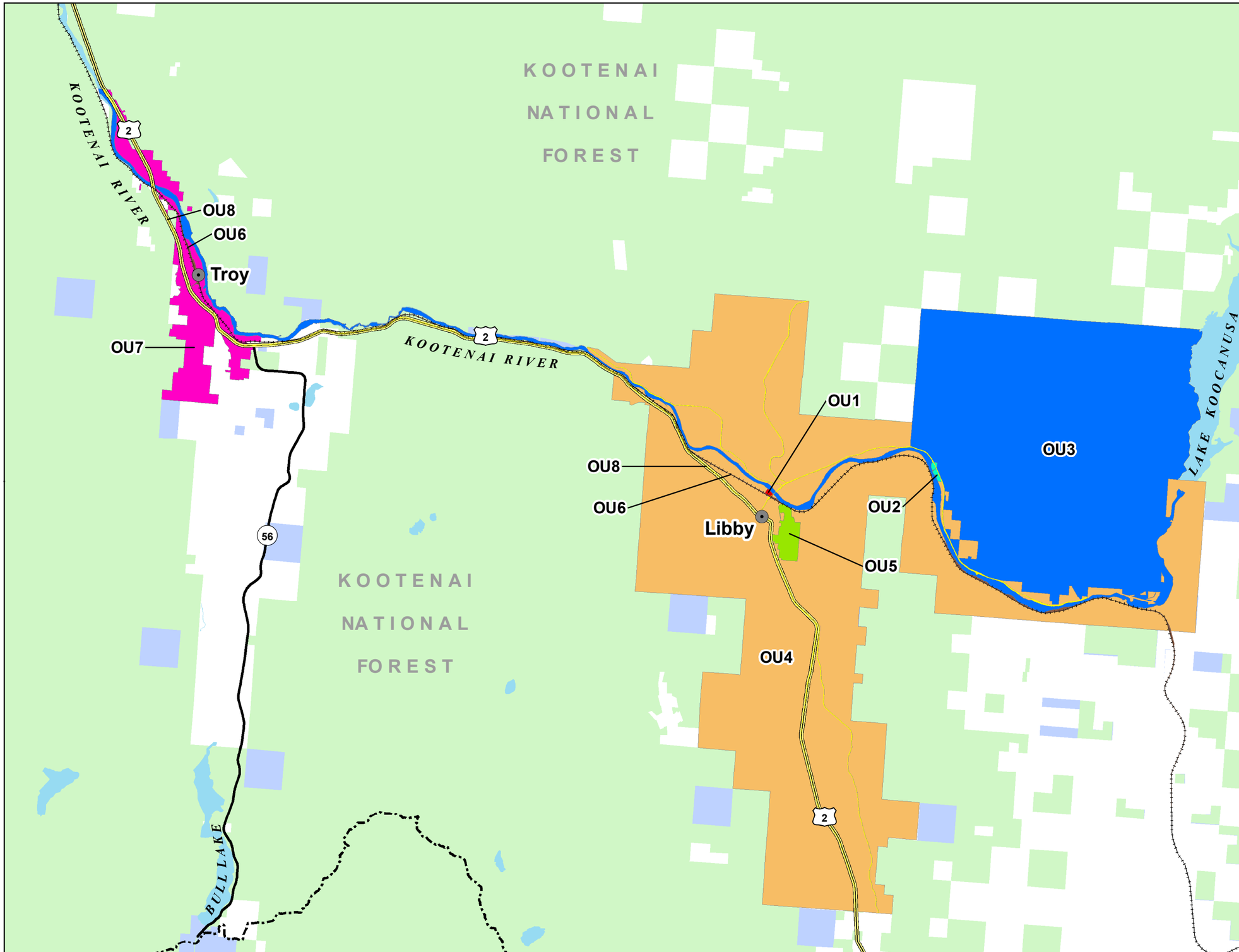
Remedial Investigation Report
Libby Asbestos Superfund Site, Libby, Montana

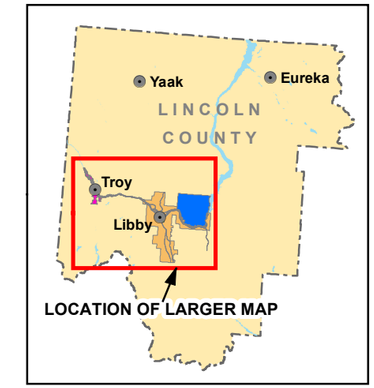
**FIGURE 1-1**

SITE LOCATION MAP

TETRA TECH EM INC.

Figure_1-1_Site_Location_Map_021914.mxd - DWH - TTEMI HELENA




**LEGEND**

- TOWNS
- LINCOLN COUNTY
- STATE HIGHWAY
- US HIGHWAY
- RAILROAD
- NATIONAL FOREST LAND
- PRIVATE LAND
- STATE LAND
- WATER

**OPERABLE UNITS**

- OU1 - FORMER EXPORT PLANT
- OU2 - FORMER SCREENING PLANT
- OU3 - MINE AND KOOTENAI RIVER
- OU4 - LIBBY
- OU5 - FORMER STIMSON LUMBER
- OU6 - BNSF RAIL CORRIDOR
- OU7 - TROY
- OU8 - STATE HIGHWAY CORRIDORS

N

0 5 Miles

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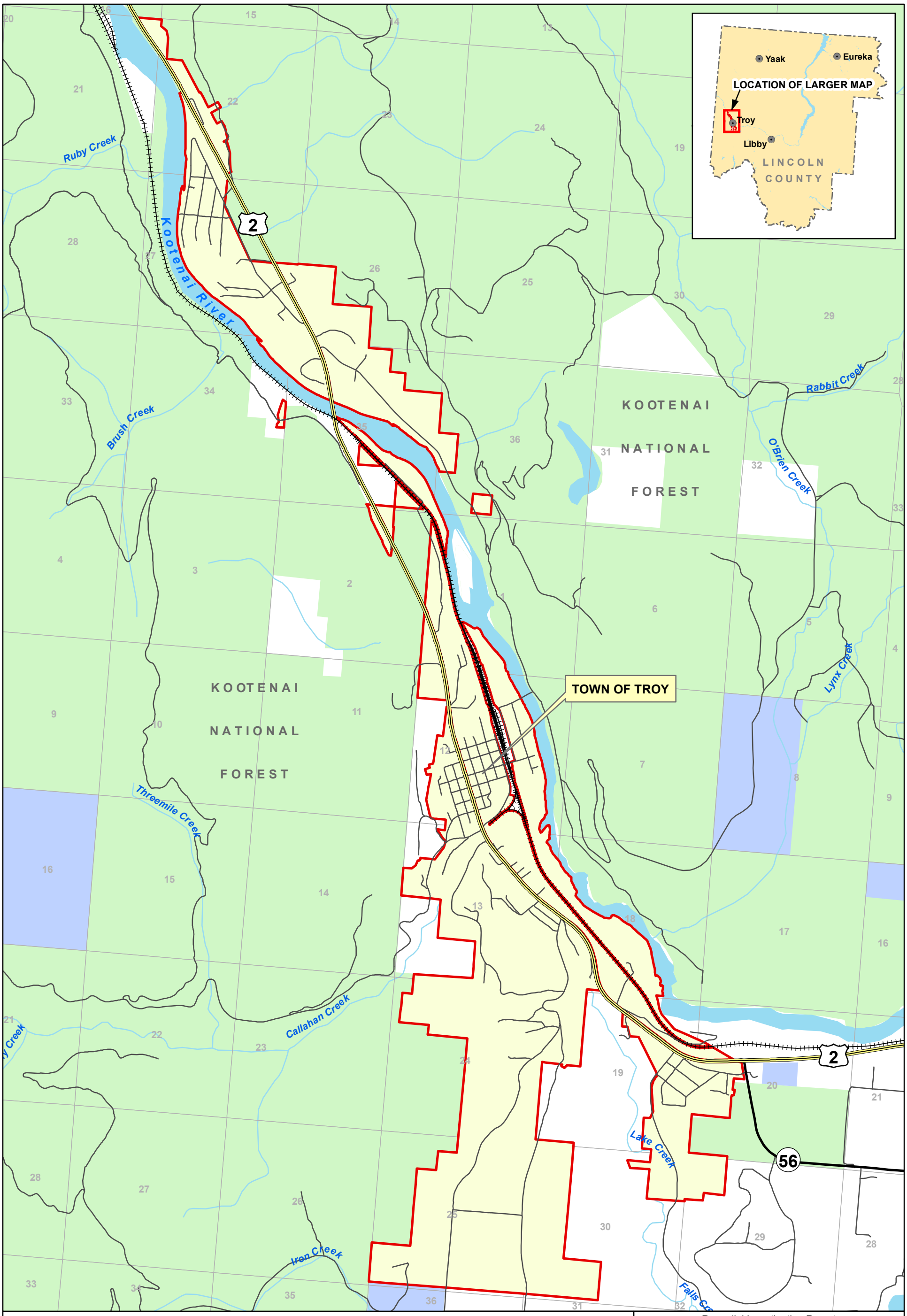
**FIGURE 1-2**

LOCATION OF ALL OPERABLE UNITS

TETRA TECH EM INC.

Figure_1-2_All_OUs_031014.mxd - DWH - TTEMI HELENA




**LEGEND**

- OPERABLE UNIT 7
- STATE HIGHWAY
- US HIGHWAY
- OTHER ROADS
- RAILROAD
- NATIONAL FOREST LAND
- PRIVATE LAND
- STATE LAND
- WATER

N

0 1 Miles

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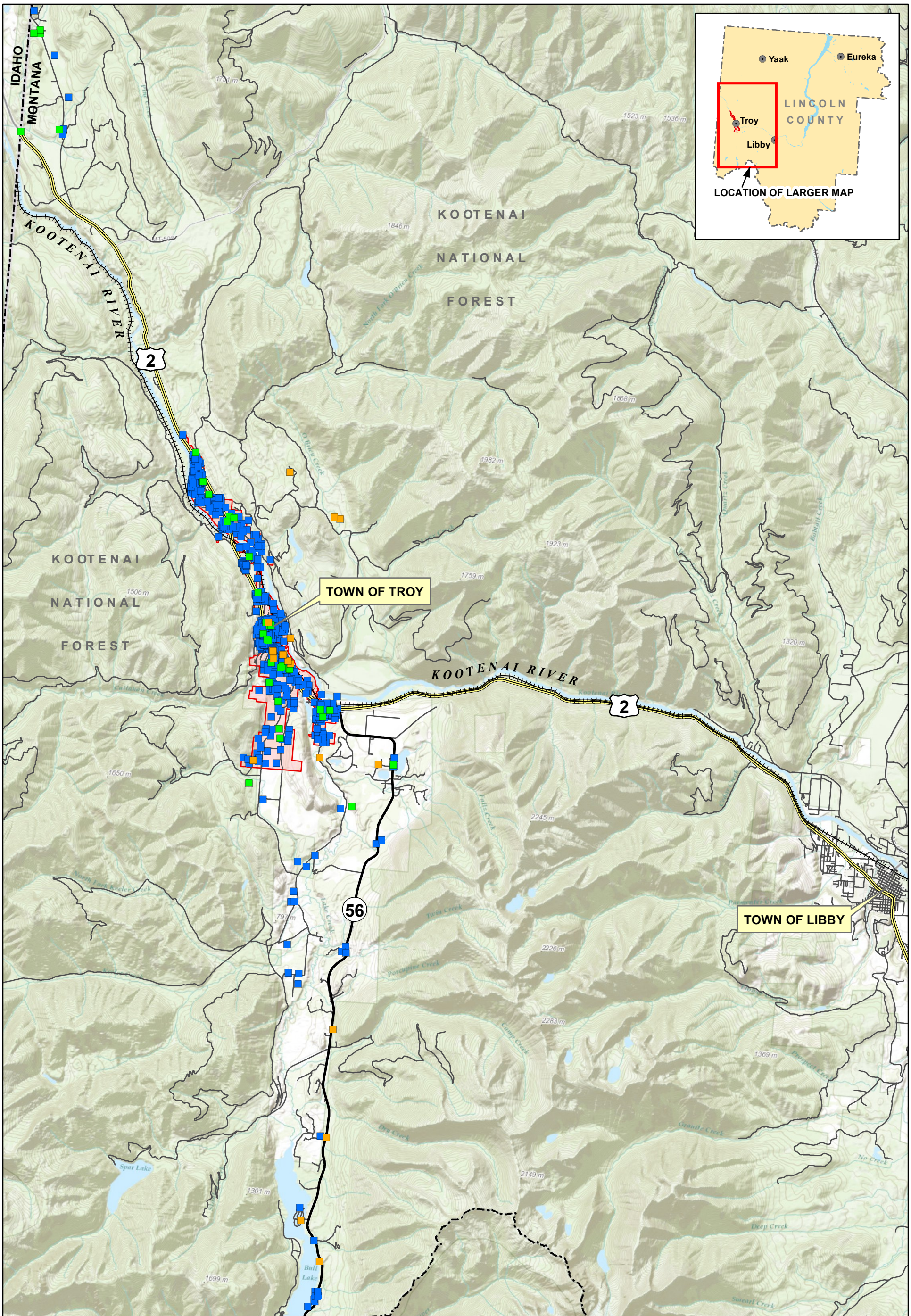
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 1-3**
OPERABLE UNIT 7
BOUNDARY

TETRA TECH EM INC.

Figure_1-3_OU7_Boundary_022114.mxd - DWH - TTEMI HELENA




Topographic Map Sources: Esri, DeLorme, NAVTEQ, TomTom, Intermap, increment P Corp., GEBCO, USGS, FAO, NPS, NRCAN, GeoBase, IGN, Kadaster NL, Ordnance Survey, Esri Japan, METI, Esri China (Hong Kong), swisstopo, and the GIS User Community

**LEGEND**

- OPERABLE UNIT 7
- LINCOLN COUNTY BOUNDARY
- STATE HIGHWAY
- US HIGHWAY
- OTHER ROADS
- RAILROAD
- INSPECTED IN 2012, 2013
- INSPECTED IN 2011
- INSPECTED PRIOR TO 2011
- TAPE TROY ASBESTOS PROPERTY EVALUATION

N

0 5 Miles

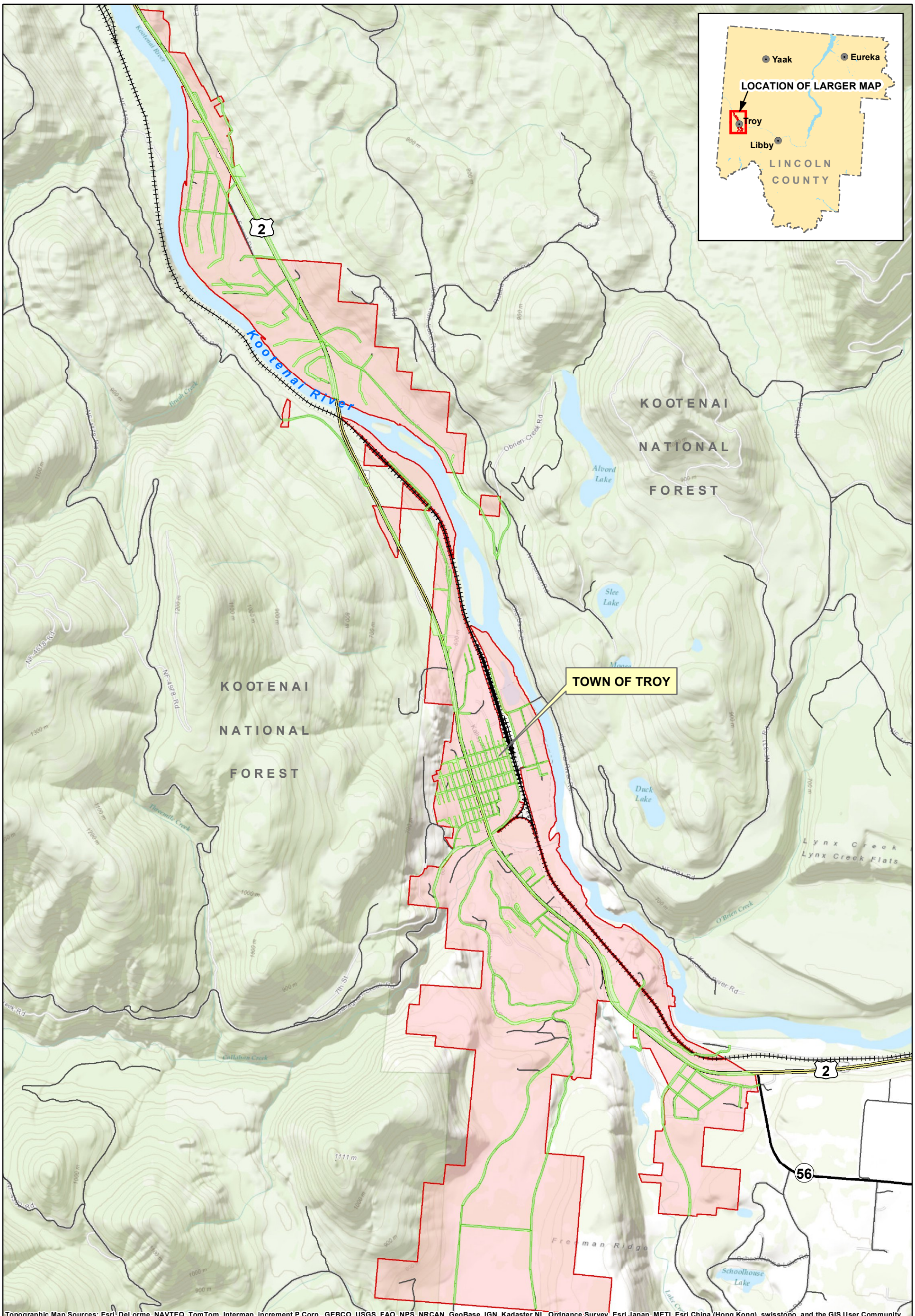
Remedial Investigation Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 4-1**

OPERABLE UNIT 7
TAPE PARCELS

TETRA TECH EM INC.

Figure_4-1_Inspected_Parcels_TAPE_031014.mxd - DWH - TTEMI HELENA




**LEGEND**

- OPERABLE UNIT 7
- ROAD AND ALLEY PARCELS INSPECTED (2009)
- STATE HIGHWAY
- US HIGHWAY
- OTHER ROADS
- RAILROAD
- TAPE — TROY ASBESTOS PROPERTY EVALUATION

N

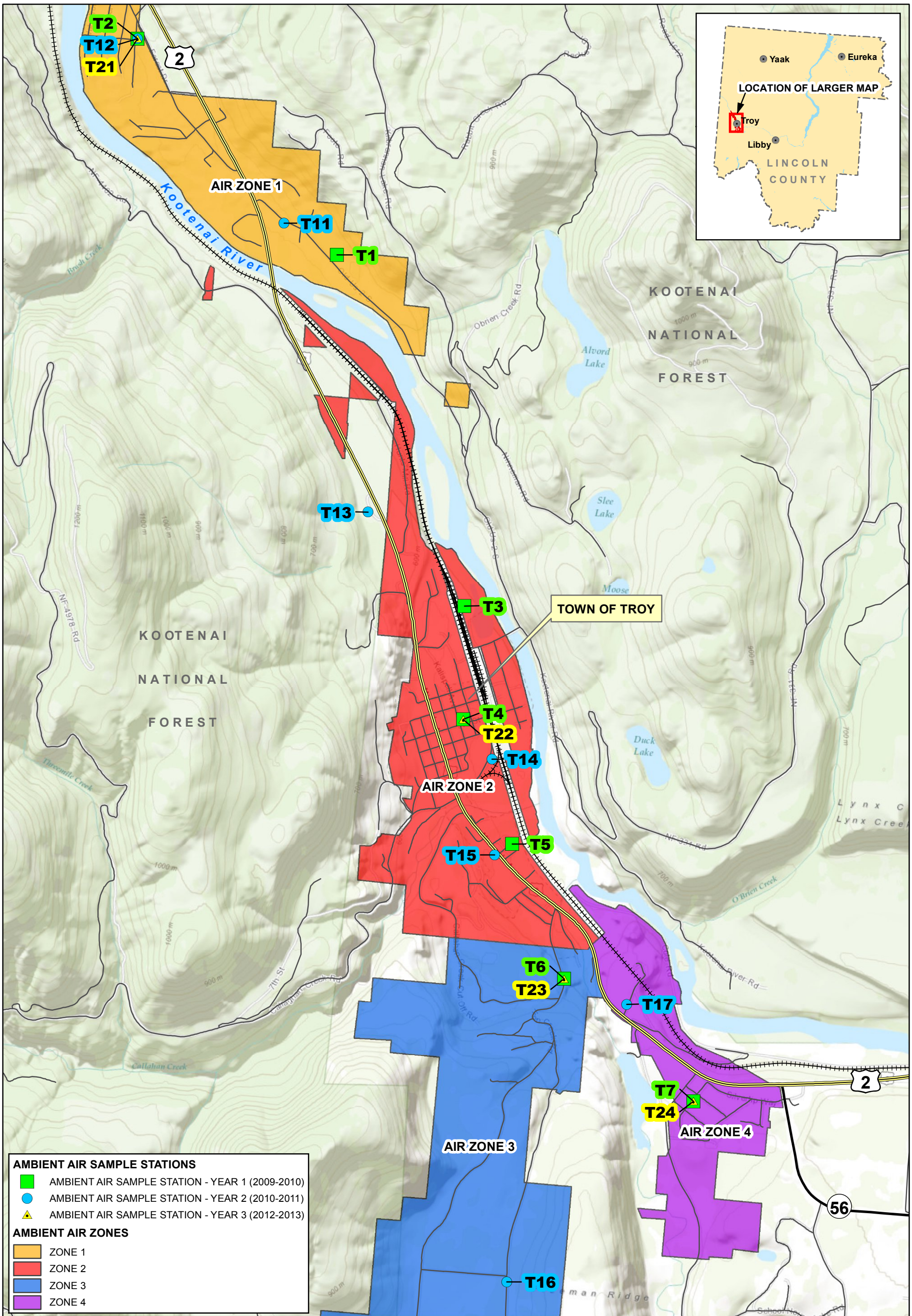
0 — 1 Miles

Remedial Investigation Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 4-2**
OPERABLE UNIT 7
ROAD AND ALLEY TAPE PARCELS

TETRA TECH EM INC.

Figure_4-2_Road_Alley_Parcels_TAPE_031014.mxd - DWH - TTEMI HELENA




Topographic Map Sources: Esri, DeLorme, NAVTEQ, TomTom, Intermap, increment P Corp., GEBCO, USGS, FAO, NPS, NRCAN, GeoBase, IGN, Kadaster NL, Ordnance Survey, Esri Japan, METI, Esri China (Hong Kong), swisstopo, and the GIS User Community

**LEGEND**

- STATE HIGHWAY
- US HIGHWAY
- OTHER ROADS
- RAILROAD

N

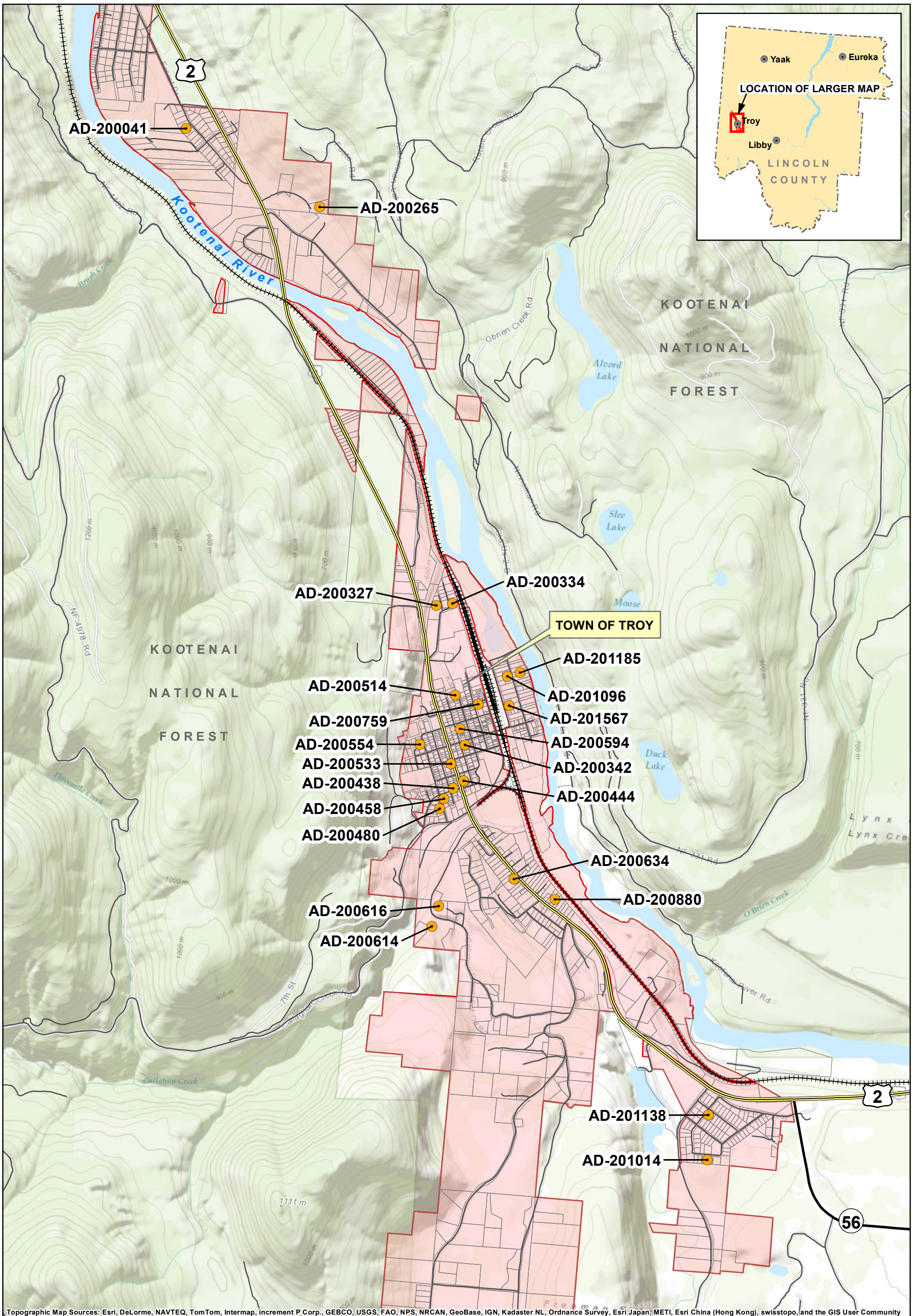
0 1 Miles

Remedial Investigation Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 5-1**
OPERABLE UNIT 7
AMBIENT AIR MONITORING PROGRAM
STATION LOCATIONS

TETRA TECH EM INC.

Figure_5-1_Ambient_Air_Monitoring_Station_Locations_022014_Topo.mxd - DWH - TTEMI HELENA




Topographic Map Sources: Esri, DeLorme, NAVTEQ, TomTom, Intermap, increment P Corp., GEBCO, USGS, FAO, NPS, NRCAN, GeoBase, IGN, Kadaster NL, Ordnance Survey, Esri Japan, METI, Esri China (Hong Kong), swisstopo, and the GIS User Community

**LEGEND**

- OPERABLE UNIT 7 BOUNDARY
- PARCEL BOUNDARY
- OUTDOOR RESIDENTIAL ACTIVITY-BASED SAMPLING LOCATION
- STATE HIGHWAY
- US HIGHWAY
- OTHER ROADS
- RAILROAD

N

0 1 Miles

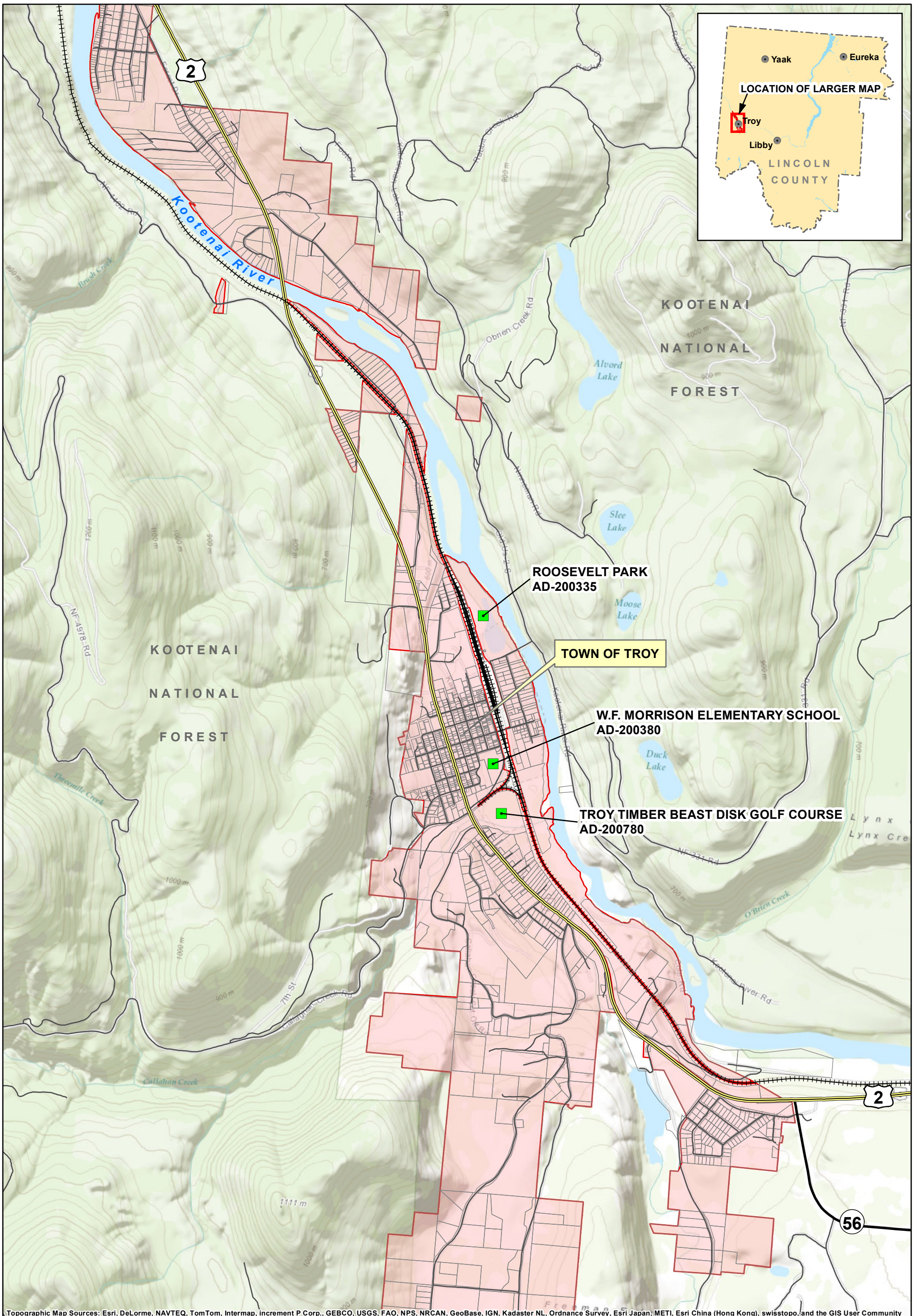
Remedial Investigation Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 6-1**

OPERABLE UNIT 7
OUTDOOR RESIDENTIAL
ACTIVITY-BASED SAMPLING LOCATIONS

TETRA TECH EM INC.

Figure_6-1_Outdoor_Residential_ABS_022114.mxd - DWH - TTEMI HELENA




**LEGEND**

- OPERABLE UNIT 7 BOUNDARY
- PARCEL BOUNDARY
- OUTDOOR RECREATIONAL ACTIVITY-BASED SAMPLING LOCATION
- STATE HIGHWAY
- US HIGHWAY
- OTHER ROADS
- RAILROAD

N

0 1 Miles

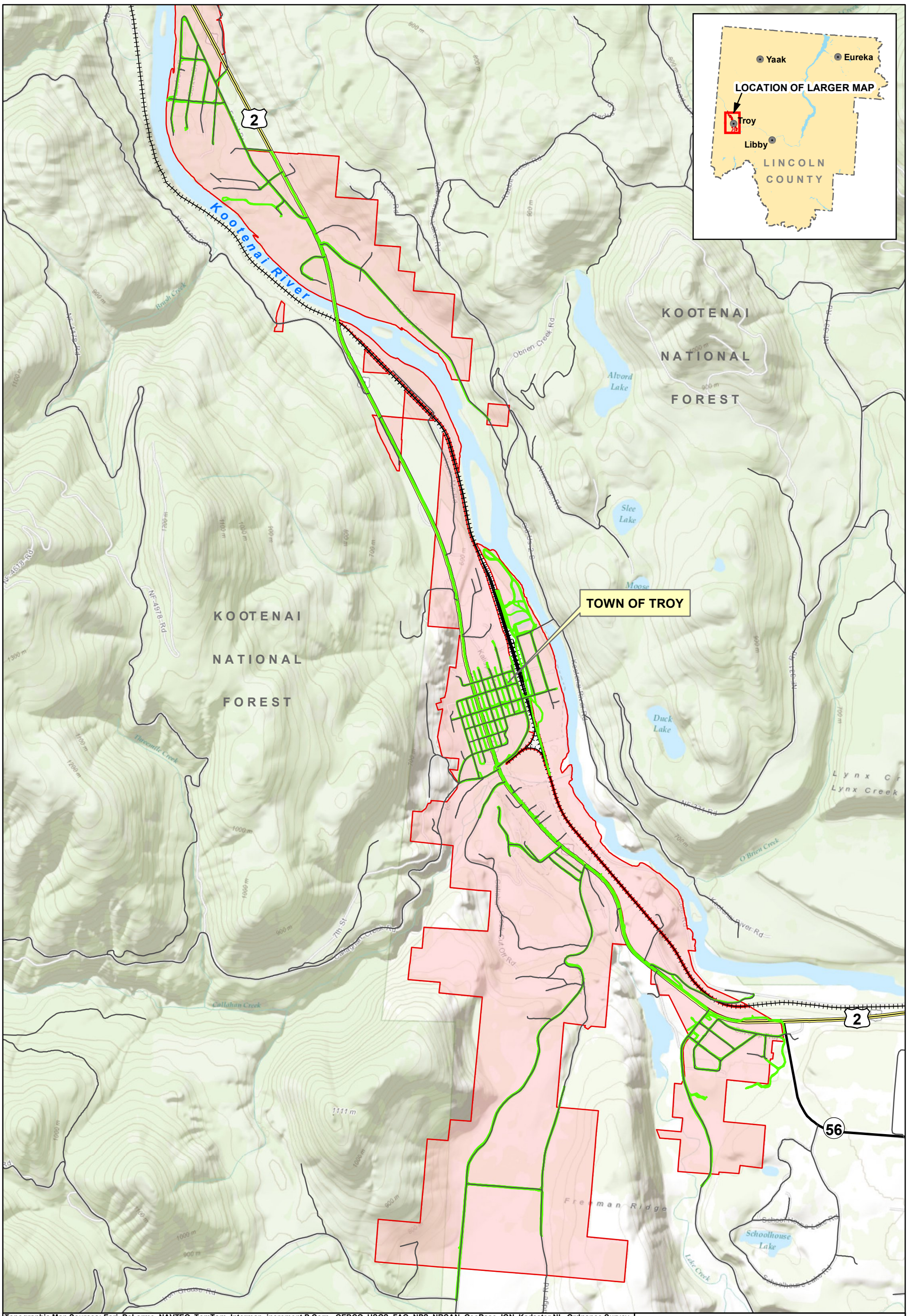
Remedial Investigation Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 6-2**

OPERABLE UNIT 7
OUTDOOR RECREATIONAL
ACTIVITY-BASED SAMPLING LOCATIONS

TETRA TECH EM INC.

Figure_6-1_Outdoor_Recreational_ABS_022514.mxd - DWH - TTEMI HELENA




Topographic Map Sources: Esri, DeLorme, NAVTEQ, TomTom, Intermap, increment P Corp., GEBCO, USGS, FAO, NPS, NRCAN, GeoBase, IGN, Kadaster NL, Ordnance Survey, Esri Japan, METI, Esri China (Hong Kong), swisstopo, and the GIS User Community

**LEGEND**

- OPERABLE UNIT 7
- OUTDOOR ACTIVITY-BASED SAMPLING ROUTE - BICYCLING (2011)
- STATE HIGHWAY
- US HIGHWAY
- OTHER ROADS
- RAILROAD

N

0 1 Miles

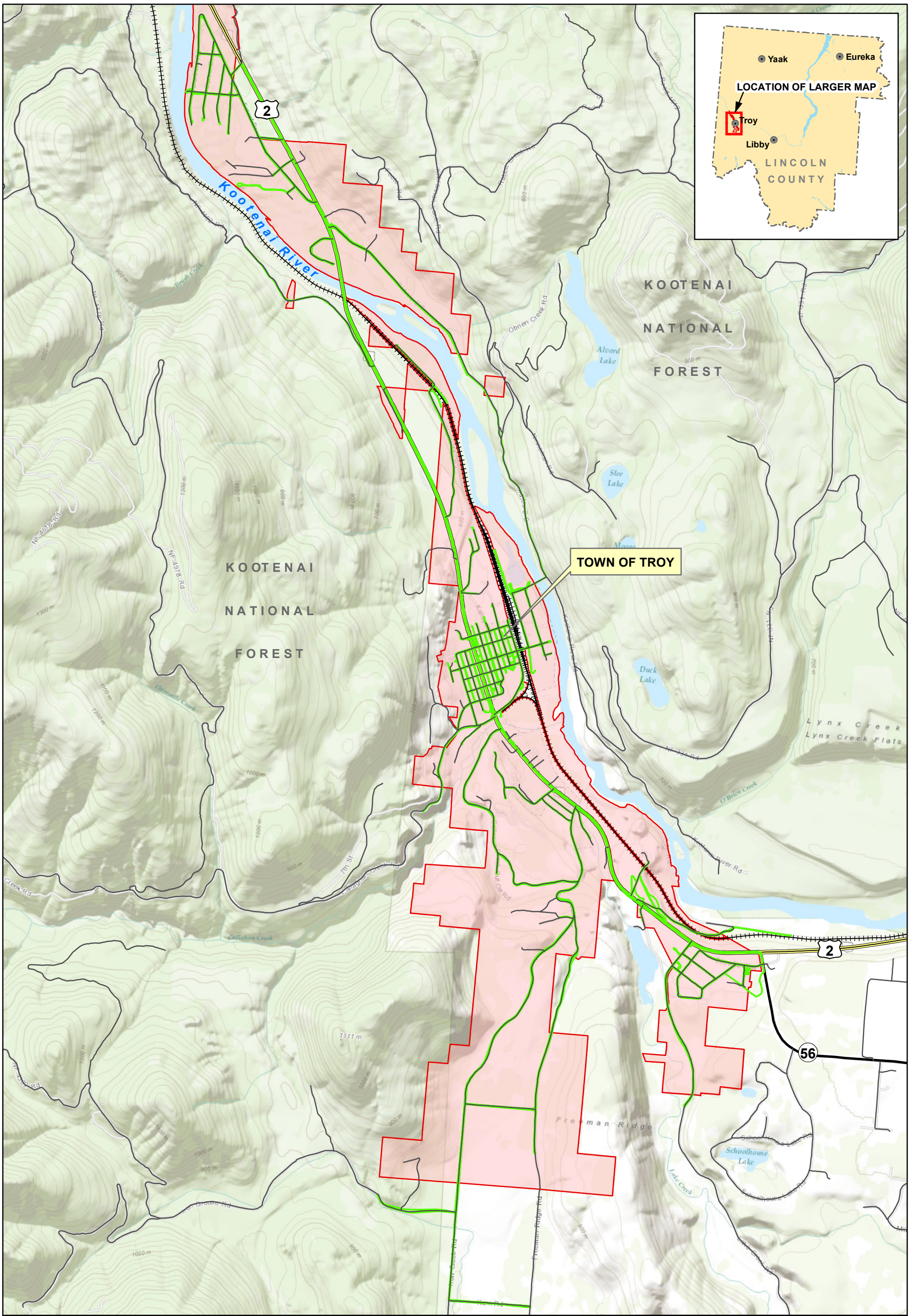
Remedial Investigation Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 6-3**

OPERABLE UNIT 7
OUTDOOR ACTIVITY-BASED
SAMPLING ROUTE - BICYCLING

TETRA TECH EM INC.

Figure_6-3_Outdoor_Biking_ABS_022114.mxd - DWH - TTEMI HELENA




Topographic Map Sources: Esri, DeLorme, NAVTEQ, TomTom, Intermap, increment P Corp., GEBCO, USGS, FAO, NPS, NRCAN, GeoBase, IGN, Kadaster NL, Ordnance Survey, Esri Japan, METI, Esri China (Hong Kong), swisstopo, and the GIS User Community

**LEGEND**

- OPERABLE UNIT 7
- OUTDOOR ACTIVITY-BASED SAMPLING ROUTE - DRIVING (2011)
- STATE HIGHWAY
- US HIGHWAY
- OTHER ROADS
- RAILROAD

N

0 1 Miles

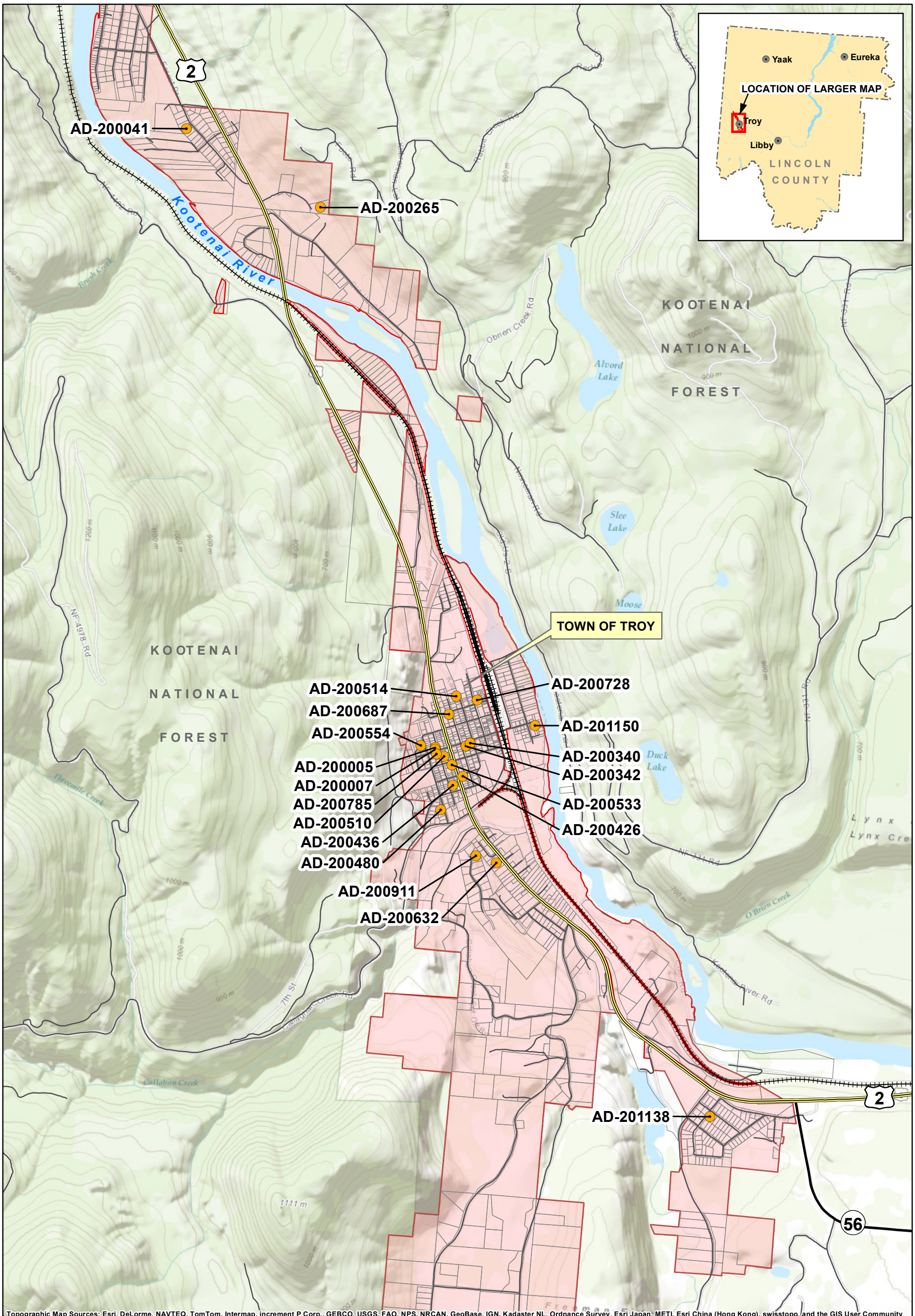
Remedial Investigation Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 6-4**

OPERABLE UNIT 7
OUTDOOR ACTIVITY-BASED
SAMPLING ROUTE - DRIVING

TETRA TECH EM INC.

Figure_6-3_Outdoor_Driving_ABS_022114.mxd - DWH - TTEMI HELENA




Topographic Map Sources: Esri, DeLorme, NAVTEQ, TomTom, Intermap, increment P Corp., GEBCO, USGS, FAO, NPS, NRCAN, GeoBase, IGN, Kadaster NL, Ordnance Survey, Esri Japan, METI, Esri China (Hong Kong), swisstopo, and the GIS User Community

**LEGEND**

- OPERABLE UNIT 7 BOUNDARY
- PARCEL BOUNDARY
- INDOOR RESIDENTIAL ACTIVITY-BASED SAMPLING LOCATION
- STATE HIGHWAY
- US HIGHWAY
- OTHER ROADS
- RAILROAD

N

0 1 Miles

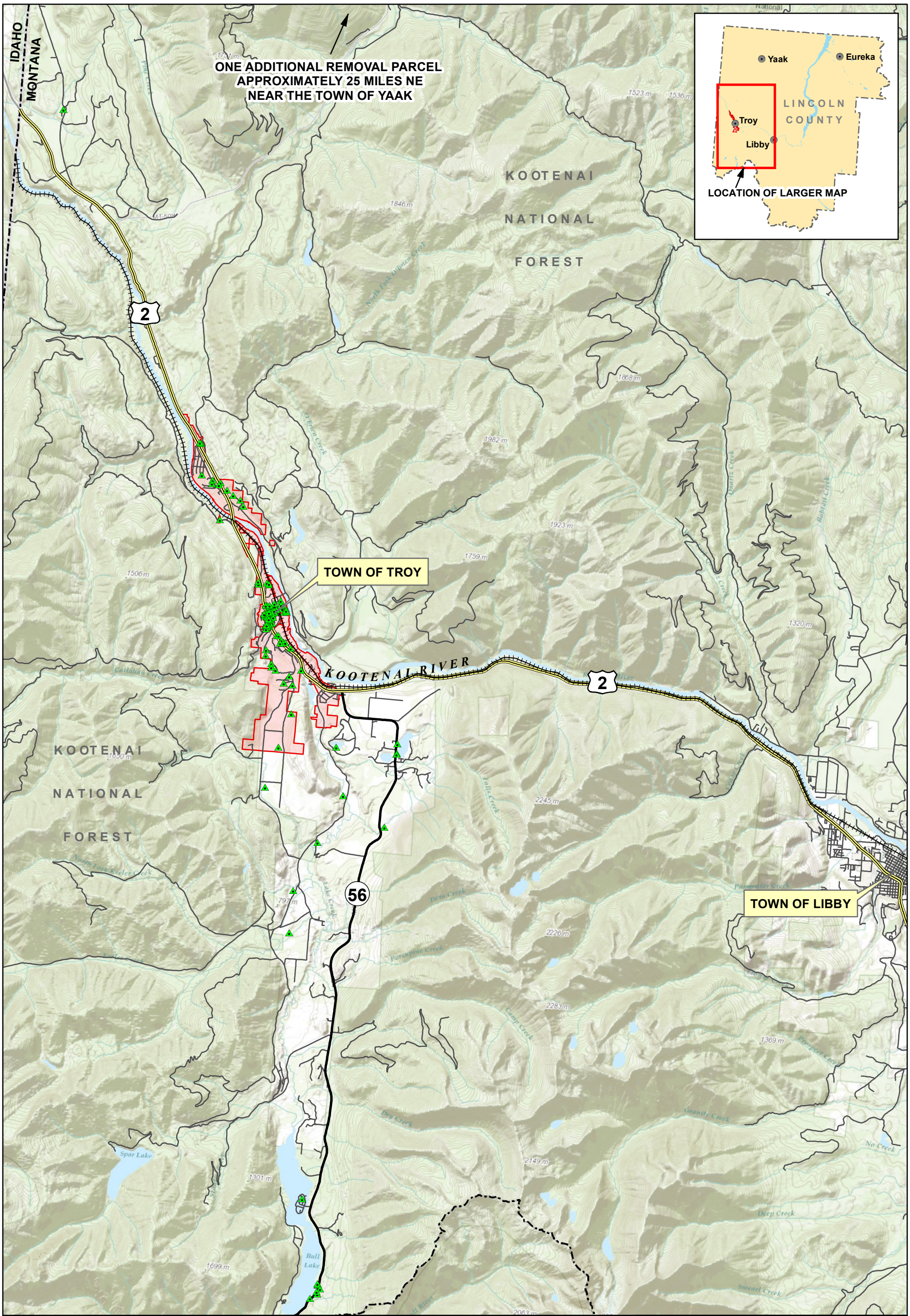
Remedial Investigation Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 7-1**

OPERABLE UNIT 7
INDOOR RESIDENTIAL
ACTIVITY-BASED SAMPLING LOCATIONS

TETRA TECH EM INC.

Figure_7-1_Indoor_Residential_ABS_030514.mxd - DWH - TTEMI HELENA




Topographic Map Sources: Esri, DeLorme, NAVTEQ, TomTom, Intermap, increment P Corp., GEBCO, USGS, FAO, NPS, NRCAN, GeoBase, IGN, Kadaster NL, Ordnance Survey, Esri Japan, METI, Esri China (Hong Kong), swisstopo, and the GIS User Community

**LEGEND**

- OPERABLE UNIT 7
- LINCOLN COUNTY BOUNDARY
- STATE HIGHWAY
- US HIGHWAY
- OTHER ROADS
- RAILROAD
- PARCEL THAT UNDERWENT REMOVAL

N

0 5 Miles

Remedial Investigation Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 10-1**

OPERABLE UNIT 7
PARCELS THAT UNDERWENT REMOVAL

TETRA TECH EM INC.

Figure_10-1_Parcels_That_Underwent_Removal_031114.mxd - DWH - TTEMI HELENA

# Appendix A
# Project-specific Laboratory Modifications and Standard Operating Procedures

# Request for Modification
to
## Laboratory Activities
**LB-000016H**

***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:_____

Requester: Mike Lenkauskas Title: QATS Contractor
Company: Shaw Environmental Date: March 19, 2012

Original Requester [LB-000016A]: Lynn Woodbury, Syracuse Research Corporation Original Request Date: 04/10/08
Original Requester [LB-000016G]: Lynn Woodbury, CDM Original Request Date: 02/06/12

Description of Modification:
Permanent modifications and clarifications to TEM structure recording rules for ISO 10312 and the documentation of previous historical modifications and clarifications. This modification applies to all Libby TEM samples where the ISO 10312 counting rules apply, regardless of sample matrix (air, dust, water, woodchip/duff, tree bark, and tissue samples).

Reason for Modification:
To provide consistency in structure recording procedures for the project across the Libby laboratories and consistency relative to historical recording practices.

Potential Implications of this Modification:
Negative implications – Comparisons of the total number of LA structures between historical results and current results may be biased (high or low) due to differences in recording rules. Positive implications – will improve consistency in structure recording practices.

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

This laboratory modification is (circle one): **NEW** **APPENDS to** _____ **SUPERSEDES LB-000016A and LB-000016G**

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: 12-5-2011*
*Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.*

*Note: ABS air samples collected in 2011 and analyzed prior to this date will be revised to reflect the recording rules specified in this modification.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): See attached for a description of the clarified recording rules.

Data Quality Indicator (circle one) – Please reference definitions on this form 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: Jeanne Spencer Date: 6/6/12
*(Laboratory Manager or designate)*

Project Review and Approval: Dania Zimmer Date: 6/6/12
*(USEPA: Project Manager or designate)*

Approved By: David L Berry Date: 6/6/12
*(USEPA: Technical Assistance Unit Chief or designate)*

MODIFICATIONS AND CLARIFICATIONS

1. Overloading Modification:

   The ISO 10312 method requirement states that if the specimen grid exhibits more than approximately 10% obscuration on the majority of the grid openings, the specimen shall be designated as overloaded. A rejection criteria of >25% obscuration and <50% intact grid openings will be used for this project. The 25% overload criteria resulted from various communications that took place 29 December 1999 between EPA Region 8, their technical contractors, and the laboratories.

2. Indirect Preparation of Air Samples Modification:

   The ISO 10312 method utilizes a direct filter preparation technique. If the sample is visibly overloaded or contains loose debris, it will be prepared indirectly according to procedures provided in SOP EPA-LIBBY-08. Secondary filters will be analyzed according to the ISO 10312 counting rules for this project. Calculations will be adjusted to contain a dilution factor. This indirect preparation procedure will enable the capture of data from samples that otherwise would be rejected. If an indirect preparation is performed, the relevant SAP Analytical Requirements Summary Sheet will specify whether or not the sample should be ashed.

3. Stopping Rule Clarification:

   Stopping rules requirements will be specified in the applicable SAP Analytical Requirements Summary Sheet.

4. Abundant Chrysotile Modification:

   Enumeration of chrysotile structures may be terminated at the end of the grid opening after which the minimum number of chrysotile structures specified in the applicable SAP Analytical Requirements Summary Sheet are counted and recorded. The analyst will continue to count amphibole structures only until the remaining grid openings to be analyzed are completed as required to satisfy the applicable stopping rule.

5. Structure Counting and Recording Modifications and Clarifications:

   a. Non-asbestos material (NAM) structures are not being recorded, unless identified as a “close call” (see the current version of LB-000066 for details). This project-specific modification stems from the need to only quantify levels of contaminants of concern (i.e., asbestos) at a given sample location.

   b. Recording rules will be as described in the ISO 10312 method except that the aspect ratio and minimum length requirement will be specified in the applicable SAP Analytical Requirements Summary Sheet.

   c. Fibers (either individual fibers or fibers within disperse matrices or clusters) shall only be recorded if they meet the dimension criteria (i.e., length, width, aspect ratio) specified in the SAP Analytical Requirements Sheet.

   d. Per the ISO 10312 method, a compact cluster or matrix is defined as an arrangement of particles where structures can be observed but cannot be disaggregated and the length and widths of individual constituent fibers or bundles cannot be unambiguously determined.

   e. The following rule applies only to high magnification (approximately 20,000X) analyses: The minimum aspect ratio criterion does not apply to bundles, compact clusters, compact matrices, or residuals. The overall aspect ratio of a bundle, compact cluster, compact matrix, or residual may have any value. Bundles shall only be recorded if they contain individual constituent fibers that meet the appropriate length, width, and aspect ratio criteria.

   f. Structures that intersect a non-countable grid bar (i.e., top and left grid bars) will be recorded on the count sheet but excluded from the structure loading and concentration calculations. These non-countable structures will be denoted as non-countable in the Total column of the EDD (i.e., with a zero in the Libby-specific TEM spreadsheets or a blank in the Libby Asbestos Data Tool [LADT] database). If a structure intersects more than one grid bar, the structure is not counted only if all grid bar intersections

are non-countable (top and left). If at least one grid bar intersection is countable, then the structure is counted. If a primary structure intersects a non-countable grid bar, then each total structure within the arrangement that does not intersect a non-countable grid bar will be counted and recorded. **Figure 1** presents examples of structures that intersect grid bars.

g. If a structure intersects more than one grid bar (i.e. if both ends of a structure are obscured by grid bars or if a structure originates in one grid opening and extends into an adjacent grid opening) then the observed length of the structure is recorded.

h. If only one end of a structure intersects a grid bar, then the recorded length should be twice the observed length and a comment (for example, XGBLD for Crosses Grid Bar Length Doubled or XNCGBLD for Crosses Non-Countable Grid Bar Length Doubled) should be recorded in the structure comment field indicating that the length was doubled.

i. For disperse clusters and disperse matrices, the primary complex structure type should be recorded as CD or MD, respectively, followed by two integers. The first integer is the total count of the component structures (fibers and bundles) that comprise the primary complex structure. If the total count is higher than 9, record a "+". The second integer is the count of the component structures that are longer than 5 µm. If the count is higher than 9, record a "+". It is not necessary to record the overall dimensions of the disperse cluster or matrix. If there are compact clusters and matrices that are component structures, these should be recorded as residuals (i.e., MR, CR). [Note: It is anticipated that residuals will occur rarely in Libby samples.] **Figure 2** presents an example that illustrates structure recording rules for a disperse matrix with more than 5 component structures.

6. Recording rules for partially obscured structures (modification to ISO 10312, Section C.4.8)

a. For partially obscured structures, the proportion of the structure that is obscured by opaque particulates shall be used as the basis for determining the appropriate recording methodology.

b. If the obscured length could not possibly be more than one-third of the total length, the structure should be recorded in accordance with the historical recording procedures for disperse clusters and matrices utilized at the Libby Site. That is, the primary complex structure (e.g., MD, CD) would be recorded with the component structure(s) (e.g., MF, MB, CF, CB) recorded separately. The recorded length for the partially obscured component structure shall be equal to the visible length of the fiber or bundle.

c. If the obscured length could be more than one-third of the total length, the structure should be recorded using the same procedure as described above, except that the structure type of the component structure(s) should be recorded with an "O" suffix (i.e., MFO, MBO).

d. The rule for recording fiber length is that the best estimate of <u>visible length</u> should always be recorded. If part of the fiber is obscured by opaque debris, record fiber length as only the visible length, not what the length could be if part of the fiber was not covered. If part of the fiber is obscured by debris that is not completely opaque, record fiber length as the best estimate of the visible length, including the part of the fiber that is embedded in electron-transparent to translucent debris.

**Example 3 in Figure 1 and all examples in Figure 3** illustrate structure recording rules for fibers that have obscuring particulate debris.

7. The definition of a PCM equivalent (PCME) structure is as follows: Any fiber, bundle, matrix, or cluster with an aspect ratio of 3:1 or greater, length longer than 5 µm, and width greater than or equal to 0.25 µm. In the event that the analytical requirements specify the use of "low magnification, PCME only" recording rules (as described in Annex E of the ISO method), then:

a. For disperse matrices and disperse clusters, only those component structures that meet PCME counting rules (i.e. length > 5 µm, width $\geq$ 0.25 µm, aspect ratio $\geq$ 3:1) will be recorded as countable structures.

b. For disperse clusters and disperse matrices, the two integers recorded (e.g. MD55) for the primary complex structure type should only include those component structures that meet PCME counting rules.

c. If the protruding fiber or bundle of a disperse matrix does not meet PCME counting rules (i.e., length > 5 μm, width ≥ 0.25 μm, aspect ratio ≥ 3:1), then it should not be recorded as a countable structure.

d. Bundles, compact clusters, or compact matrices that meet PCME counting rules (i.e., length > 5 μm, width ≥ 0.25 μm, aspect ratio ≥ 3:1) will be recorded as countable structures.

e. Bundles, compact clusters, or compact matrices that do not themselves meet PCME counting rules (i.e., length > 5 μm, width ≥ 0.25 μm, aspect ratio ≥ 3:1), but for which component PCME fibers can be visually observed but not disaggregated, should be recorded on the bench sheet and identified as non-countable (as indicated by marking the total column for the structure with a zero in the EDD). These structures will not be included in calculated PCME structure counts and concentrations, but are provided for documentation purposes only. Bundles, compact clusters, or compact matrices that do not themselves meet PCME counting rules and for which component PCME fibers cannot be visually observed, should be not be recorded on the bench sheet.

8. For analyses with < 50 grid openings, selection of grid openings should be random and adjacent grid openings should be avoided when possible. For analyses with ≥ 50 grid openings, preferential selection of adjacent grid openings is permissible.

OVERVIEW OF HISTORICAL RECORDING CRITERIA

At the beginning of the Libby project, analytical laboratories (primarily EMSL and RESI) were following the ISO method with regard to structure recording (i.e., recording only those structures meeting an aspect ratio of greater than or equal to 5:1).

Approximately the time of the Phase 2 Investigation (late Spring 2001), project laboratories were instructed by Chris Weis (EPA, Region 8) to record all structures regardless of minimum length or aspect ratio. This recording rule change enabled data users to gain a better understanding of the dimension attributes for structures at the Libby site and allowed for the calculation of PCM equivalent (PCME) structures. In the ISO report generated by the TEM EDD spreadsheet, structures with an aspect ratio less than 5:1 were counted in Bin A and structures with a length less than 0.5 um were counted in Bin B. Also at this time, the TEM EDD spreadsheet was modified to allow for the capture of the raw structure data, as entered from the laboratory bench sheet, into the Libby site database.

Although it is uncertain exactly when the recording rules changed after the Phase 2 Investigation, based on analyst interviews, project laboratories reverted back to following the ISO method (i.e., recording only those structures meeting an aspect ratio of greater than or equal to 5:1) beginning approximately December 2001, unless specifically requested otherwise in project-specific SAPs and/or QAPPs (e.g., the Supplemental Remedial Investigation samples collected under the SQAPP specified an aspect ratio criterion of greater than or equal to 3:1).

Laboratory modifications LB-000016B through LB-000016F (available in the archive directory) document the historical laboratory and analyst-specific deviations in recording/counting rules for ISO based on analyst interviews conducted in August and September 2006.

The following is a time-line of other modifications to the preparation, analysis, and recording of results for samples prepared and analyzed by ISO Method 10312:

- Effective December 21, 2006, preparation techniques and recording rules were further refined as part of LB-000053, whereby all Libby samples were classified as "investigative" or "non-investigative". Samples classified as "investigative" were to utilize an aspect ratio criterion of 3:1, and samples classified as non-investigative were to utilize an aspect ratio criterion of 5:1, unless program-specific sampling and analysis plans (SAPs) specify otherwise or specifically requested otherwise.

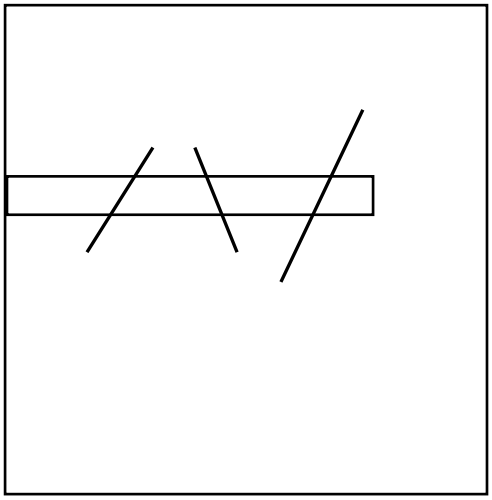
  Note that LB-000053 was retired in February 2012, and aspect ratio requirements are currently specified in SAP Analytical Requirements Summary Sheets, which are distributed to the participating laboratories.

- Effective August 29, 2006, all project laboratories began utilizing an aspect ratio criterion of 3:1, unless specifically requested otherwise.

- From October 2011 to May 2012, various discussions among all members of the Libby Lab Team were held to discuss problems in counting and recording partially obscured, compact cluster, and compact matrix structures. LB-000016 was updated to version G in December 2011 and then to H to document the outcome of these discussions and represents what the Libby Lab Team considers to be the best consensus approach for staying consistent with how structures were counted historically, the intent of the ISO method, and the needs of data users.

**FIGURE 1**
**ILLUSTRATIONS OF STRUCTURES THAT INTERSECT GRID BARS**

**Example 1**

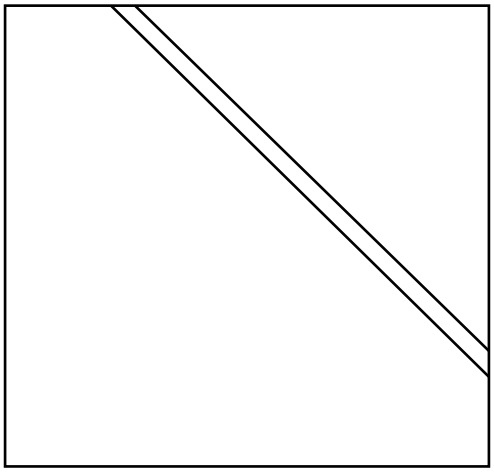


Record as a disperse cluster (CD) with 3 countable fibers (F).

CD44
CF 0
CF 1
CF 2
CF 3

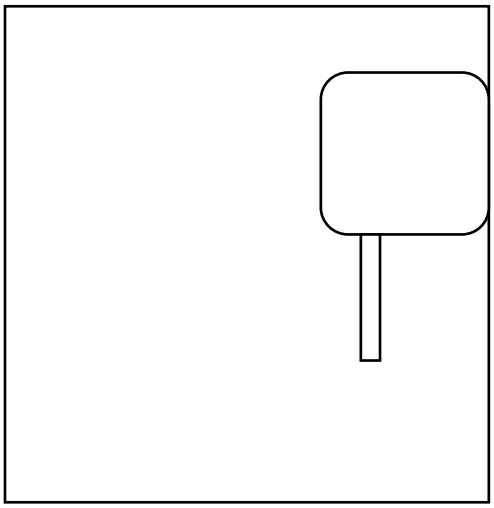
The large structure is excluded because it crosses a non-countable (left) grid bar.

**Example 2**



Count as one fiber (F). The structure is counted because it intersects a countable (right) grid bar. Record the length as that observed without doubling.

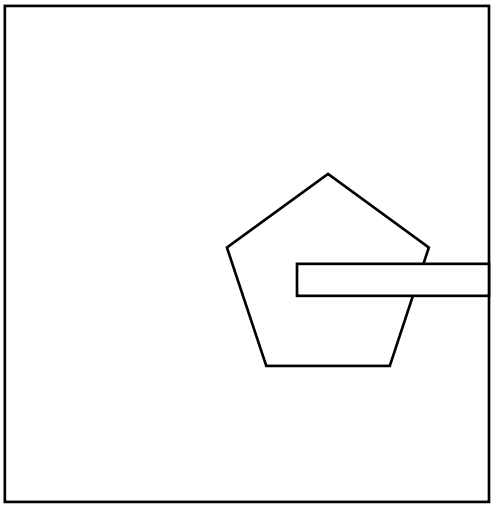
**Example 3**



Count as 1 disperse matrix (MD) consisting of 1 fiber longer than 5 µm.

Record the primary structure as MD11, followed by 1 total structure recorded as MFO. When recording the MFO, do not double the length.

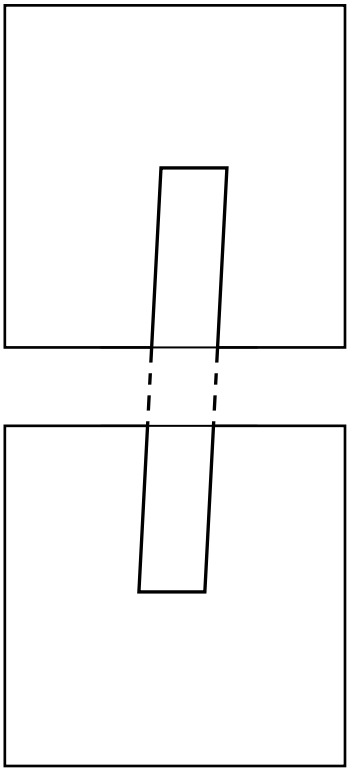
**Example 4**



Count as 1 disperse matrix consisting of 1 fiber longer than 5 µm.

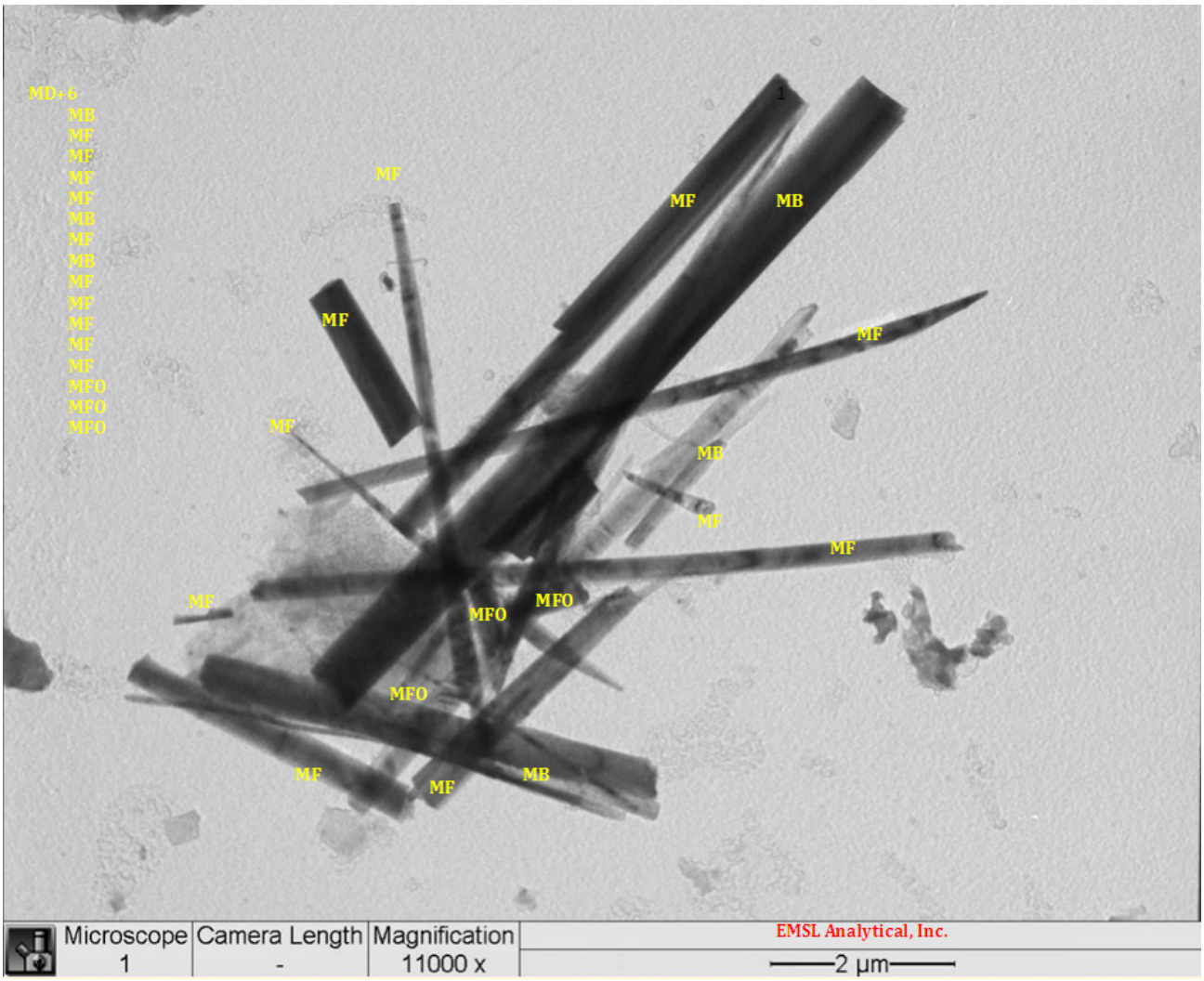
Record the primary structure as MD11, followed by 1 total structure recorded as MF. When recording the MF, double the length of the observed fiber.

**Example 5**



Count as 1 fiber (F). Record the actual length, including the protrusion into adjacent grid opening.

**FIGURE 2**
**ILLUSTRATION OF STRUCTURE RECORDING RULES**
**FOR A DISPERSE MATRIX WITH MORE THAN 5 COMPONENT STRUCTURES**



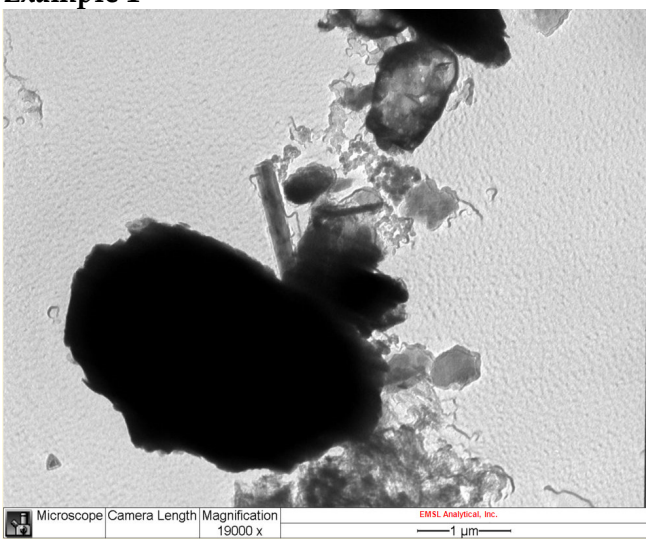

Count as 1 disperse matrix consisting of more than 10 component fibers/bundles, of which 6 have a length that is greater than 5 µm in length.

Record the primary structure as MD+6, followed by 16 total structures recorded as MF, MB, and MFO, as appropriate. Recorded length of each component structure should be equal to the visible length.

Photo courtesy of EMSL Analytical, Inc.

**FIGURE 3**
**ILLUSTRATION OF STRUCTURE RECORDING RULES**
**FOR PARTIALLY OBSCURED STRUCTURES**

**Example 1**

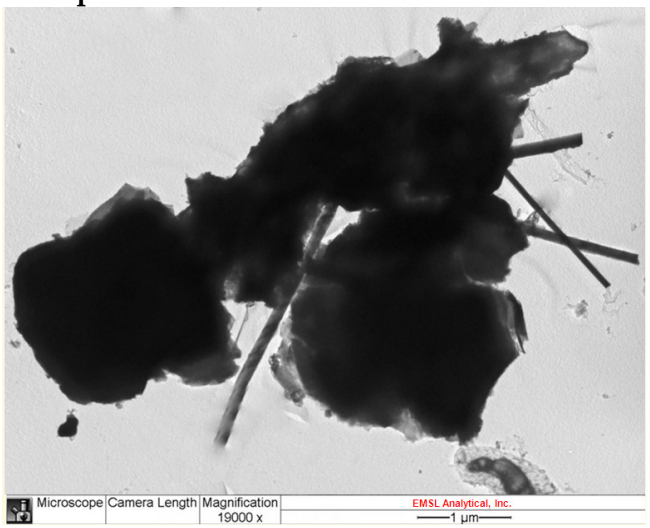



Count as 1 disperse matrix consisting of 1 component fiber that is less than 5 µm in length.

Record the primary structure as MD10, followed by 1 total structure recorded as MFO. Recorded length for the MFO should be equal to the visible length, which in this case is the same as the protruding length.

Photo courtesy of EMSL Analytical, Inc.

**Example 2**

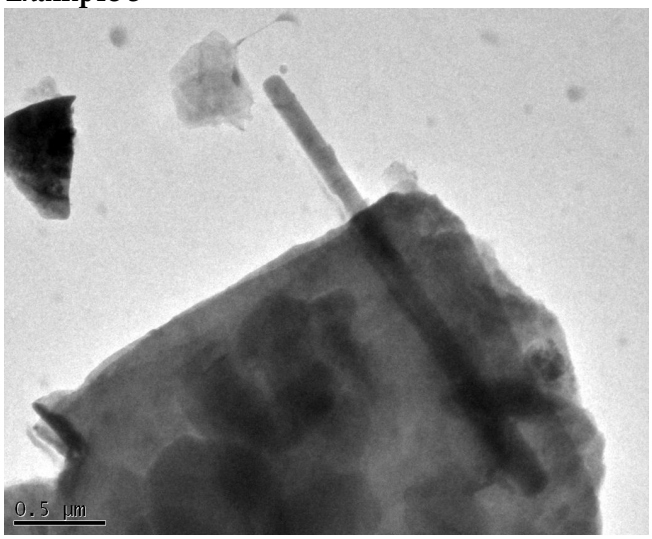



Count as 1 disperse matrix consisting of 4 component fibers that are all less than 5 µm in length.

Record the primary structure as MD40, followed by 4 total structures each recorded as MFO. Recorded length for the 3 fibers at upper right should be equal to the visible length, which in this case is the same as the protruding length. The recorded length of the fiber at left should be the visible length (about 2.5 µm, from the free end to the top of the triangle of light at the center of the arrangement).

Photo courtesy of EMSL Analytical, Inc.

**Example 3**

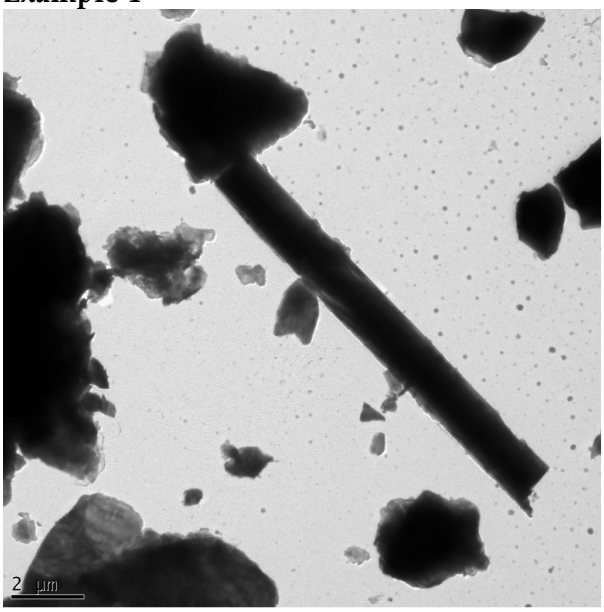



Here is an LA fiber on an electron-translucent mica flake. Count as 1 disperse matrix consisting of 1 component fiber that is less than 5 µm in length.

Record the primary structure as MD10, followed by 1 total structure recorded as MF. Recorded length for the MF should be equal to the visible length (protruding length plus the part of the fiber embedded in the electron-translucent mica flake).

Photo courtesy of ESAT Region 8

**Example 4**

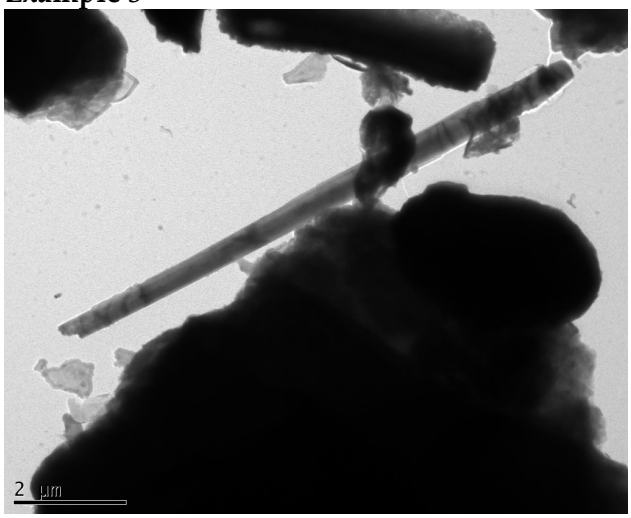



Count as 1 disperse matrix consisting of 1 component fiber that is greater than 5 µm in length.

Record the primary structure as MD11, followed by 1 total structure recorded as MF. Since the particle at the top left end of this LA fiber is opaque, recorded length for the MF should be equal to the protruding length (ignoring the small pieces of dust stuck to the sides of the fiber).

Photo courtesy of ESAT Region 8

**Example 5**

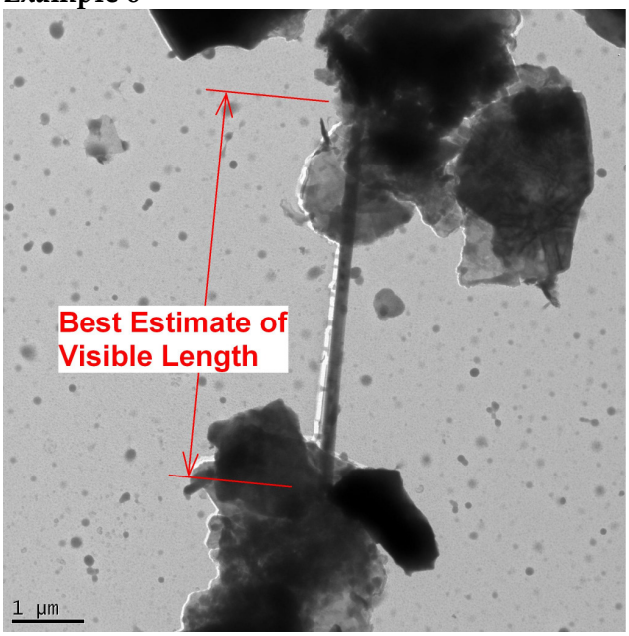



Here is an LA fiber with a piece of dirt sticking to it. It is not a matrix since both ends are free. Count as 1 fiber (primary and total structure).

Record as F. Recorded length should be equal to the visible length of the fiber.

Photo courtesy of ESAT Region 8

**Example 6**




Here is a "bridge" LA fiber. Count as 1 disperse matrix consisting of 1 component fiber that is greater than 5 µm in length.

Record the primary structure as MD11, followed by 1 total structure recorded as MF. Since both fiber ends are semi-visible, ascertain where the fiber ends are as accurately as possible and record fiber length as the best estimate of visible length (about 5.3 µm).

Photo courtesy of ESAT Region 8

# Request for Modification
To
## Laboratory Activities
**LB-000019**

***Instructions to Requester: E-mail form to contacts at bottom of form for review and approval. File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:***

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs

Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable):TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies

Requester: R. K. Mahoney Title: Senior Analyst/Special Projects Coordinator

Company: EMSL Analytical, Inc. Date: 21 January 2003

Description of Modification:
Clarification of bench sheet recording format for grid openings in which no countable structures are recorded.

Reason for Modification:
The electronically deliverable spread sheet for TEM analysis developed for the Libby project requires "ND" (None Detected) to be entered for grid openings in which no countable structures are recorded. The ND code has been used on all electronic deliverables for the Libby project. The code "NSD" (No Structure Detected) has been used on hand written bench sheets up until this date. As of 21 January 2003, "ND" will be used on the bench sheets as well as the electronically deliverables.

Potential Implications of this Modification:
There are no potential negative implications resulting from this clarification of terms.

Laboratory Applicability (circle one): All Individual(s) EMSL Analytical, Inc.

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: 21 January 2003
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):

Technical Review: R. K. Mahoney Date: 27 March 2003
(Laboratory Manager or designate)

Project Review and Approval: Mark Raney Date: 7 March 2003
(Volpe: Mark Raney)

Approved By: Mary Goldade Date: 7 March 2003

Title: EPA Regional Chemist
(USEPS: Mary Goldade)

# Request for Modification
To
## Laboratory Activities
**LB-000028**

***Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.***
***File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:***

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable):TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies

Requester: R. K. Mahoney Title: Senior Analyst / Special Projects Coordinator
Company: EMSL Analytical, Inc. Date: 17 June 2003

Description of Modification:
This is a clarification pertaining to the re-analysis of TEM samples when some of the originally read grid openings in a sample selected for re-analysis have become unreadable. In the event that more than half of the originally read grid openings have become unreadable, select the closest adjacent sample from the same sample delivery group with adequate intact grid openings for re-analysis. If half or less of the original openings on the sample selected are unreadable, make note in the Comments box in Data Entry 1 of the TEM EDD as to which grid openings are unreadable, and proceed with analysis of the original sample.

Reason for Modification:
This clarification is intended to provide more complete TEM re-analysis data.

Potential Implications of this Modification:
There are no negative implications to this clarification.

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

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: 17June 2003

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):

Technical Review: R. K. Mahoney EMSL Date: 18 July 2003
*(Laboratory Manager or designate)*

Project Review and Approval: Mark Rance Date: 7/18/03
*(Volpe: Project Technical Lead or designate)*

Approved By: Mary Goldade Date: 6/24/03

Title: Project Chemist
*(USEPA: Project Chemist or designate)*

## Statistical Comparison of Two Poisson Rates

Selected Confidence Interval: 0.95

| First Evaluation (Rate 1) | | Second Evaluation (Rate 2) | | |
|---|---|---|---|---|
| Structure Count | Sensitivity [a] | Structure Count | Sensitivity [a] | Poisson Ratio Rate Comparison (CI=95%) |
| 10 | 0.001 | 1 | 0.1 | [0.01-4.34] The rates are not different |

[a] air sensitivity units: $(cc)^{-1}$
dust, tree bark sensitivity units: $(cm)^{-2}$
water sensitivity units: $(L)^{-1}$
soil, duff, wood waste, tissue sensitivity units: $(g)^{-1}$

# Request for Modification
to
## Laboratory Activities
**LB-000029E**

***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 Date: January 16, 2014

Original Requester [LB-000029]: William Brattin, Syracuse Research Corporation — Original Request Date: August 23, 2003

Original Requester [LB-000029A]: William Brattin, Syracuse Research Corporation — Original Request Date: November 18, 2003

Original Requester [LB-000029B]: Lynn Woodbury, Syracuse Research Corporation — Original Request Date: December 7, 2006

Original Requester [LB-000029C]: Lynn Woodbury, CDM Smith — Original Request Date: May 4, 2012

Original Requester [LB-000029D]: Lynn Woodbury, CDM Smith — Original Request Date: March 12, 2013

Description of Modification:
Permanent clarifications to laboratory-based quality control (QC) analysis requirements for TEM. The purpose of the attached is to standardize the frequency of analysis and procedures for the selection and interpretation of the results for laboratory-based TEM QC analyses (regardless of sample medium).

Reason for Modification:
This modification is needed to standardize the frequency with which different types of TEM QC analysis are performed by TEM laboratories in the Libby laboratory program, and to ensure that all results are evaluated in accord 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-000029, LB-000029A, LB-000029B, LB-000029C, LB-000029D

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: February 11, 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 attached pages)

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: 2/6/14
*(Laboratory Manager or designate)*

Project Review and Approval: _____ Date: 2/11/14
*(USEPA: Project Manager or designate)*

Approved By: David L. Berry Date: 2/11/14
*(USEPA: Technical Assistance Unit Chief or designate)*

## *TEM QC Sample Type Definitions*

There are three types of TEM laboratory QC analyses: Blanks, Recounts, and Repreparations. Each type of QC analysis is described below:

Blanks

Laboratory Blank (LB) –TEM grids are prepared from a new, unused filter at the laboratory and analyzed using the same preparation procedure as used for field samples. The purpose of an LB is to provide information on whether asbestos contamination is introduced during sample preparation or analysis at the analytical laboratory. The laboratory must prepare one LB for each batch of samples prepared together. The LB accompanies the field samples through the entire preparation process (including the indirect preparation process, if applicable).

Recounts

Recount analyses include recount same, recount different, verified analyses, and inter-laboratory analyses. In all cases, the analysis is a re-examination (or recount) of TEM grid openings that were evaluated during the initial analysis. The purpose of recount analyses is to provide information on the reproducibility of analytical results, both within and between analysts of the same laboratory, and between analysts from different laboratories. Each of these recount analyses are described in more detail below:

- Recount Same (RS) – Select original TEM grid openings are re-examined by the same microscopist who performed the initial examination.
- Recount Different (RD) – Select original TEM grid openings are re-examined by a different microscopist in the same laboratory than who performed the initial examination.
- Inter-laboratory (IL) – Select original TEM grid openings are re-examined by a microscopist in a different laboratory than who performed the initial examination.
- Verified Analysis (VA) – This analysis is similar to a RD but has different documentation requirements. A VA must be recorded in accordance with the protocol provided in NIST (1994).

Repreparations

Repreparation (RP) –TEM grids are prepared from a new aliquot of the same filter used to prepare the original grids, which will be either the original filter (for a direct preparation) or the secondary filter (for an indirect preparation). Typically, this is done by the same laboratory that performed the original analysis; however, a different laboratory may also prepare grids from a new piece of the filter. If the RP is done by the same laboratory, the RP and re-analysis must be done by a different analyst than who performed the original analysis. The purpose of RP analyses is to provide information on the degree of inherent variability in preparation and analysis methods.

## *Frequency*

The minimum frequency for TEM QC analyses within each laboratory (across all media) shall be as follows:

| **QC Sample Type** | **Minimum Analysis Frequency** |
|---|---|
| Laboratory blank | 4% |
| Recount same | 1% |
| Recount different | 2.5% |
| Inter-laboratory | 1% |
| Verified analysis | 1% |
| Repreparation | 1% |
| **Total** | **10.5%** |

Each laboratory must analyze LBs, recounts (RS, RD, and VA), and RPs at the minimum frequencies specified in the table above. An investigation-specific Sampling and Analysis Plan (SAP) and its associated Analytical Requirements Summary Sheet can supersede the percentages in the above table for a specific method and/or medium. The procedures for the selection of samples for the purpose of performing recounts (RS, RD, and VA) and RPs are provided in Attachment 1. Samples for IL comparisons will be selected *post hoc* by the EPA Quality Assurance Technical Support (QATS) contractor (or their designee) in accordance with the procedure provided in Attachment 2.

### *Procedure for Evaluating QC Samples and Responses to Exceptions*

The procedure for evaluating QC sample results varies depending on sample type. These procedures are presented below.

*Note: The procedures for evaluating QC samples presented below are based in part on professional judgement and experience at the Libby Asbestos Superfund Site (Site) to date. These procedures and rules for interpretation may be reviewed and revised as more data are collected.*

Laboratory Blanks

The area of filter to analyze for the LB will depend upon the specified analytical requirements for the associated laboratory job, which will be specified in the investigation-specific SAP. At a minimum, a filter area of at least 0.10 $mm^2$ must be analyzed for each LB. If one or more asbestos structures are detected, the laboratory shall immediately investigate the source of the contamination and take immediate steps to eliminate the source of contamination before analysis of any investigative samples are resumed. Detection of any asbestos on LBs shall be communicated to the laboratory coordinator (LC) immediately.

Recounts

Recount analyses are evaluated on a grid opening (GO) and structure-specific basis by comparing the raw data benchsheets prepared by each analyst. The following criteria will be used to identify cases where results are concordant (in agreement) or discordant (not in agreement). The Libby Amphibole (LA) criteria were established by TEM analysts experienced in the analysis of LA, and serve as review criteria developed using their professional experience. As the database continues to grow and we learn more, these criteria may be revisited and revised. Changes to the criteria for LA structures will be accompanied by scientific justification to support the change. Criteria for concordance on non-LA fibers (Other Amphibole [OA] and Chrysotile [CH]) are the same as described in NIST (1994).

| **Measurement Parameter** | **Concordance Rule** |
|---|---|
| Number[1] of LA structures within each grid opening | For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10 percent (%) as calculated as RPD (((maximum count – minimum count)/average count)*100%). |
| Asbestos class of structure (LA, OA, CH) | Must agree 100% on CH vs. amphibole. For assignment of amphiboles to LA or OA bins, must agree on at least 90% of all amphibole structures. |
| LA Structure length | For fibers and bundles (all methods) and CF, CB, MF, and MB structures (ISO), must agree within 1 micron (μm) or 10% (whichever is less stringent).<br>For clusters and matrices (AHERA and ASTM) and CFO, CBO, CC, MFO, MBO, and MC structures (ISO), must agree within 2 μm or 20% (whichever is less stringent). |

[1] For air samples, concordance will be evaluated based on total and PCME structure counts.

| Measurement Parameter | Concordance Rule |
|---|---|
| LA Structure width | For fibers and bundles (all methods) and CF, CB, CFO, CBO, MF, MB, MFO, and MBO structures (ISO), must agree within 0.5 μm or 20% (whichever is less stringent).<br>For clusters and matrices (AHERA and ASTM) and CC and MC structures (ISO), there is no quantitative rule for concordance. |
| Presence of Sodium (Na) and Potassium (K) | There is no rule for concordance, but must be tabulated to identify potential trends that may indicate inconsistencies in recording practices or interpretation of spectra. |

Whenever a RS or RD occurs with one or more discordance, the sample will undergo a VA as described by NIST (1994). The VA process described in NIST (1994) consists of assignment of TPs (true positives), FPs (false positives), and FNs (false negatives) to individual structures for each analyst. The NIST (1994) VA process applies to all VA and IL analyses as well. Re-examination of grids is not needed if TPs, FPs, and FNs can be determined by examination of the benchsheets and spectra by the senior laboratory analyst and discussion among all analysts involved. Otherwise, re-examine the grids in the TEM to resolve discrepancies for structures in dispute. In the rare event that a structure cannot be confirmed during the VA process, then it should be identified as ambiguous or not located as described in NIST (1994). Ambiguous structures can occur because of excessive beam damage to a structure or obscuring particulate debris that interferes with EDS and/or diffraction. Not located structures can occur because of small amounts of grid damage (e.g. tears or roll-ups in the carbon) in a GO that is otherwise intact. As described in NIST (1994), ambiguous and not located structures do not count as FPs or FNs and are not included in the TNS (Total Number of Structures) verified count.

The laboratory will track rates of true positives, false positives, and false negatives temporally for each analyst as VAs are performed. Section 3.2 of NIST (1994) states that, "Experienced analysts should attain TP/TNS values ≥ 0.85 and FP/TNS ≤ 0.05." These target rates of true positives and false positives will be applied to the Libby Project. Whenever one or more discordances occur in a recount, the senior laboratory analyst will use the VA process to determine the basis of the discordance and decide if the discordance is severe enough to warrant corrective actions (e.g. retraining in counting rules, quantification of size, identification of asbestos types, etc.) or not on a case by case basis. Any corrective actions taken in response to discordance will be described in the case narrative that is included in the data package. Because some rate of discordance is expected over the course of routine analysis, not every instance of discordance will result in corrective actions. If the senior laboratory analyst determines the discordance is not severe enough to warrant corrective actions, indicate a brief justification of why corrective actions were not taken in the case narrative.

The following paragraph refers only to RSs, RDs, and VAs (concordance for ILs is a separate matter and is described in Attachment 2). If the original and recount results are concordant, report them as is using standard deliverable procedures and no further action is required. In the event that one result (original or recount) is determined to be correct whereas the other is not, identify whichever result is correct by entering the word "Confirmed" in the sample comment field of the electronic data deliverable (EDD). If the original and/or recount results were found to be incorrect, report them as is and do not correct the results in the EDD. In the special case where the original and the reanalysis are both determined to have one or more areas of discordance, a third EDD will be prepared that contains the correct results. This will be identified as QC Type = "Reconciliation." In accordance with the usual procedures and requirements of the National Voluntary Laboratory Accreditation Program (NVLAP), the laboratory must maintain records of all cases of discordant results and of actions taken to address problems. In addition, each laboratory must notify the LC and the EPA QATS contractor of any significant exceptions and corrective actions through a job-specific (temporary) laboratory record of modification (ROM) form.

<u>Repreparations</u>
RP samples will be evaluated by the analytical laboratory by comparing the results for the original and the RP analyses. In order to be ranked as concordant the results must not be statistically different from each other at

the 90% confidence interval, as evaluated using the Poisson ratio statistical procedure documented in Attachment 3. If the RP results are found to be statistically different from the original analysis results, a senior analyst will investigate to see if this discordance may be related to laboratory procedures, and will take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, quantification of size, identification of asbestos types, etc.) if necessary.

### *Program-Wide Goals*

While each laboratory monitors the results of the QC samples analyzed within their laboratory and takes actions as described above, the overall performance of the program shall be monitored by assembling summary statistics on QC samples, combining data within and across laboratories. Overall performance will be assessed by QATS on an annual basis with the results provided in the Annual QA/QC Summary report. The program-wide goals shall be interpreted as follows:

| QC Sample Type | Metric | Program-Wide Criteria | | |
|---|---|---|---|---|
| | | Good | Acceptable | Poor |
| Lab Blanks | % with ≥1 asbestos structures | 0% - 0.1% | 0.2% - 0.5% | >0.5% |
| Recounts | Concordance on LA count | >95% | 85-95% | <85% |
| | Concordance on type (chrysotile vs. amphibole) | >99% | 95%-99% | <95% |
| | Concordance on LA length | >90% | 80%-90% | <80% |
| | Concordance on LA width | >90% | 80%-90% | <80% |
| Repreparations | Concordance on LA concentration/loading | >95% | 90-95% | <90% |

If TEM laboratory QC results are ranked as good, no action is necessary. If results are ranked as acceptable, no action is necessary, unless temporal trends indicate an issue within a single laboratory. In this case, the LC will coordinate with the laboratory managers to investigate potential reasons for concordance issues. If TEM laboratory QC results are ranked as poor, the LC shall investigate and may request corrective action, as necessary.

As the database continues to grow and we learn more, these project-wide goals may be revisited and revised. Changes to the project-wide goals will be accompanied by appropriate justification to support the change.

## REFERENCES

NIST. 1994. Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0. National Institute of Standards and Technology, Washington DC. NISTIR 5351. March 1994.

## ATTACHMENT 1

### Selection Procedure and Laboratory SOP for Recounts (RS, RD, VA) and Repreparations (RP)

Selection Procedure

As specified in the Frequency section above, the frequency of Recount Same (RS) shall be no less than 1%, the frequency of Recount Different (RD) shall be no less than 2.5%, the frequency of Verified Analyses (VA) shall be no less than 1%, and the frequency of Repreparations (RP) shall be no less than 1%, corresponding to a total within-laboratory quality control (QC) frequency of 5.5% for these analysis types. This is approximately one (1) QC sample per 20 field samples. QC frequency is calculated as the number of QC's performed in a given time period divided by the number of requested analyses performed (generally field samples, or any other submitted samples such as field or lot blanks) for the same time period. If multiple filter analyses are requested on the same sample (e.g. tree bark or duff samples analyzed by the laboratory in triplicate) each filter replicate counts toward the number of requested analyses. If a laboratory elects to perform QC analyses on samples collected internally by the laboratory (e.g. health and safety samples from the laboratory areas), those QC analyses do not count towards the 5.5% QC frequency and should not be reported to the LC and/or data users. Based on this 5.5% frequency, it is possible to determine which laboratory job(s) will have one or more samples selected for recount or RP analysis.[2]

*Standard Selection Procedure* – For laboratory jobs where a recount or RP sample is to be selected, the analyst will record the total number of LA structures observed in each sample. The sample(s) selected for recount or RP analyses must be those within the laboratory job with the highest number of countable LA structures per grid opening (GO) area examined (calculated as the number of GOs evaluated * the GO area). If all samples within the laboratory job are non-detect, a non-detect sample may be selected; however every effort must be made to select laboratory jobs containing sample analyses that yielded countable LA structures. This selection procedure will ensure that the recount and RP analyses yield a dataset best suited to assess concordance[3].

*Alternate Selection Procedure* – In some cases (e.g., when extended GO counts are necessary) it is not feasible to wait until the analysis of an entire laboratory job is complete to select samples for recount or RP analysis. In this instance, for laboratory jobs where a recount or RP sample is to be selected, any sample with at least 3 countable LA structures recorded can be considered a suitable candidate for recount or RP selection. If all samples within the laboratory job are non-detect, a non-detect sample may be selected. The Analytical Requirement Summary Sheet for each investigation will specify when the alternate selection procedure is appropriate for use. *Unless specified otherwise, the standard selection procedure should be used.*

When selecting samples for RP, if possible, preferentially select samples in which the total number of GOs is 40 or less. Because RP concordance is evaluated based on concentration, in order to achieve adequate statistical power, RPs must prepare and evaluate the GOs using the same analytical requirements as the original analysis to ensure results comparability. Hence, the selection of samples with 40 GOs or less will reduce analytical costs associated with RPs.

When selecting samples for recount, it is not necessary to impose a minimum or maximum number of GOs because concordance is evaluated on a GO and structure basis for a subset of 10 GOs rather than a concentration basis.

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[2] In addition to the samples selected by the laboratories for RS, RD, VA and RP analyses, data users reserve the right to select QC samples *post hoc*. One example of when this might occur would be the observation that too many non-detect samples are being selected for QC analyses.

[3] It should be noted that this selection procedure will tend to result in the preferential selection of samples with the highest values. Thus, summary statistics should not include laboratory QC samples, which will tend to be biased high.

Laboratory SOP for Recount Analyses

1. For recount samples, re-analyze the selected sample in accordance with the appropriate procedures for each type of recount (RS, RD, or VA). If more than 10 GOs were evaluated in the original analysis, the original analyst or laboratory director will select the 10 GOs with the highest number of LA structures to re-analyze in the recount analysis. The original analyst or laboratory director must also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of countable LA structures per GO area examined, which may be analyzed in the event that a selected GO is damaged and cannot be re-evaluated.

2. Record the results using the most recent version of the Libby-specific TEM electronic data deliverable (EDD) spreadsheet. Identify the Laboratory QC Type as "Recount Same", "Recount Different", or "Verified Analysis", as appropriate, in the TEM EDD. The tag of a RS, RD, or VA analysis must match the tag of the original (Not QC) analysis. Be sure that the grid and GO names match exactly with the names evaluated in the original analysis (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), do not arbitrarily select a different GO for evaluation, but utilize the list of 5 alternative GOs provided by the original analyst or laboratory director to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).

3. If any results between the original analysis and the recount analysis are discordant according to the table on pages 4 and 5, the sample will undergo VA as described by NIST (1994), and the senior laboratory analyst will determine the basis of the discordance and take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of asbestos types, etc.) as needed.

4. Submit the recount TEM EDD to the project data manager using standard deliverable procedures.

Laboratory SOP for Repreparations

1. Prepare new TEM grids using the standard preparation methods at the Libby Site. If the sample was prepared directly, prepare new grids from the original filter. If the sample was prepared indirectly, prepare new grids from the secondary filter used for the original analysis.

2. Select the appropriate number of grids and examine the GOs using the same TEM counting and stopping rules utilized in the original analysis, splitting the number of GOs examined evenly across the prepared grids. Place any remaining grids in storage.

3. Record the results using the most recent version of the TEM EDD. Identify the QC Type as "Repreparation." For RP analyses performed by the same lab that performed the original analysis of the sample, the tag of the QC analysis must match the tag of the original (Not QC) analysis.

5. Submit the RP TEM EDD to the project data manager using standard deliverable procedures.

## ATTACHMENT 2

### Selection Procedure and Laboratory SOP for Inter-laboratory Analyses (IL)

Selection Procedure

1. Inter-laboratory (IL) samples will be selected on an annual basis, with samples selected for distribution at the beginning of each year. Sample selection will be performed by the QATS contractor (or their designee), who will compile a list of all samples for which TEM analysis (across methods and media) was completed in the previous year.

2. Identify the target number of inter-laboratory analyses needed to meet the quality control (QC) requirements for IL analyses specified in the Frequency section on Page 3 (1.0%). This is accomplished by multiplying the desired IL frequency by the total number of TEM analyses performed by each laboratory in the preceding year. For example, 178 TEM air analyses * 1% = 1.78 (which is rounded up to 2). At a minimum, at least one sample for each media (i.e. air, duff and water) will be selected for each analytical laboratory.

3. For each media, rank the order of the TEM analyses from the preceding year by the total number of countable Libby amphibole (LA) structures per grid opening (GO) area examined (calculated as the number of GOs evaluated * the GO area). Selecting from analyses with a high number of LA structures per GO area examined increases the likelihood that the GOs evaluated as part of the inter-laboratory analysis will have one or more LA structures.

4. Exclude any samples previously selected for IL evaluation.

5. For each laboratory select the appropriate number of IL analyses from the available samples for which the total number of countable LA structures per GO area examined is higher than 0 (i.e., LA detects). If the total number of samples with LA detects is equal to the desired number of IL analyses, select all detected samples for IL analysis. If the total number of samples with LA detects is less than the desired number of IL analyses, select non-detect samples for IL analysis.

6. Submit the list of selected IL analysis to the laboratory coordinator (LC). This list should also identify which laboratory will perform the IL analysis in accordance with the following table:

| 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 | |

7. The LC will provide each originating laboratory with the list of samples selected for IL analysis, which will be prepared and provided to the appropriate IL laboratory for analysis.

8. In addition to the sample list, the LC will also provide each of the originating laboratories with a chain-of- custody (COC), which will be used to transfer the grid preparations to the IL Laboratory. The COC will also identify the tag to be used when completing the RP and IL EDDs.

Laboratory Procedures

**At the Originating (RP) Laboratory:**

1. Upon receipt of the IL sample list and grid transfer COC from the LC, locate the appropriate sample filter. If less than ¼ of the sample filter is available, contact the LC to identify an IL replacement sample.

2. Prepare new grids using the standard preparation methods at the Libby Site. If the sample was originally prepared directly, prepare new grids from the original filter. If the sample was originally prepared indirectly, prepare new grids from the same secondary filter utilized to prepare the grids for the original analysis. For media types where multiple filter replicates were prepared and analyzed (e.g., tree bark, duff), select only the first replicate (REP1) to serve as the IL sample. When preparing the grids, ensure a heavy carbon coat is applied to the collapsed filter to maximize durability of the grid preparations, but not too thick that it could interfere with obtaining diffraction patterns. Do not use grids with a GO area > 0.011 $mm^2$ for inter-labs unless you have written approval to do so from the LC.

3. Select the appropriate number of grids and examine GOs using the same TEM counting and stopping rules utilized in the original analysis, splitting the number of GOs examined evenly across the prepared grids. If, due to the counting rules, fewer than 10 GOs were analyzed in the RP, analyze an additional 5 GOs beyond what is needed (or until the maximum number of structures counting rule is met) to serve as alternate GOs in the event any are damaged. Regardless of the original stopping rules, the maximum number of GOs to be examined for RPs associated with ILs is 200 GOs. Place any remaining grids in storage.

4. Record the grid orientation using the Grid Orientation Form (see Attachment 4). Record the relevant magnification on the GO sketch form (see Attachment 5).

5. When performing the TEM analysis, identify the relative position of each structure within the GO using the template provided as Attachment 5. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst must record the structure number (i.e., the sequential integer recorded in the total column) which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any “close-call” non-asbestos mineral (NAM) structures (see LB-000066 for additional information on “close-call” NAM structures). As appropriate, NAM debris can be included in the sketch to provide additional information on relative structure location. Use a new sketch template for each GO. Do not prepare sketches for GOs where no asbestos or “close-call” NAM structures are observed.

6. Record the results using the most recent version of the TEM EDD. Identify the QC Type as “Repreparation” and use the tag indicated on the new COC provided to the laboratory for the grid shipment. If you have any questions as to what the tag should be, please contact the LC for verification.

7. If more than 10 GOs were evaluated in the RP analysis, the RP analyst or laboratory director will select the 10 GOs with the highest number of countable LA structures to re-analyze in the IL analysis. Except in the cases where the maximum number of structures counting rule was met prior to 10 GOs being analyzed, the RP analyst or laboratory director shall also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of countable LA structures, which may be analyzed in the event that the selected GO is damaged and cannot be re-evaluated.

8. Submit scanned copies of the cover letter that will accompany the RP grids to the IL laboratory (this cover letter will specify the GOs identified for IL analysis as well as the list of alternate GOs), the RP benchsheet(s), and the GO sketches to the QATS contractor for review. Unless informed otherwise, this step must be completed within 2 weeks of notification by the LC that a sample has been selected for IL analysis. For those analyses that are performed at both high and low magnification, indicate on the cover letter which GOs have been read at low magnification.

9. After the QATS contractor has reviewed the RP information and determined that it is complete, the QATS contractor (or their designee) will direct the RP laboratory to ship the grids to the IL laboratory indicated on the associated grid transfer COC.

10. Submit the RP TEM EDD using standard deliverable procedures, also copying QATS on the associated e-mail. The RP should have a stand-alone data package (i.e. there should be one data package for each RP analysis).

11. Ship the grid(s) for the IL sample to the appropriate laboratory using the grid transfer COC provided. When packaging the grids for shipping, secure the grids and add sufficient packaging material (e.g., bubble wrap, newspaper) to minimize movement during shipment. For each IL sample, include a cover letter that lists which GOs should be evaluated for each grid as well as the list of alternate GOs, which may be analyzed in the event that a selected GO is damaged and cannot be re-evaluated. The names of the grid and GOs provided in the cover letter should match exactly with those recorded on the RP TEM EDD (including dashes, underscores, and spaces). Also include any relevant grid preparation information that the IL will need to produce an EDD, which can be accomplished by providing a copy of the Data Entry 1 tab of the RP EDD or by copying the first page of the benchsheet for the RP analysis (ensuring that any structure information on the benchsheet is hidden). Include the completed grid orientation form.

12. After the laboratory has completed their IL analysis, they will request copies of the hard copy laboratory benchsheet(s), the GO sketches, and TEM EDD for the associated RP analysis.

13. If areas of discordance are noted, the senior laboratory analyst from the IL laboratory will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of asbestos types, etc.).

**At the Inter-laboratory:**

1. For each grid provided for IL analysis, place the grid into the TEM grid holder ensuring that the grid orientation matches that which was specified by the originating laboratory (see Attachment 4 for details) for the magnification at which the analysis will be performed. Otherwise, structures will be oriented differently and grid bars considered non-countable by one lab may be considered countable by the other. If the laboratory is unable to perform the IL analysis promptly after receipt of the grids, carefully examine the grids with a hand lens to determine if they are intact so that the RP laboratory can be notified quickly if the grids are damaged and a second RP needs to be prepared.

2. For the GOs identified for IL analysis, perform TEM analysis using the analysis method and counting rules specified on the referenced Analytical Requirement Summary Sheet (which is specified on the COC). Be sure that the grid and GO names match exactly with the names provided on the cover letter (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), <u>do not</u> arbitrarily select a different GO for evaluation, but utilize the list of 5 alternative GOs provided by the originating laboratory to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed). In the event that the number of intact alternate GOs is not sufficient to complete the IL analysis, the laboratory should notify the original laboratory and the LC. Unless specified otherwise by the LC, the original laboratory will perform a new RP analysis for IL evaluation.

3. When performing the IL analysis, identify the relative position of each structure within the GO using the template provided as Attachment 5. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst must record the structure number (i.e., the sequential integer recorded in the total column) which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any “close-call” NAM structures (see LB-000066 for

additional information on “close-call” NAM structures). As appropriate, NAM debris can be included in the sketch to provide additional information on relative structure location. Use a new sketch template for each GO. Do not prepare sketches for GOs where no asbestos or “close-call” NAM structures are observed.

4. Record the results using the most recent version of the TEM EDD. Identify the Laboratory QC Type as “Interlab.” The assigned Tag for the IL analysis must be the same as that for the RP analysis (i.e., if the RP analysis Tag is AL2, the IL analysis Tag is AL2), as indicated on the COC that accompanied the grid shipment.

5. Submit the IL TEM EDD using standard deliverable procedures, also copying QATS on the associated e-mail.

6. Contact the originating laboratory to request copies of the hard copy laboratory benchsheet(s), GO sketches, and TEM EDD for the associated RP analysis.

7. A senior laboratory analyst at the IL laboratory will assess the between-laboratory concordance based on a review of the benchsheets and any supporting documentation (spectra, photos, etc.). This will be done on both a GO-by-GO basis and on a structure-by-structure basis, using the Libby-specific recount concordance rules on Page 4 of this modification. The IL analyst will be responsible for determining matched and unmatched structures in basic accordance with NIST (1994). If areas of discordance are noted, the senior laboratory analyst will contact the originating laboratory to discuss the basis of the discordance. If necessary, the IL grids may be re-loaded into the TEM to examine structures in dispute. If the IL analyst finds discordances (i.e. false positives and/or negatives for one or more structures) in the RP’s analysis, the IL analyst will send their verified analysis report (prepared in basic accordance with NIST [1994]), to the RP laboratory for review. If the RP laboratory accepts the findings of the verified analysis, the IL laboratory can submit their evaluation summary as described below. If the RP laboratory contests the findings of the IL analyst’s verified analysis report, then they should request the grids back to re-examine them, and discuss the matter with the IL analyst until it is resolved. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc.).

8. Should reconciliation be necessary, only reconcile those GOs in which discrepancies were identified; a discrepancy is defined as either GOs or structures that do not meet the criteria described on **Page 4** of this laboratory modification. When performing reconciliation on a specific GO, only those structures in question need be recorded on the reconciliation benchsheet and EDD. In addition, the “total” structure number, from either the repreparation or the inter-laboratory analysis, of the structure for which a discrepancy was observed and investigated must be recorded in the “total” column of the reconciliation benchsheet and EDD.

9. Summarize the results of the IL analysis as described in NIST (1994) and document any changes, if necessary, in laboratory procedures or analyst training that were implemented to address noted discordances. Provide a copy of this report to the LC and the EPA QATS contractor using standard transmittal procedures. In addition, email notification of IL results (concordant or discordant) will be provided to the EPA QATS contractor as follows:

    - If the RP and IL analyses are in agreement, this email should indicate “The RP and IL results for sample *[xxxx]* were concordant.” No reconciliation EDD is necessary.

    - If the RP and IL analyses are not in agreement and either the RP or the IL analysis was subsequently determined to be correct, this email should indicate “The [RP or IL] results for sample *[xxxx]* were determined to be correct.” In this case, a reconciliation EDD is not necessary, but corrective actions may be necessary depending upon the nature of the discordance.

- If the RP and IL analyses are not in agreement and neither the RP nor the IL analysis was subsequently determined to be correct, this email should indicate "The RP and IL results for sample *[xxxx]* were discordant, the necessary corrective actions have been initiated, and reconciliation EDD is provided." The reconciliation EDD will provide the consensus results and should only include the GOs where discordances occurred. The original RP EDD and IL EDD should <u>not</u> be modified to reflect the consensus result that is indicated in the reconciliation EDD.

10. There should be one data package for each IL analysis. If reconciliation was performed, it should be included with the IL data package.

11. Ship the grid(s) back to the originating laboratory along with the following:

    - The IL hard copy benchsheet(s)
    - GO sketches from the IL analysis
    - NIST (1994) IL evaluation summary
    - The Reconciliation EDD (if prepared)

# ATTACHMENT 3

## Statistical Comparison of Two Poisson Rates

### 1.0 INTRODUCTION

An important part of the quality control (QC) plan for the Libby Asbestos Project is the repreparation and analysis of TEM grids for the quantification of asbestos. Because of random variation, it is not expected that results from repreparation analyses should be identical. This attachment presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

### 2.0 STATISTICAL METHOD

This method is taken from *Applied Life Data Analysis* (Nelson 1982). Input values required for the test are as follows:

N1 = Fiber count in first evaluation
S1 = Sensitivity of first evaluation
N2 = Fiber count in second evaluation
S2 = Sensitivity of second evaluation

The test is based on the confidence interval around the ratio of the two observed Poisson rates:

Rate 1 = N1 · S1
Rate 2 = N2 · S2
Ratio = Rate 1 / Rate 2

$$Lower\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1}{N2+1}\right) / F\left[\frac{1+\gamma}{2}; 2 \cdot N2 + 2, 2 \cdot N1\right]$$

$$Upper\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1+1}{N2}\right) \cdot F\left[\frac{1+\gamma}{2}; 2 \cdot N1 + 2, 2 \cdot N2\right]$$

where γ is the confidence interval (e.g., 0.95) and F[δ; df1, df2] is the 100δth percentile of the F distribution with df1 degrees of freedom in the numerator and df2 degrees of freedom in the denominator.

If the lower bound of the ratio is > 1, then it is concluded that rate 1 is greater than rate 2 at the 100(1-γ)% significance level. If the upper bound of the ratio is < 1, then it is concluded that rate 1 is less than rate 2 at the 100(1-γ)% significance level. Otherwise, it is concluded that rate 1 and rate 2 are not different from each other at the 100(1-γ)% significance level.

<u>Example:</u>

N1 = 4 structures
S1 = 0.0001 $(cc)^{-1}$
Rate 1 = 4 · 0.0001 = 0.0004 s/cc

N2 = 6 structures
S2 = 0.001 $(cc)^{-1}$
Rate 2 = 6 · 0.001 = 0.006 s/cc

γ = 0.95

$$Lower\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4}{6+1}\right) / F\left[\frac{1+0.95}{2}; 2\cdot 6+2, 2\cdot 4\right] = 0.014$$

$$Upper\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4+1}{6}\right) \cdot F\left[\frac{1+0.95}{2}; 2\cdot 4+2, 2\cdot 6\right] = 0.281$$

In this example, because the upper bound of the ratio is <1, it is concluded that Rate 1 (0.0004 s/cc) is less than Rate 2 (0.006 s/cc) at the 95% significance level.

An Excel spreadsheet tool has been developed to assist laboratories in the evaluation of repreparation analyses. This tool is available in the Libby Lab eRoom (see file “Poisson ratio comparison_v1.xlsx”).

## 3.0 REFERENCES

Nelson W. 1982. *Applied Life Data Analysis*. John Wiley & Sons, New York. pp 438-446.

# ATTACHMENT 4

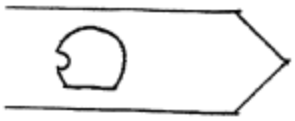
## Grid Orientation Form

**LB-000029E Grid Orientation Form**

This form only needs to be done once for each TEM model and grid type in use at the lab. Steps 3 and 4 are optional for analyses that are performed at high magnification and low magnification respectively.

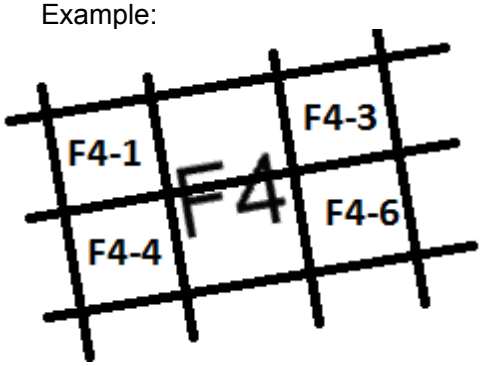
1. Load the grid into the specimen holder ensuring that the grid bars are oriented as close as possible to the north-south and east-west directions. Sketch the orientation of the grid in the specimen holder as shown in the example below. Indicate if the grid is loaded into the holder carbon side up or carbon side down.

   EXAMPLE:

2. At the magnification used to select suitable grid openings for analysis (approximately 200X), sketch the orientation of the grid bars and the letter F and provide a grid map if necessary, or insert a photo that provides the same information. Indicate the magnification used: _____

   Example:

3. Sketch the orientation of the grid bars and the letter F at low magnification (approximately 5,000X) for analysis of PCME structures only. Indicate the magnification used: _____

   Example:

4. Sketch the orientation of the grid bars and the letter F at high magnification (approximately 20,000X). Indicate the magnification used: _____

   Example:

# ATTACHMENT 5

## Grid Opening Template for Sketching the Relative Position of Observed Structures

## STRUCTURE LOCATIONS WITHIN GRID OPENING

****NOTE: Sketches only need to be completed for interlab analyses and repreps associated with interlabs*

Lab Name: _____ Lab Job Number: _____

Index ID: _____ Lab Sample ID: _____

Lab QC Type (circle one): Reprep for interlab Interlab

Grid: _____ Grid Opening: _____

upper left corner

traverse direction

Comments:

# Request for Modification
To
## Laboratory Activities
**LB-000030**

***Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.***
***File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:***

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin Title: Technical consultant
Company: Syracuse Research Corporation Date: 5 August 2003

Description of Modification:
All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance, morphology, and orientation relative to any nearby landmarks, if present.

Reason for Modification:
This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no loner need to be identified before analysis.

Potential Implications of this Modification:
There are no potential negative implications resulting from this standardization of QC procedures.

Laboratory Applicability (circle one): All Individual: _____

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: 8/14/03 (insert based on date of final approval)
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):

Technical Review: W J Brattin Date: 8/14/03
*(Laboratory Manager or designate)*

Project Review and Approval: [signature] Date: 8/14/03
*(Volpe: Project Technical Lead or designate)*

Approved By: Mary Goldade Date: 8/14/03
*(USEPA: Project Chemist or designate)*

# Request for Modification
to
## Laboratory Activities
**LB-000053**

***Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.***
***File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:***
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

**Method (circle one/those applicable):** TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other:_____

Requester: Mark Raney Title: Volpe Libby Project Technical Lead
Company: US DOT Volpe Center Date: 25 April 2007

**Description of Modification:**
This laboratory modification relates to the project specific SOP for Indirect Preparation of Air and Dust Samples for TEM analysis (SOP # EPA-LIBBY-08). SOP # EPA-LIBBY-8 provides a standardized procedure for the indirect preparation of Libby air and dust samples that minimizes the loss of sensitivity and allows for the retention of a portion of the original filter for archive whenever possible. The SOP indicates two general indirect preparation procedures for samples, one that includes ashing of the primary filter and one that does not include ashing of the primary filter.

For air samples, whether ashing is required is dependant on whether the air sample is considered a project investigative or a non-investigative sample. Investigative air samples are defined as samples specifically collected to characterize concentration values in air for use in risk assessment and risk management decision making. In general non-investigative air samples are defined as samples that are intended only to help characterize the exposure level of EPA workers at the site and are used mainly to support health and safety assessment and monitor removal activities.

Table 1 of this laboratory modification provides a list of which sample prefix codes shall be identified as investigative and which shall be identified as non-investigative. In cases where there is a conflict regarding sample type between the sample prefix as defined by this modification and the chain of custody instructions, the chain of custody instructions take precedent. ALL investigative air samples shall require ashing as part of the sample indirect preparation procedures. Dust samples will NOT automatically require ashing regardless whether the sample is considered an investigative or non-investigative sample. For dust samples, ashing will be incorporated opportunistically as part of the indirect preparation procedures as determined necessary by the analyst.

**Reason for Modification:**
Air samples for which ashing may be warranted include indoor samples collected from properties with elevated levels of organic particulates (e.g., due to cigarette smoke or use of a wood-burning stove). In these samples, ashing may further reduce particulate loading, thus allowing for an improved analytical sensitivity. It was determined that for investigative air samples which require lower sensitivities the additional sample preparation time for incorporating ashing as part of the indirect preparation procedures was warranted. For Libby non-investigative samples, which typically require a faster analytical turnaround and higher target sensitivity, it was determined that the longer ashing procedures are not needed. It was also determined that ashing would be included only opportunistically for dust samples, since historically only a few dust samples have required ashing.

**Potential Implications of this Modification:**

**Potential Implications of this Modification:**
There are no known negative implications of this modification. Positive impacts to this modification are clarification of sample preparation steps.

**Laboratory Applicability (circle one):** All Individual(s)

**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 12, 2006
*Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.*

**Data Quality Indicator (circle one):** Please reference definitions on reverse side for direction on selecting data quality indicators:

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

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

Technical Review: _____ Date: _____
*(Laboratory Manager or designate)*

Project Review and Approval: _____ Date: 12/08/06
*(Volpe: Project Technical Lead or designate)*

Approved By: _____ Date: 12/7/06
*(USEPA: Project Chemist or designate)*

## DATA QUALITY INDICATOR DEFINITIONS

***Reject*** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect 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.

**TABLE 1 – Active Sample Prefixes**
(October 5th, 2007 update)

| Sample Prefix | Operable Unit | Prefix Description | Sampling and Analysis Plan Date | Investigative OR Non-Investigative? |
|---|---|---|---|---|
| 1 | 4 | Phase 1 | Dec 99 (Rev 0)<br>Jan 00 (Rev 1) | INVESTIGATIVE |
| 2 | 4 | Phase 2 | Mar 01 (Rev 0 ) | INVESTIGATIVE |
| 1R | 4 | Phase 1R | Sept 00 (Rev 1)<br>Nov 2003 | NON |
| CS | 4 | Contaminant Soil Screening Study | Apr 02 (Rev 0)<br>May 03 (Rev 1) | INVESTIGATIVE |
| 1D | 4 | Design Phase | Nov 03 | INVESTIGATIVE |
| CE | 4 | Clean-up Evaluation | Nov 05 | INVESTIGATIVE |
| SQ | 4 | SQAPP | Jun 05 (Rev 0)<br>Aug 05 (Rev 1) | INVESTIGATIVE |
| CR | 4 | Cumulative Risk Pilot Study | Dec 2005 | INVESTIGATIVE |
| AA | 1,2,4,5,6 | Outdoor Ambient Air | Dec 2006<br>July 2007 (addendum) | INVESTIGATIVE |
| DM | NA | Demolition Sampling | May 2007 | NON |
| DP | 4 | Dust Sampling Pilot Study | May 2007 | INVESTIGATIVE |
| TT | 7 | Troy Asbestos Project Evaluation | Jul 2007 | INVESTIGATIVE |
| IN | 4 | Indoor Activity-Based Sampling | Jul 2007 | INVESTIGATIVE |
| EX | 4 | Outdoor Activity-Based Sampling | Jul 2007 | INVESTIGATIVE |
| P1 | 3 | OU 3 Phase 1 Remedial Investigation | Oct 2007 | INVESTIGATIVE |
| EP | 1 | Export Plant—Data Gaps and Collection | Sept 2007 | INVESTIGATIVE |
| SL | 5 | Stimson Lumber Mill—Initial Soil Data Gap Investigation & Building Data Gap Sampling | Sept 2007<br>Nov 2007 | INVESTIGATIVE |
| FC | 4 | Flower Creek Investigation | Aug 2007 | INVESTIGATIVE |
| | | | | |

**NOTE: If an analyst encounters a sample requiring an indirect preparation that has a prefix that is NOT included in Table 1 above, the analyst shall not proceed with any sample preparation. Instead, the analyst shall archive the sample and contact the CDM laboratory coordinator for clarification on whether the sample is considered an investigative sample and if it requires ashing.**

**Update Approvals:**

Volpe Project Review and Approval: _____ Date: 1/17/08
(Volpe: Technical Lead or designate)

EPA Review and Approval: _____ Date: 2/4/08
(EPA: Project Chemist or designate)

**TABLE 1 – Active Sample Prefixes**
(May 11th, 2007 update)

| Sample Prefix | Prefix Description | Investigative OR Non-Investigative? |
|---|---|---|
| 1 | Phase 1 | INVESTIGATIVE |
| 2 | Phase 2 | INVESTIGATIVE |
| 1R | Phase 1R | NON |
| CS | Contaminant Screening Study | INVESTIGATIVE |
| 1D | Design Phase | INVESTIGATIVE |
| CE | Clean-up Evaluation | INVESTIGATIVE |
| SQ | SQAPP | INVESTIGATIVE |
| CR | Cumulative Risk | INVESTIGATIVE |
| AA | Outdoor Ambient Air | INVESTIGATIVE |
| DM | Demo Sampling | NON |
| DP | Dust Pilot | INVESTIGATIVE |
| TT | Troy Asbestos Project Evaluation | INVESTIGATIVE |
| IN | Indoor Activity-Based Sampling | INVESTIGATIVE |
| EX | Outdoor Activity-Based Sampling | INVESTIGATIVE |

**NOTE:** **If an analyst encounters a sample requiring an indirect preparation that has a prefix that is NOT included in Table 1 above, the analyst shall not proceed with any sample preparation. Instead the analyst shall archive the sample and contact the CDM laboratory coordinator for clarification on whether the sample is considered an investigative sample and if it requires ashing.**

Update EPA Approval: Mary Goldade 6/5/07

**TABLE 1 – Active Sample Prefixes**
(April 25th, 2007 update)

| Sample Prefix | Prefix Description | Investigative OR Non-Investigative? |
|---|---|---|
| 1 | Phase 1 | INVESTIGATIVE |
| 2 | Phase 2 | INVESTIGATIVE |
| 1R | Phase 1R | NON |
| CS | Contaminant Screening Study | INVESTIGATIVE |
| 1D | Design Phase | INVESTIGATIVE |
| CE | Clean-up Evaluation | INVESTIGATIVE |
| SQ | SQAPP | INVESTIGATIVE |
| CR | Cumulative Risk | INVESTIGATIVE |
| AA | Outdoor Ambient Air | INVESTIGATIVE |
| DM | Demo Sampling | NON |
| TT | Troy Asbestos Project Evaluation | INVESTIGATIVE |
| IN | Indoor Activity-Based Sampling | INVESTIGATIVE |
| EX | Outdoor Activity-Based Sampling | INVESTIGATIVE |

NOTE: **If an analyst encounters a sample requiring an indirect preparation that has a prefix that is NOT included in Table 1 above, the analyst shall not proceed with any sample preparation. Instead the analyst shall archive the sample and contact the CDM laboratory coordinator for clarification on whether the sample is considered an investigative sample and if it requires ashing.**

**TABLE 1 – Active Sample Prefixes**
(December 6th, 2006)

| Sample Prefix | Prefix Description | Investigative OR Non-Investigative? |
|---|---|---|
| 1 | Phase 1 | INVESTIGATIVE |
| 2 | Phase 2 | INVESTIGATIVE |
| 1R | Phase 1R | NON |
| CS | Contaminant Screening Study | INVESTIGATIVE |
| 1D | Design Phase | INVESTIGATIVE |
| CE | Clean-up Evaluation | INVESTIGATIVE |
| SQ | SQAPP | INVESTIGATIVE |
| CR | Cumulative Risk | INVESTIGATIVE |
| AA | Ambient Air | INVESTIGATIVE |
| DM | Demo Sampling | NON |

**NOTE:** **If an analyst encounters a sample requiring an indirect preparation that has a prefix that is NOT included in Table 1 above, the analyst shall not proceed with any sample preparation. Instead the analyst shall archive the sample and contact the CDM laboratory coordinator for clarification on whether the sample is considered an investigative sample and if it requires ashing.**

## Raney, Mark

**From:** Raney, Mark
**Sent:** Friday, December 08, 2006 4:44 PM
**To:** Anni Autio (E-mail)
**Cc:** Lynn Woodbury (E-mail); 'Goldade.Mary@epamail.epa.gov'
**Subject:** RE: LAB MODs #53 EPA Approved

LB-000053 (MR 2-08-06 email)..

Anni,

Attached is a copy of the final approved version of Lab MOD #LB-000053. The attached modification will be considered effective immediately following discussion during Tuesday's lab call. Please distribute the attached to the laboratories for their review prior to Tuesday's call.

Note: I will be mailing the signed hardcopy to Mary for her signature. Mary will then forward the final/original signed hardcopy to CDM.

Let me know if you have any questions.

Mark.

-----Original Message-----
From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]
Sent: Thursday, December 07, 2006 5:14 PM
To: Raney, Mark
Cc: Anni Autio (E-mail); Lynn Woodbury (E-mail)
Subject: Re: LAB MODs #53 EPA Approved

EPA approves Lab Mod 53 with changes as attached.
(See attached file: LB-000053 (MR 12-06-06 email) (MG 12-7-06).doc)

"Raney, Mark" <Mark.Raney@Volpe.dot.gov>

12/06/2006 03:31 PM

To
Mary Goldade/EPR/R8/USEPA/US@EPA, "Mary Goldade (E-mail 2)" <mgoldade@peakpeak.com>

cc
"Anni Autio (E-mail)" <autioah@cdm.com>, "Lynn Woodbury (E-mail)" <woodbury@syrres.com>

Subject
LAB MODs #53 and 29b

Mary,

Attached is a draft for your review of Lab Mod LB-000053 (to accompany the indirect SOP).

I have also reviewed Lab MOD LB-000029b. I have no comments to LB-000029b other than a formatting comment. The numbering format needs to be corrected for the steps on Attachment 2, under "At the Interlab Laboratory".

Let me know if you have any questions.

Mark.

Mark E. Raney, RTV-4E
Environmental Engineer / Project Manager
US DOT / RITA / Volpe Center
55 Broadway
Cambridge, MA 02142
raney@volpe.dot.gov
Office: 617-494-2377
Mobile: 617-694-8223
Fax: 617-494-2789

<<LB-000053 (MR 12-06-06 email).doc>> <<LB-000029b v7.doc>> [attachment "LB-000029b v7.doc" deleted by Mary Goldade/EPR/R8/USEPA/US]

NISTIR 5351

# Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0

Shirley Turner
Eric B. Steel

U.S. DEPARTMENT OF COMMERCE
Technology Administration
National Institute of Standards
and Technology
Microanalysis Research Group
Surface and Microanalysis Science Division
Chemical Science & Technology Laboratory
Gaithersburg, MD 20899

March 1994

U.S. DEPARTMENT OF COMMERCE
Ronald H. Brown, Secretary

TECHNOLOGY ADMINISTRATION
Mary L. Good, Under Secretary for Technology

NATIONAL INSTITUTE OF STANDARDS
AND TECHNOLOGY
Arati Prabhakar, Director

## Preface

This Interagency Report (IR) is one of a series of IRs that will form the basis of a method for analysis of airborne asbestos by transmission electron microscopy. The form and style of the American Society for Testing and Materials (ASTM) was adopted as a standard format for this series of reports.

## 1. Scope

1.1 This test method describes a procedure for verified analysis of asbestos by transmission electron microscopy.

1.2 The method is applicable only when sufficient information has been collected during the analyses of a grid square so that individual asbestos structures can be uniquely identified.

1.3 The method is written for the analysis of a grid square by two TEM operators but can be used for more than two operators with slight modifications. Due to the analysis of a grid square by more than one TEM operator, the test method can be applied only when contamination and beam damage of particles are minimized. The two TEM operators can use the same TEM for the analysis or the analyses can be done on different TEMs (in the same or in different laboratories).

1.4 The method can be used with any set of counting rules applied by all analysts. Though the method describes verification of asbestos particles, the method can also be used for verification of analyses of nonasbestos particles if all analysts use the same counting rules.

## 2. Terminology

2.1 *Definitions:*

2.1.1 *TEM*--transmission electron microscope.

2.1.2 *grid square, grid opening*--an area on a grid used for analysis of asbestos by transmission electron microscopy.

2.1.3 *verified analysis*--a procedure in which a grid opening is independently analyzed for asbestos by two or more TEM operators and in which a comparison and evaluation of the correctness of the analyses are made by a verifying analyst. Detailed information -- including absolute or relative location, a sketch, orientation, size (length, width), morphology, analytical information and identification -- is recorded for each observed structure.

2.1.3.1 *Discussion*--Verified analysis can be used to determine the accuracy of operators and to determine the nature of problems that the analyst may have in performing accurate analyses. Verified counts can be used to train new analysts and to monitor the consistency of analysts over time.

2.2 *Description of Terms Specific to This Standard:*

2.2.1 *counting rules*--rules used to determine the amount of asbestos present in an asbestos- containing sample. Counting rules are a part of most methods for analysis of asbestos by transmission electron microscopy including the AHERA method and the ISO method (see definitions below).

2.2.2 *AHERA method*[1]--procedure for analysis of asbestos by transmission electron microscopy developed by the Environmental Protection Agency with subsequent modifications by the National Institute of Standards and Technology.

2.2.3 *ISO method*[2]--procedure for analysis of asbestos by transmission electron microscopy developed by the International Standards Organization.

2.2.4 *particle*--an isolated collection of material deposited on a grid or filter.

2.2.5 *structure*--a particle or portion of a particle that contains asbestos and that is considered countable under the method used for asbestos analysis. A structure is a basic unit used in many methods of asbestos analysis to report the amount of asbestos present in a particle.

2.2.6 *TEM operator, TEM analyst*--person that analyzes a grid square by transmission electron microscopy to determine the presence of asbestos.

2.2.7 *verifying analyst*--person that compares the analyses of a grid square by two or more TEM operators. The reported asbestos is compared on a structure-by-structure basis by the verifying analyst. Structures that are not matched are relocated and reanalyzed by the verifying analyst. The verifying analyst is

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[1]Code Fed. Reg. 1987, *52* (No. 210), 41826-41905.

[2]ISO 10312 1993, in press.

preferably not one of the TEM operators. If this cannot be avoided, the job of verifying analyst should be rotated between the TEM operators.

2.2.8 *TEM analysis form*--form on which the analysis of a grid square is recorded. The information recorded for a verified analysis should include at least a sketch of the structure and information related to the absolute or relative location, size, identification and analytical data for the reported structures.

2.2.9 *report form*--form on which the evaluation of verified analyses is summarized. The form should be identical to or include all information given in Figure X1.1 of Appendix X1.

2.2.10 *SR (structures reported)*--the number of structures reported by a TEM analyst.

2.2.11 *TP (true positive)*--structure that is: 1) reported by both TEM operators or 2) reported by one operator and confirmed by the verifying analyst, or 3) reported by neither TEM operator but is found by the verifying analyst. The three types of true positives are discussed in the next three terms.

2.2.12 *TPM (true positive-matched)*--structure that is reported on the TEM analysis forms of both TEM operators.

2.2.12.1 *Discussion*--To qualify as a match, the structures should be comparable in the following characteristics: 1) absolute or relative location, 2) appearance in the sketch, 3) orientation, 4) size (length, width), 5) morphology (shape, hollow tube), 6) analytical information (chemistry and/or diffraction data), and 7) identification. In addition, the structures should be reported as countable by both analysts.

2.2.13 *TPU (true postive-unmatched)*--structure that is reported on the TEM analysis form of only one operator and that is confirmed as countable by the verifying analyst.

2.2.14 *TPV (true positive found by verifying analyst)*--structure not found by the two TEM operators but found by the verifying analyst.

2.2.15 *TNS (total number of structures)*--the number of structures determined to be in a grid opening by verified analysis of the grid opening. This value corresponds to the number of unique true positives found by the TEM operators and the verifying analyst.

2.2.15.1 *Discussion*--The value for the total number of structures is not necessarily the actual number on the grid square because both the TEM analysts and the verifying analyst may have missed one or more structures. The probability of a missed structure, however, decreases with an increased number of analysts.

2.2.16 *FN (false negative)*--structure that has not been reported as countable by one of the TEM analysts. False negatives can be divided into two categories-type A and type B as discussed in the next two terms.

2.2.17 *FNA (false negative-type A)*--false negative that was recorded on a TEM analyst's TEM analysis form but not reported as a structure. Some reasons for this type of false negative include: 1) structure misidentified as nonasbestos, 2) confusion with the counting rules, 3) incorrect length determination.

2.2.18 *FNB (false negative-type B)*--false negative that was not recorded on a TEM analyst's TEM analysis form. A reason for this type of false negative is that a structure was missed by an analyst.

2.2.19 *FP (false positive)*--reported particle that is incorrectly identified as a structure. Some reasons for false positives include: 1) structures counted more than one time, 2) materials misidentified as asbestos, 3) confusion with the counting rules, 4) incorrect length determination.

2.2.20 *TN (true negative)*--reported particle that is correctly characterized as zero structures.

2.2.21 *NL (not located structure)*--structure reported on one TEM analyst's TEM analysis form that cannot be located by the verifying analyst.

2.2.21.1 *Discussion*--The value for NL should be zero for most verified analyses, especially if the grid has not been removed from the TEM between the two analysts' counts. If, however, a grid has been removed from an instrument, there is a small possibility of fiber loss.

2.2.22 *AMB (ambiguous structure)*--a structure that 1) is identified as a structure by only one TEM operator and 2) is found by the verifying analyst but cannot be unambiguously identified as a structure due to beam damage, contamination, or other factors.

## 3. Significance and Use

3.1 The analysis of asbestos by transmission electron microscopy is important for the determination of the cleanliness of air or water and for research purposes. Verified analyses provide more accurate values for the concentration of asbestos on a grid opening than obtained by other methods. The accuracy should increase with an increased number of analysts participating in the verified count.

3.2 The test method can be used as part of a quality assurance program for asbestos analyses and as a training procedure for new analysts. The values for TP/TNS and FP/TNS can be plotted *vs* time on control charts to show improvements or degradations in the quality of the analyses. Experienced analysts should attain TP/TNS values $\geq 0.85$ and FP/TNS values $\leq 0.05$. The test method can be used to characterize the types and, in many cases, the causes of problems experienced by TEM analysts.

3.3 The average of values obtained for TP/TNS and FP/TNS can be used to determine the analytical uncertainty for routine asbestos analyses.

## 4. Procedure

NOTE 1-- This test method involves two TEM operators and a verifying analyst. The steps discussed in items 4.1 and 4.2 are to be followed by the person coordinating the analyses by the TEM operators. This person can be one of the TEM operators, the verifying analyst or an independent person (e.g., a quality assurance officer). The steps discussed starting with item 4.3 are to be followed by the verifying analyst.

4.1 Obtain analyses of a grid square for asbestos by two TEM operators. Conduct the analyses independently so that the second operator has no knowledge of the results obtained by the first operator.

4.1.1 Require that the TEM operators record on the TEM analysis form information related to the absolute location of the structures or conduct analyses so that the relative location of the structures can be compared.

NOTE 2– The absolute location of the structures can be recorded by various means including use of a digital voltmeter or computer readable stepping motors to record the position of a structure. To preserve information about the relative location of the reported structures, the analyses must be conducted so that both analysts: 1) orient the grid in the TEM in the same fashion, 2) start the analysis from the same corner of the grid square, 3) initially scan in the same direction, and 4) scan the grid square in parallel traverses.

4.1.2 Require that the TEM operators record on the TEM analysis form a sketch of the structure, the dimensions of the structure, analytical data and whether the structure is countable. The sketch of the structure should include any nearby features that could aid in subsequent identification - for instance, nearby particles, sample preparation features or grid bars.

4.2 Submit the analyses of the two TEM operators to the verifying analyst.

NOTE 3– The remainder of this section describes procedures to be followed by the verifying analyst. The procedure for comparison of the TEM analysis forms is given in items 4.3-4.6 and examples of comparisons of count sheets are given in Figs. X2.1-X2.9 of Appendix 2. Appendix 3 contains a summary of the comparison process (Fig. X3.1) and a flow chart for comparison of structures in the TEM (Fig. X3.2). The procedure for completion of the report form is given in item 4.7.

4.3 Compare the two TEM analysis forms on a structure-by-structure basis. If a match of asbestos structures is observed, label both sketches with a TPM(number) either in the sketch box or in a column specifically designated for verified counts. An example is given in Fig. X2.1 of Appendix X2.

NOTE 4– The next step in the procedure (item 4.4) is optional. The most prudent approach is to examine unmatched structures in the TEM (item 4.5).

4.4 Determine if the status of any of the unmatched structures can be unambiguously decided by examining the TEM analysis forms. If there is ambiguity in determining the status of a structure, the verifying analyst must examine the structure in the TEM as described in items 4.5-4.6. The comparison of TEM analysis forms and labelling of unmatched structures can be relatively straight foward as shown in Fig. X2.2 - X2.4 of Appendix X2 or more complex as described in the next item.

4.4.1 For most cases, the identification of true positives, false positives and false negatives can be done on a structure-by-structure basis. This cannot be done, however, in cases where analysts determine different numbers of countable structures in an asbestos-containing particle. In such cases, both analysts should be assigned one TPM(number) for identifying the particle as containing countable asbestos. The remaining structures are assigned TPU, FP or FN depending on the particular situation. Examples of such cases are given in Fig. X2.5 and Fig. X2.6 of Appendix X2.

4.5 Determine the status of any remaining unlabelled structures by examining the grid square in the TEM. Examples of TEM analysis forms containing structures that must be examined by transmission electron microscopy are given in Figs. X2.7 - X2.9 of Appendix 2. For each unlabelled structure requiring examination by transmission electron microscopy, follow items 4.5.1-4.5.7 and 4.6 until the structure is labelled. If there is another unlabelled structure, go back to item 4.5.1 and repeat the procedure. Continue until all structures are labelled. A summary flow chart for examination by TEM is given in Fig. X3.2. The procedure and flowchart do not cover the counting discrepancy discussed in item 4.4.1. If such a situation is recognized, the verifying analyst should follow the procedure given in item 4.4.1 and in the examples in Figs. X2.5 and X2.6.

NOTE 5-- The procedure in items 4.5.1-4.5.7 should cover the great majority of cases encountered when attempting to determine the status of the structures. There may, however, be more complex situations not covered in the procedure. If so, the verifying analyst should apply the basic principles outlined in items 4.5.1-4.5.7 and 4.4.1.

4.5.1 Determine if the reported structure can be located. If the structure cannnot be found, label the reported structure NL (place the label next to the sketch or in a column specifically designated for verified analyses).

4.5.2 If the reported structure is found, determine if a judgement can be made as to its countability. If the structure cannot be judged as to its countability due to beam damage, contamination or other factors, label the reported structure AMB.

4.5.3 If a judgement can be made as to the countability of the reported structure, determine if the structure is countable. If the reported structure is not countable, label it FP(number). A unique number is given to the FP label so that it can be specifically referred to in the report form. Optional: Check the other analyst's TEM analysis form. If the other analyst sketched the particle and correctly reported it as noncountable, label the particle TN(number). Note: The values for TN are not recorded on the report form.

4.5.4 If the reported structure is correctly identified as a structure, determine if it was reported as countable elsewhere on the same analyst's TEM analysis form (i.e., the analyst counted the structure twice). If it is a duplicate, label the reported structure FP(number).

4.5.5 If the reported structure is not a duplicate, label the structure TPU(number).

4.5.6 Determine if the other TEM operator recorded a sketch of the structure. If the other TEM operator did not report the structure on his/her TEM analysis form, place an FNB(number) on their TEM analysis form in the approximate location where the structure should have been found. The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.5.7 If the other TEM operator recorded a sketch of the structure, label the sketch with an FNA(number). The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.6 Countable asbestos structures reported by neither TEM operator but found by the verifying analyst in the course of examining a grid square should be recorded on a separate TEM analysis form and labelled

TPV(number). The TEM operators should be assigned an FNA(number) or FNB(number) as described in items 4.5.6-4.5.7.

4.7 Complete the report form as described in items 4.7.1-4.7.10.

4.7.1 Complete the heading of the report form and fill in the initials or names of the two TEM operators on the first line of the report form table.

4.7.2 Count the number of asbestos structures obtained by each analyst and enter the value as SR (structures reported) on the report form.

4.7.3 Determine the number of true positives that are matched (TPM), the number of true positives that are unmatched (TPU) and the total number of true positives (TP) obtained for each TEM operator on the grid square and enter the values on the report form.

4.7.4 Determine and record on the report form the number of true positives found by the verifying analyst (TPV).

4.7.5 Determine and record on the report form the total number of structures (TNS) on the grid square.

4.7.6 Determine and record on the report form for each operator the following: 1) the number of false positives (FP), 2) the number of false negatives (FN), 3) the number of false negatives of type A and type B (FNA, FNB), 4) the number of structures that were not located (NL) and 5) the number of ambiguous structures (AMB).

4.7.7 Determine and record the values for TP/TNS, FP/TNS to two decimal places.

4.7.8 List on the report form the suspected reasons for the false positives obtained by each analyst. Some examples would be as follows: incorrect length measurement, structures counted twice, problem with interpretation of the counting rules, misidentification of a structure.

4.7.9 List on the report form the suspected reasons for false negatives (FNA and FNB). Some examples would be: incorrect length measurement, problem with interpretation of the counting rules, misidentification of material as asbestos, possible loss of sense of direction, and insufficient overlap of traverses.

4.7.10 Append any other relevant comments to the report form (quality of the preparation, etc.).

4.8 Check the numbers on the report form using the equations given in the calculation section.

## 5. Calculation

5.1 The values on the report form should be consistent with the following equations:

For both analyses:

$$TNS = TPM + TPU(\text{Operator 1}) + TPU(\text{Operator 2}) + TPV$$

For a given analysis:

$$SR = TP + FP + NL + AMB$$

$$TP = TPM + TPU$$

$$FN = FNA + FNB$$

$$TNS = TP + FN$$

$$1 = TP/TNS + FN/TNS$$

## 6. Precision and Bias

6.1 To determine the precision of the method, independent verified analyses were conducted by operators in two laboratories on a set of 21 grid squares. The mean value for TNS for the data set was 16.2 structures/grid square and the pooled standard deviation of the pairs of verified count determinations was 1.12 structures/grid square. The confidence at approximately the 95% level (2 standard deviations) of a reported verified count value in this data set is 2.24 structures/grid square or 13.9% of the mean value for TNS. We use 13.9% as an estimate of the imprecision of the method.

NOTE 6-- The differences in the values obtained for the independent verified analyses described in item 6.1 are, for the most part, due to differences in interpretation of the counting rules. The structures analyzed in the study were complex and therefore the imprecision estimate discussed above likely represents an upper bound to the imprecision for the method.

6.2 The bias in the method will vary depending upon interpretation of the counting rules used in the analysis by the TEM operators and verifying analyst.

## 7. Keywords

7.1 asbestos; quality assurance; transmission electron microscopy; verified analysis

## APPENDIXES

**(Nonmandatory Information)**

## X1. TEST REPORT FORM

Fig. X1.1 The following format is suggested for use by the verifying analyst to report the comparison of the TEM operators' TEM analysis forms.

Grid box: _____ Date: _____

Grid slot: _____ Verifying Analyst: _____

Grid square: _____

| | Analysis 1 | Analysis 2 |
|---|---|---|
| TEM Operator | | |
| Structures Reported (SR) | | |
| True Positives (TP) | | |
| *TPM | | |
| TPU | | |
| *TPV | | |
| *Total # Structures (TNS) | | |
| False Positives (FP) | | |
| False Negatives (FN) | | |
| FNA | | |
| FNB | | |
| Not Located (NL) | | |
| Ambiguous (AMB) | | |
| TP/TNS | | |
| FP/TNS | | |

*The values for these items will be the same for both analyses.

Test Report Form (continued)

1) List details of suspected reasons for false positives. For each analyst describe reasons for FP1, FP2, FP3, etc. Note - it may not be possible to determine the reason for false positives for some structures.

2) List details of suspected reasons for false negatives (type A and type B). For each analyst describe reasons for FNA1, FNA2, etc.; FNB1, FNB2, etc. Note - it may not be possible to determine the reasons for false negatives for some structures.

## X2. EXAMPLES OF COMPARISONS OF TEM ANALYSIS FORMS

[Note: The TEM analysis forms shown in the examples are abbreviated and do not contain analysis information. The AHERA counting rules (1987) were used for all analyses.]

Analyst 1

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 1.3 | 0.1 | | TPM1 | 1 | Chr |
| 0.7 | 0.1 | | TPM2 | 1 | Chr |
| 1.0 | 0.1 | | TPM3 | 1 | Chr |

Analyst 2

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 1.3 | 0.1 | | TPM1 | 1 | Chr |
| 1.0 | 0.1 | | TPM3 | 1 | Chr |
| 0.7 | 0.1 | | TPM2 | 1 | Chr |

Fig. X2.1 Example of matching structures on two TEM analysis forms (refer to item 4.3 of the procedure). Three structures on a grid square were found by both analysts. The relative order of the last two structures is different on the two TEM analysis forms; this may be due to the nature of the traverses by the analysts. Matching structures are indicated by TPM(number).

Analyst 1

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 1.3 | 0.1 | | TPM1 | 1 | Chr |
| 0.7 | 0.1 | | TPM2 | 1 | Chr |
| 1.0 | 0.1 | | TPM3 | 1 | Chr |
| 0.7 | 0.1 | | FP1 | 1 | Chr |

Analyst 2

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 1.3 | 0.1 | | TPM1 | 1 | Chr |
| 1.0 | 0.1 | | TPM3 | 1 | Chr |
| 0.7 | 0.1 | | TPM2 | 1 | Chr |
| | | | | | |

Fig. X2.2 Example of determining the status of an unmatched structure from TEM analysis forms (refer to item 4.4 of the procedure). Three of the structures match in the two analyses. The last structure of analyst 1 is unmatched but can be seen from the TEM analysis form to be a duplicate of the second structure obtained by the same analyst (the two structures have the same identification, dimensions, orientation and a similar nearby particle). The duplicate structure is therefore assigned an FP1.

Analyst 1

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 0.6 | 0.1 | | TPU1 | 1 | Chr |

Analyst 2

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 0.6 | 0.1 | | FNA1 | 0 | Chr |

Fig. X2.3 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the structure. However, analyst 2 has reported that the particle is not a structure (the cause of this oversight is not known). Analyst 1 is assigned a TPU1 and analyst 2 an FNA1.

Analyst 1

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 0.4 | 0.1 | / | FP1 | 1 | Chr |

Analyst 2

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 0.4 | 0.1 | / | TN1 | 0 | Chr |

Fig. X2.4 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the particle on both TEM analysis forms. However, analyst 1 has reported that the particle is a structure (the cause of this oversight is not known). Analyst 1 is assigned an FP1 and analyst 2 a TN1.

Analyst 1

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 1 | 0.6 | | TPM1<br>FNA1 | 1 | Chr |
| | | | | | |
| | | | | | |

Analyst 2

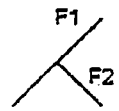
| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| | |   | | | |
| 1 | 0.1 | F1 | TPM1 | 1 | Chr |
| 0.6 | 0.1 | F2 | TPU1 | 1 | Chr |

Fig. X2.5 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one countable structure and analyst 2 has reported two countable structures. Under the AHERA counting rules, analyst 2 is correct. The structure reported by analyst 1 is assigned both a TPM1 and an FNA1. The two structures reported by analyst 2 are assigned a TPM1 and a TPU1, respectively.

Analyst 1

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 5 | 3 | | TPM1 | 1 | Chr |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

Analyst 2

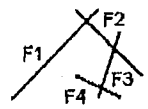
| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| | |   | | | |
| 5 | 0.1 | F1 | TPM1 | 1 | Chr |
| 3 | 0.1 | F2 | FP1 | 1 | Chr |
| 2 | 0.1 | F3 | FP2 | 1 | Chr |
| 1 | 0.1 | F4 | FP3 | 1 | Chr |

Fig. X2.6 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one structure and analyst 2 has reported four structures. Under the AHERA counting rules, analyst 1 is correct. The structure reported by analyst 1 is assigned a TPM1. The first structure reported by analyst 2 is labelled TPM1 and the remaining three reported structures are labelled FP1-FP3.

Analyst 1

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 0.4 | 0.1 | | | 0 | Chr |

Analyst 2

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 0.6 | 0.1 | | | 1 | Chr |

a

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 0.4 | 0.1 | | FNA1 | 0 | Chr |

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 0.6 | 0.1 | | TPU1 | 1 | Chr |

b

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 0.4 | 0.1 | | TN1 | 0 | Chr |

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 0.6 | 0.1 | | FP1 | 1 | Chr |

c

Fig. X2.7 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same asbestos-containing particle as indicated by the identification and orientation of the fiber and by the presence of a similar particle nearby. However, the dimensions reported by the analysts differ and analyst 1 has reported zero structures and analyst 2 has reported one structure. The verifying analyst should determine the correct length of the fiber and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 2 is assigned a TPU1 and analyst 1 an FNA1. c) A second possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 1 is assigned a TN1 and analyst 2 an FP1.

Analyst 1

| Length (um) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 1.3 | 0.1 | | TPM1 | 1 | Chr |
| 0.6 | 0.1 | | | 1 | Chr |
| 1.0 | 0.1 | | TPM2 | 1 | Chr |

Analyst 2

| Length (um) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 1.3 | 0.1 | | TPM1 | 1 | Chr |
| 1.0 | 0.1 | | TPM2 | 1 | Chr |
| | | | | | |

a

Fig. X2.8 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Analyst 1 has reported one structure that analyst 2 has not reported. The verifying analyst should attempt to find the particle and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is assigned a TPU1 and analyst 2 is assigned an FNB1. c) Another possible outcome is that the reported structure is not located. Analyst 1 is assigned an NL. Other possibilities (not illustrated) are that analyst 1 is incorrect (the particle is then labelled FP) or that the structure is too contaminated for characterization (the particle is then labelled AMB).

Analyst 1

| Length (um) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 1.3 | 0.1 | | TPM1 | 1 | Chr |
| 0.6 | 0.1 | | TPU1 | 1 | Chr |
| 1.0 | 0.1 | | TPM2 | 1 | Chr |

Analyst 2

| Length (um) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 1.3 | 0.1 | | TPM1 | 1 | Chr |
| 1.0 | 0.1 | | FNB1<br>TPM2 | 1 | Chr |
| | | | | | |

b

| Length (um) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 1.3 | 0.1 | | TPM1 | 1 | Chr |
| 0.6 | 0.1 | | NL1 | 1 | Chr |
| 1.0 | 0.1 | | TPM2 | 1 | Chr |

| Length (um) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 1.3 | 0.1 | | TPM1 | 1 | Chr |
| 1.0 | 0.1 | | TPM2 | 1 | Chr |
| | | | | | |

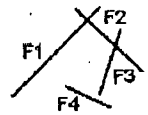
c

Fig. X2.8 (caption on previous page).

Analyst 1

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 5 | 3 | | | 1 | Chr |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

Analyst 2

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| | |   | | | |
| 5 | 0.1 | F1 | | 1 | Chr |
| 3 | 0.1 | F2 | | 1 | Chr |
| 2 | 0.1 | F3 | | 1 | Chr |
| 1 | 0.1 | F4 | | 1 | Chr |

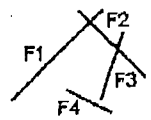
a

Fig. X2.9 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same particle as indicated by the identification and orientation of the fibers. However, analyst 1 has recorded all fibers as touching (or intersecting) and has therefore counted the fiber arrangement as one structure under the AHERA method. Analyst 2 has reported four structures. The verifying analyst should find and examine the arrangement in the TEM to determine if the fiber labelled as F4 by analyst 2 is touching or intersecting the fiber labelled as F3. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is then assigned a TPM1 and analyst 2 is assigned a TPM1 and three FPs. Other possibilities (not illustrated) are that analyst 2 is correct (the structures reported by analyst 2 are then assigned a TPM and 3 TPUs and the structure reported by analyst 1 is assigned a TPM) or that the particle is too contaminated for identification (the structure reported by analyst 1 is then assigned a TPM and those reported by analyst 2 are assigned a TPM and three AMBs).

Analyst 1

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| 5 | 3 | | TPM1 | 1 | Chr |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

Analyst 2

| Length (µm) | Width (µm) | Sketch | Verification | # Structures | ID |
|---|---|---|---|---|---|
| | |   | | | |
| 5 | 0.1 | F1 | TPM1 | 1 | Chr |
| 3 | 0.1 | F2 | FP1 | 1 | Chr |
| 2 | 0.1 | F3 | FP2 | 1 | Chr |
| 1 | 0.1 | F4 | FP3 | 1 | Chr |

b

Fig. X2.9 (caption on previous page)

## X3. SUMMARY OF THE PROCEDURE FOR COMPARISON OF TWO TEM ANALYSIS FORMS

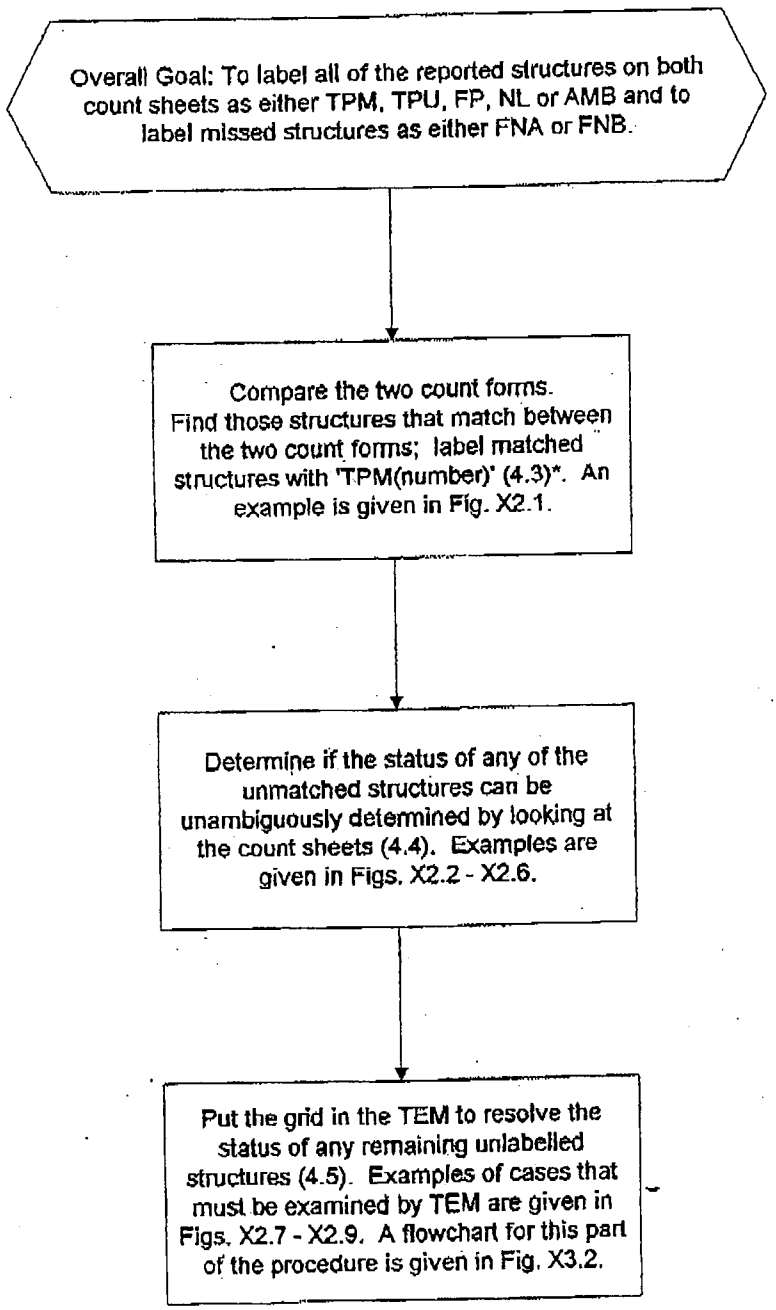



Fig. X3.1 Summary of the overall procedure for comparison of TEM analysis forms by the verifying analyst. *Numbers in parentheses in each block refer to the item number in the procedure.

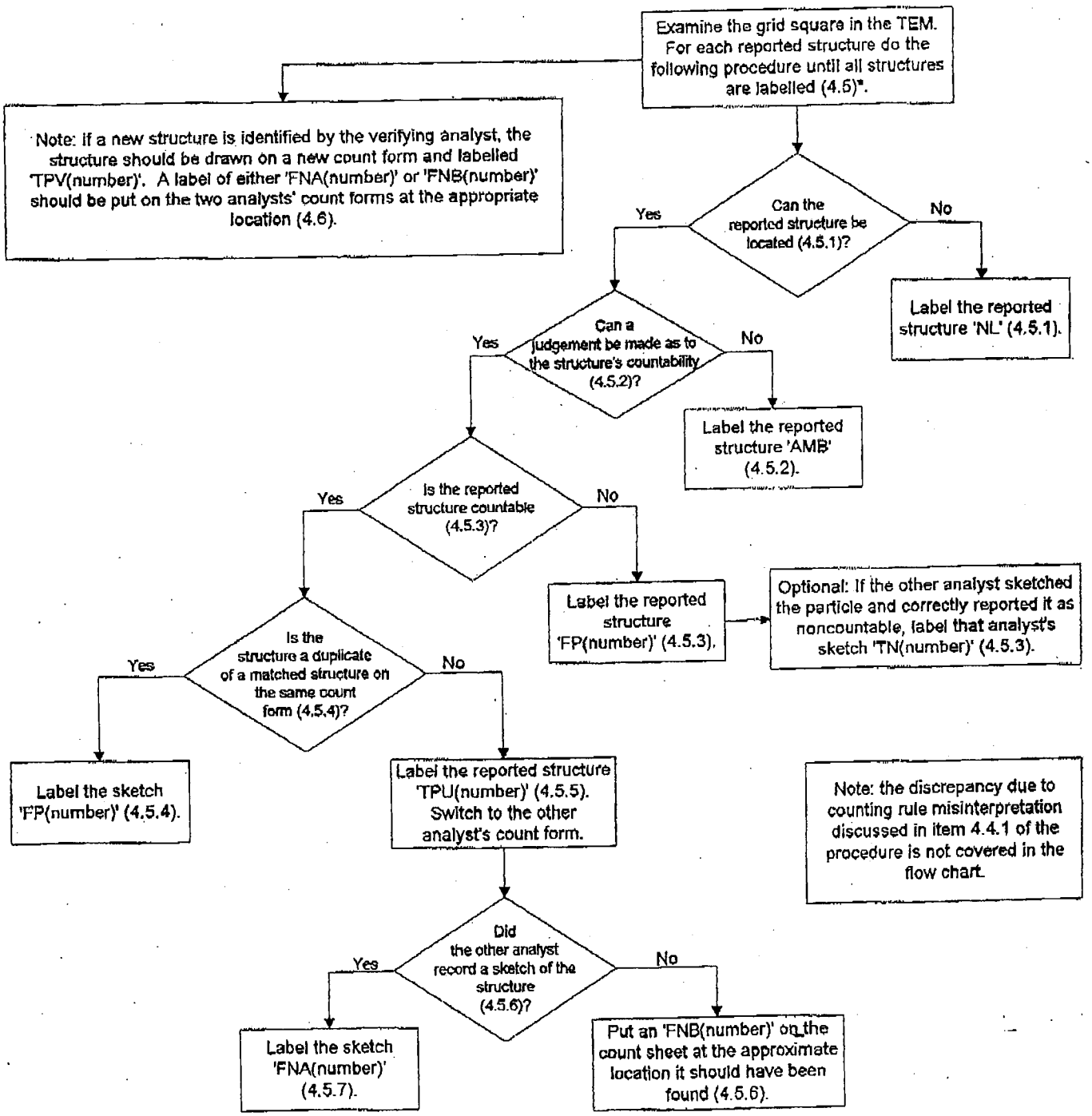



Fig. X3.2 Flowchart for examination of a structure in the TEM. The flowchart is an expansion of the last block in Fig. X3.1. *Numbers in parentheses in each block refer to the item number in the procedure.

# ATTACHMENT 4

## Statistical Comparison of Two Poisson Rates

### 1.0 INTRODUCTION

An important part of the Quality Control plan for this project is the repreparation and reanalysis of a number of TEM grids for quantification of asbestos fiber concentrations in air and dust. Because of random variation, it is not expected that results from repreparations samples should be identical. This attachment presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

### 2.0 STATISTICAL METHOD

This method is taken from "Applied Life Data Analysis" (Nelson 1982). Input values required for the test are as follows:

N1 = Fiber count in first evaluation
S1 = Sensitivity of first evaluation
N2 = Fiber count in second evaluation
S2 = Sensitivity of second evaluation

The test is based on the confidence interval around the ratio of the two observed Poisson rates:

Rate 1 = N1 · S1
Rate 2 = N2 · S2
Ratio = Rate 1 / Rate 2

$$Lower\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1}{N2+1}\right) / F\left[\frac{1+\gamma}{2}; 2 \cdot N2 + 2, 2 \cdot N1\right]$$

$$Upper\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1+1}{N2}\right) \cdot F\left[\frac{1+\gamma}{2}; 2 \cdot N1 + 2, 2 \cdot N2\right]$$

where γ is the confidence interval (e.g., 0.95) and F[δ; df1, df2] is the 100δth percentile of the F distribution with df1 degrees of freedom in the numerator and df2 degrees of freedom in the denominator.

If the lower bound of the ratio is > 1, then it concluded that rate 1 is greater than rate 2 at the 100(1-γ)% significance level. If the upper bound of the ratio is < 1, then it concluded that rate 1 is less than rate 2 at the 100(1-γ)% significance level. Otherwise, it is concluded that rate 1 and rate 2 are not different from each other at the 100(1-γ)% significance level.

<u>Example:</u>

N1 = 4 structures
S1 = 0.0001 $(cc)^{-1}$
Rate 1 = 4 · 0.0001 = 0.0004 s/cc

N2 = 6 structures
S2 = 0.001 $(cc)^{-1}$
Rate 2 = 6 · 0.001 = 0.006 s/cc

γ = 0.95

$$Lower\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4}{6+1}\right) / F\left[\frac{1+0.95}{2}; 2\cdot 6+2, 2\cdot 4\right] = 0.014$$

$$Upper\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4+1}{6}\right) \cdot F\left[\frac{1+0.95}{2}; 2\cdot 4+2, 2\cdot 6\right] = 0.281$$

In this example, because the upper bound of the ratio is < 1, it is concluded that Rate 1 (0.0004 s/cc) is less than Rate 2 (0.006 s/cc) at the 95% significance level.

## 3.0 REFERENCES

Nelson W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

# ATTACHMENT 5

## NVLAP Airborne Asbestos Proficiency Test 98-2: Grid Orientation

## *Instructions for Form 1*

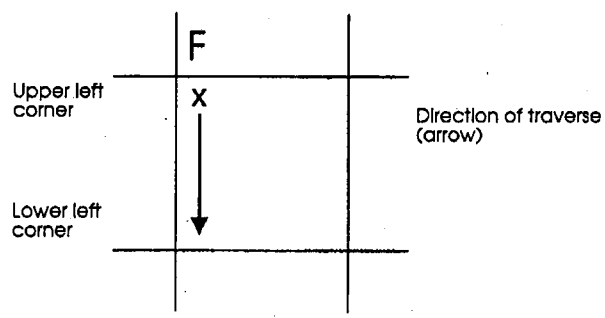
The following procedure is designed to ensure that all laboratories count the grid squares in the same orientation and scan direction to allow for verified analyses which will be performed in the next round of proficiency testing.

1. Put a grid into the TEM. Find a particle at the magnification typically used for asbestos analysis. Move the particle using one stage translation and record the direction of movement of the particle on *Form 1*. Move the particle using the other stage translation knob and record the direction of movement. Recording the two directions of movement should roughly form a cross. The cross represents the translation directions of your microscope at the magnification used for asbestos analysis. ***Draw the letter "F" onto the cross so the sides of the letter are parallel to the translation directions and the letter is upright and is not inverted.*** See the example on *Form 1*.

2. Decrease the magnification and locate the letter "F" on the finder grid. Increase the magnification of the TEM to that typically used for asbestos analysis by your lab, keeping the letter "F" in the field of view. Compare the orientation of the "F" to the cross drawn in step 1. If the letter "F" is not oriented as shown in your sketch, remove the specimen holder and rotate or invert the grid as necessary to correctly align the grid. This may require several iterations.

3. When the correct orientation is found, record the grid's position in the specimen holder as shown in the example of the second part of *Form 1*. Indicate in your drawing where the straight side and the notched portion of the grid are located. All grids analyzed in this proficiency test should be oriented in the same manner (always check that the letter "F" is in the correct orientation and that the X-Y translation directions allow translation roughly parallel to the grid bars).

4. The starting point of the traverse for structure counting must correspond to the upper left corner on the grid square. The "X" marks the starting corner of the traverse (your grid square may be at an angle to that shown in the example):



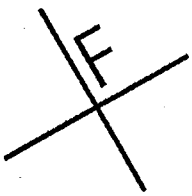

The initial direction of traverse must be from the upper left corner to the lower left corner of the grid square. If correctly oriented, the edge of the grid bar will remain in the field of view during the entire initial traverse (some allowance must be made for curvature or irregularly shaped grid bars.) If the grid is not oriented properly, go back to step 2.

NVLAP Lab Code: _____

**Form 1.** ***Grid Orientation***

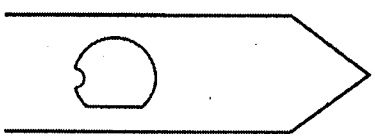
1. Sketch the orientation of the X-Y translation directions of the electron microscope as projected onto the electron microscope stage. Record the letter "F" as shown in the example below:

EXAMPLE:




2. Sketch below the orientation of the grid relative to the sample holder as shown in the example below:

EXAMPLE:



# ATTACHMENT 6

## Grid Opening Template for Sketching the Relative Position of Observed Structures

# STRUCTURE LOCATIONS WITHIN GRID OPENING

****NOTE: Sketches only need to be completed for interlab analyses and repreps associated with interlabs*

Lab Name: _____ Lab Job Number: _____

Index ID: _____ Lab Sample ID: _____

Lab QC Type (circle one): Reprep for interlab Interlab

Grid: _____ Grid Opening: _____

upper left corner

traverse direction

Comments:

# Request for Modification
to
## Laboratory Activities
**LB-000066E**

***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 D5755 | TEM 100.2 Mod 20 | SRC-LIBBY-03 |
| SRC-LIBBY-01 | NIOSH 9002 | Other:_____ | |

Revised by: Douglas Kent Title: Senior Asbestos Microscopist

Company: TechLaw/ESAT Region 8 Date: August 15, 2013

Original Requester: [LB-000066C]: Bill Brattin, SRC Original Request Date: September 11, 2007

Original Requester: [LB-000066D]: Douglas Kent, ESAT Original Request Date: July 2, 2010

Description of Modification:

This permanent modification applies to all investigative samples analyzed by TEM (regardless of the specific TEM method or sample matrix) for the Libby Asbestos Site, unless otherwise indicated by the relevant SAP Analytical Summary Sheet. Based on this modification, all analytical laboratories shall: 1) Indicate on the count sheet presence or absence of sodium and potassium in all recorded structures (except chrysotile); 2) Record on the count sheet "close-call" NAM particles (defined in Attachment 1); 3) Record NR particles (defined in Attachment 1); 4) Record the probable mineral species (if known) of each recorded structure; 5) Record EDS (energy dispersive spectrometry) spectra of LA, NR, and close-call NAM particles (at a frequency described in Attachment 1); and 6) Record one photomicrograph of a SAED (selected area electron diffraction) pattern from an LA structure for each sample in which LA is found. Photographs of structure morphology are not required for Libby Project samples unless specifically indicated by the relevant SAP Analytical Summary Sheet.

Reason for Modification:

Studies of asbestos from the mine near Libby indicate that the asbestos spans several mineral species, including winchite and richterite (these are the primary forms) as well as tremolite and possibly actinolite (these are minor forms) (Meeker et al, 2003). Consequently, all analytical laboratories supporting the Libby Project are currently directed to classify as "LA" any particle that: 1) Meets the minimum length, width, and aspect ratio requirements as defined in the relevant SAP (e.g. length at least 0.5 microns and aspect ratio at least 3:1); 2) Has an SAED pattern consistent with amphibole; and 3) has an EDS spectrum consistent with the range of asbestos mineral forms observed at the mine (EPA, 2005). However, a project that included collection of air samples from locations outside of Libby highlighted a potential limitation of this approach. That is, tremolite and actinolite are part of the suite of minerals that compose LA and are found at Libby, but these types of fibers may also occur as results of releases from sources that are not related to the mine at Libby (e.g. commercial products or natural sources). Also, some other minerals (e.g. pyroxenes) are sometimes difficult to distinguish from tremolite and actinolite (Bern et al, 2002). Because mineralogical data may or may not increase our understanding of the toxicity of LA, delineating amongst these mineral types is desirable at this stage of data collection. Therefore, the primary objective of this modification is to collect more detailed data on the chemical composition of amphibole fibers encountered at the Libby Site.

Potential Implications of this Modification:

This modification clarifies requirements for recording representative EDS spectra and SAED patterns and defines valid values for Mineral Class, Mineral Description, and structure NaK codes. There are no significant changes from LB-000066C or LB-000066D and these requirements will not increase analytical time/cost or affect data quality. Hence, there are no negative implications of this modification.

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

This laboratory modification is (circle one): **APPENDS to** _____ **SUPERSEDES** LB-000066C, LB-000066D

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: August 15, 2013

*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 and attachments.

**REFERENCES**


Bern A, Meeker G, and Brownfield I, 2002. Guide to Analysis of Soil Samples from Libby, Montana for Asbestos Content by Scanning Electron Microscopy and Energy Dispersive Spectrometry. USGS Administrative Report.

Meeker G, Bern A, Brownfield I, Lowers H, Sutley S, Hoeffen T, and Vance J, 2003. The Composition and Mineralogy of Amphiboles from the Rainy Creek Complex, Near Libby, Montana. American Mineralogist 88: 1955-1969.

Gunter M, Dyar M, Twamley B, Foit F, Cornelius S, 2003. Composition, $Fe^{3+}/\Sigma Fe$, and Crystal Structure of Non-Asbestiform and Asbestiform Amphiboles From Libby, Montana, USA. American Mineralogist 88: 1970-1978.

USEPA, 2005. EDS Spectra Characteristics Study for Libby-Type Amphiboles. Report prepared by Syracuse Research Corporation, Denver, CO for USEPA Region 8, Denver, CO.

Wylie A and Verkouteren J, 2000. Amphibole Asbestos from Libby, Montana: Aspects of Nomenclature. American Mineralogist 85: 1540-42.

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: Robyn L. Denton Date: 28 August 2013
*(Laboratory Manager or designate)*

Project Review and Approval: Dania Zimmer Date: 8/28/13
*(USEPA: Project Manager or designate)*

Approved By: David L. Berry Date: 8/28/2013
*(USEPA: Technical Assistance Unit Chief or designate)*

**ATTACHMENT 1**
**Additional Structure Recording Rules for Libby TEM Investigative Analyses**

1) Continue to classify structures as LA, OA, CH, or NAM in accord with current procedures.
2) For all NAM structures that were "close calls" (i.e., they required careful assessment to determine they were not LA), record the NAM structure on the count sheet and in the EDD. Be sure to place zero(s) in the structure count field(s) so the particle is not included in the asbestos count. NAM particles such as vermiculite, biotite, gypsum, rutile ($TiO_2$), and other minerals that are clearly not amphiboles should not be recorded.
3) For all LA, OA, and NAM (but not chrysotile) structures recorded, indicate the presence or absence of sodium and potassium by recording one of the following codes in the EDXA Observation field on the count sheet and in the EDD:

| Code | Meaning |
|---|---|
| NaK | Both Na and K are clearly present |
| NaX | Only Na is clearly present |
| XK | Only K is clearly present |
| XX | Neither Na nor K are clearly present |

4) For all structures recorded, indicate the structure's probable mineral identification by recording one of the following codes for Mineral Class and Mineral Description on the count sheet and in the EDD:

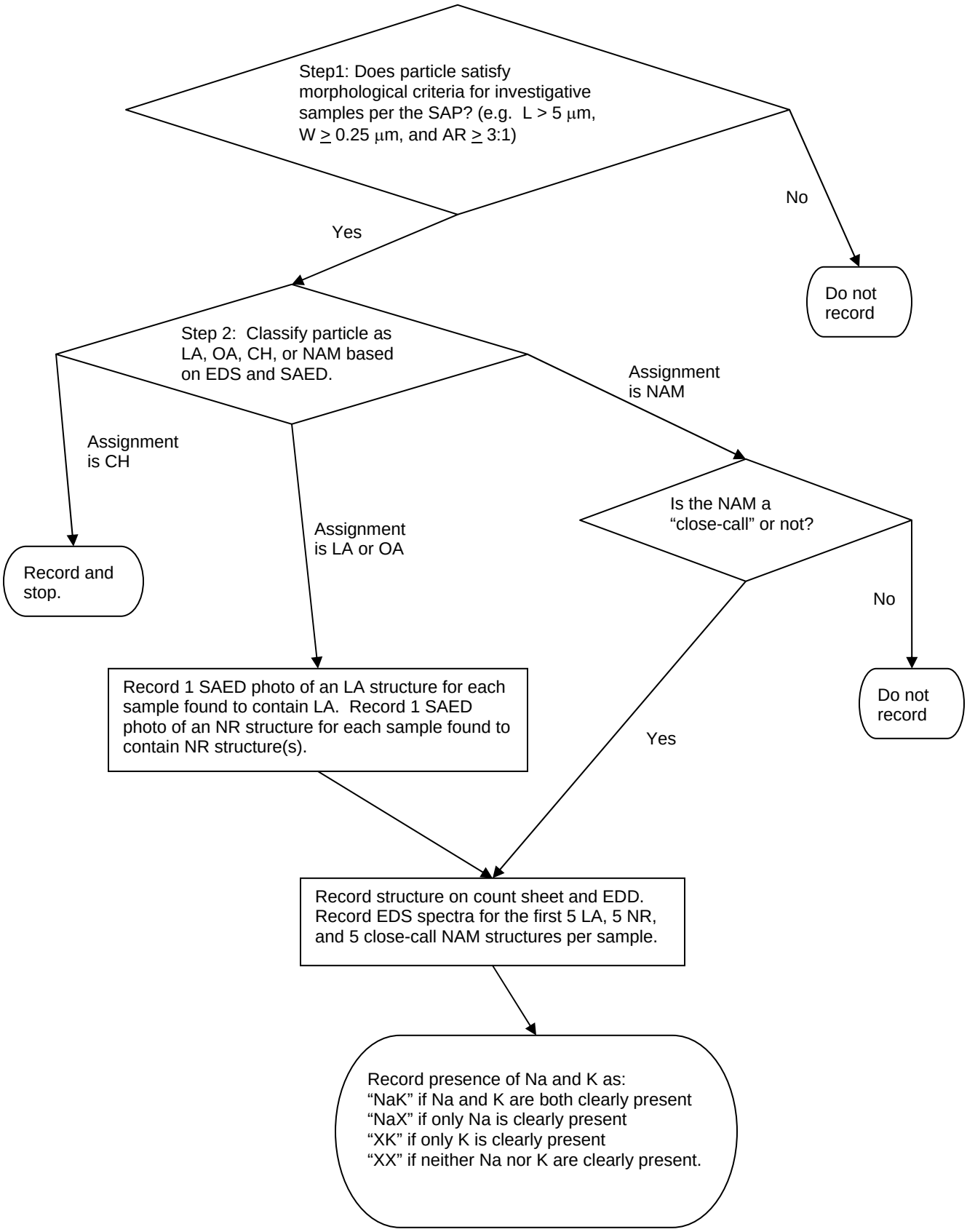
| Mineral Class | Mineral Description | Meaning |
|---|---|---|
| LA | WRTA | Winchite/Richterite/Tremolite/Actinolite |
| | AC | Actinolite |
| | TR | Tremolite |
| | AT | Actinolite/Tremolite (too close to call) |
| OA | AM | Amosite |
| | AN | Anthophyllite |
| | CR | Crocidolite |
| | NR | Non-LA and Non-Regulated Amphibole |
| CH | CH | Chrysotile |
| NAM | PY | Pyroxene |
| | OT | Other |
| | UN | Unknown |

5) Structures identified as WRTA, AC, TR, or AT are LA structures. To be classified as WRTA, a structure must display an amphibole SAED pattern and have an EDS spectrum consistent with the range of LA compositions from the mine as described in the available literature (see references) and the lab's experience with the Libby Starting Material (a.k.a. the Dirty 30). All labs should have prepared grids of the Dirty 30 and example spectra showing the range of compositions typical of the Dirty 30 for each EDS detector type (beryllium or thin window) used at the lab. It is expected that most AC, TR, and AT particles will be "XX" for sodium and potassium content.
6) Structures identified as AM, AN, CR, or NR are OA structures. NR structures are rarely found in Libby Project samples and are suspected amphiboles that do not have a composition that is consistent with either Libby Amphibole (as described in the available literature, see references) or hornblende. They often have a very high aspect ratio and a composition consistent with magnesio-riebeckite or magnesio-arfvedsonite. An example EDS spectrum from a NR structure is provided in Attachment 2.
7) A NAM should be indicated as either PY, OT, or UN.
8) Although any additional comments in the structure comment field are welcome, do not use any codes for mineral description that are not in the list.
9) For each LA, NR, and "close call" NAM structure, record (either electronically or as a hardcopy print-out) the structure's EDS spectrum, up to a maximum of 5 LA, 5 NR, and 5 close-call NAM's per sample. To the extent practical, collect the EDS spectra for a sufficient length of time so that key peaks (e.g. sodium,

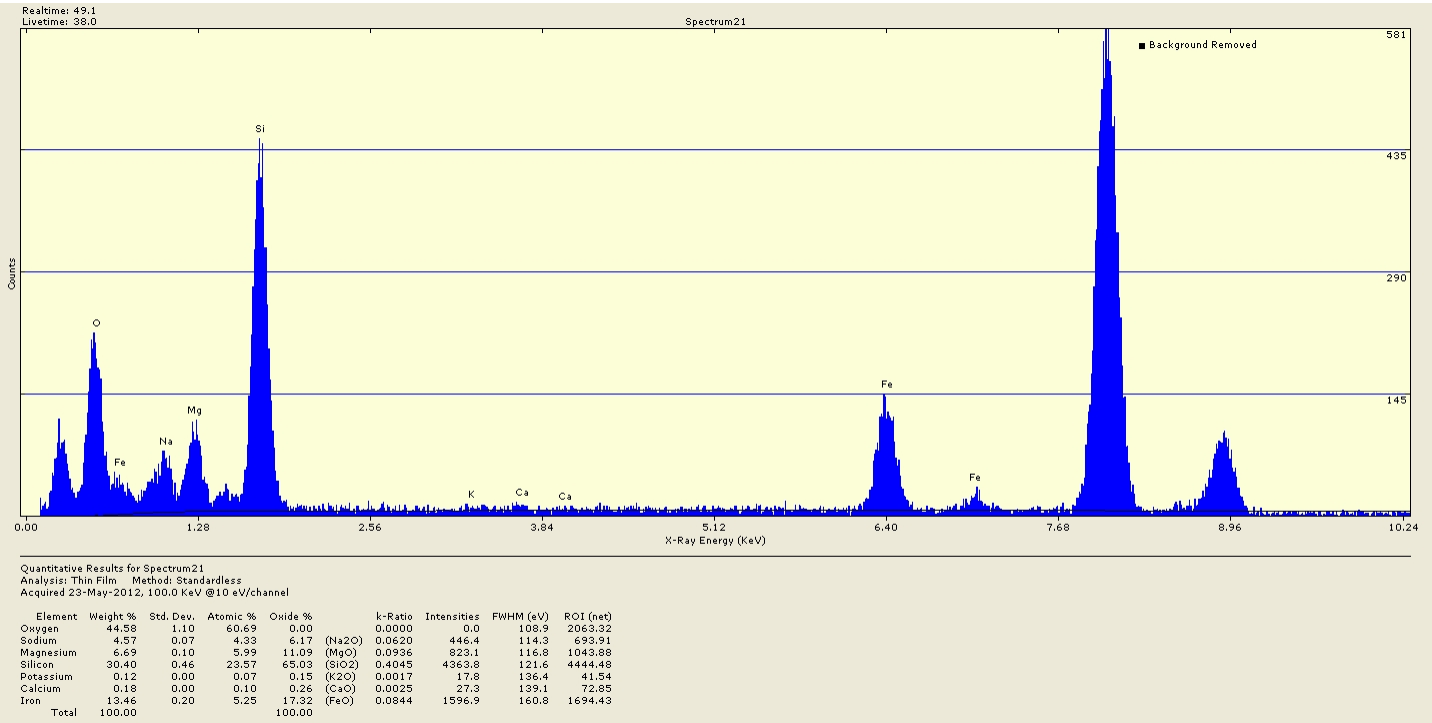
potassium, and aluminum) can be distinguished from the background. The silicon peak should contain 1,000 or more counts whenever possible.

10) Record (either on film or digitally) one SAED pattern from an LA structure for each sample found to contain LA. If more than one LA structure is encountered in a sample, try to find a quality zone axis pattern to serve as the diffraction pattern that is photographed.
11) Record (either on film or digitally) one SAED pattern from an NR structure for each sample found to contain one or more NR structures.
12) Be sure that each EDS spectrum and SAED photo recorded can be linked to a specific structure on the count sheets (e.g., by indicating the name of the photo or spectrum in the structure comment field on the count sheet).
13) SAED photos do not need to be recorded for NAM structures.
14) SAED photos and/or EDS spectra do not need to be recorded for AM, AN, CR, or CH structures unless specifically required by a laboratory's internal standard operating procedures.
15) The EDS spectra described above should be included (e.g., as scans provided in a .pdf) in the data package for a sample delivery group. Because SAED photos do not have meaning unless they have been measured (which is outside the scope of routine analysis), and because of the amount of time required to unload and develop photographic film, SAED photos do not need to be provided in data packages but should be kept on file at the laboratory and made available upon request. The laboratory should measure a representative fraction of the SAED photos taken as required by their internal QA procedures. Indexing of a SAED photo is preferred when possible but can only be performed when a quality zone axis pattern has been achieved. If a SAED photo selected for measurement is not indexed, the row spacing (expected to be approximately 0.53 nm for amphibole) should be measured.
16) If morphology photos are required by the SAP, they should be included in the data package and be taken of the first 5 LA structures (but not NAM's) found in a sample, unless otherwise indicated by the SAP.
17) Figure 1 provides a flow chart that summarizes the process implemented by this modification.

**FIGURE 1**
**Flow Chart Summarizing This Laboratory Modification**

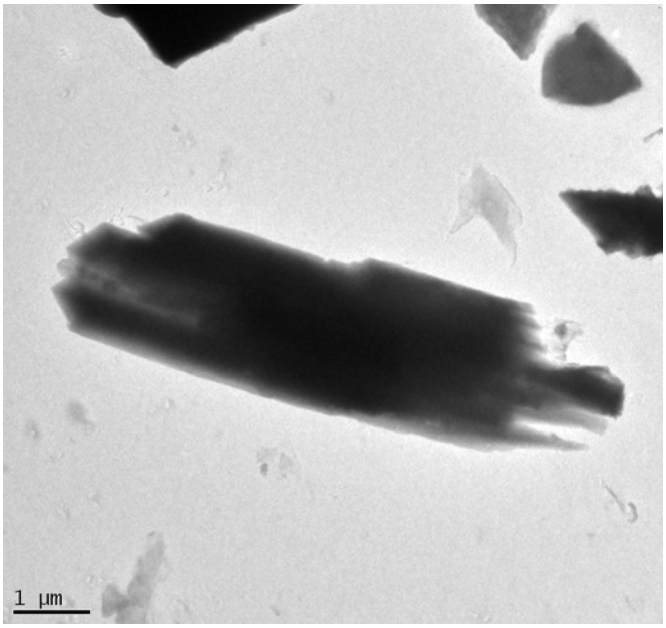


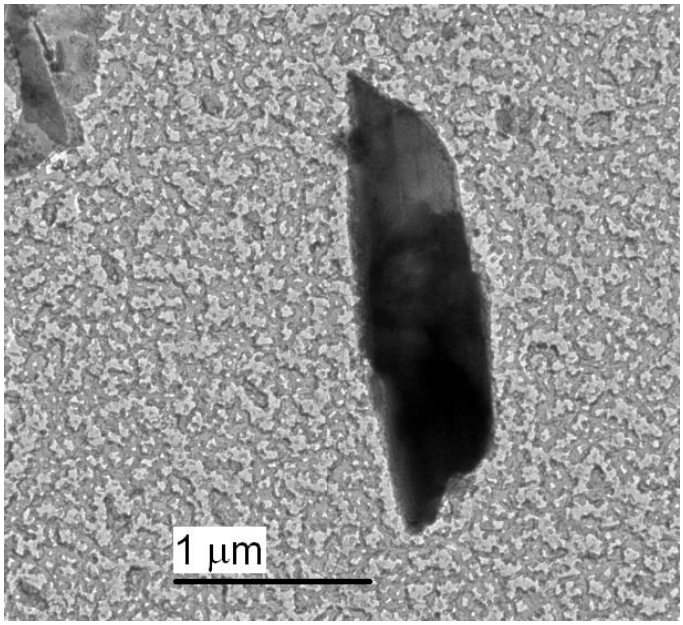
**ATTACHMENT 2**
**Example EDS Spectra and Photos**




| Element | Weight % | Std. Dev. | Atomic % | Oxide % | | k-Ratio | Intensities | FWHM (eV) | ROI (net) |
|---|---|---|---|---|---|---|---|---|---|
| Oxygen | 44.58 | 1.10 | 60.69 | 0.00 | | 0.0000 | 0.0 | 108.9 | 2063.32 |
| Sodium | 4.57 | 0.07 | 4.33 | 6.17 | (Na2O) | 0.0620 | 446.4 | 114.3 | 693.91 |
| Magnesium | 6.69 | 0.10 | 5.99 | 11.09 | (MgO) | 0.0936 | 823.1 | 116.8 | 1043.88 |
| Silicon | 30.40 | 0.46 | 23.57 | 65.03 | (SiO2) | 0.4045 | 4363.8 | 121.6 | 4444.48 |
| Potassium | 0.12 | 0.00 | 0.07 | 0.15 | (K2O) | 0.0017 | 17.8 | 136.4 | 41.54 |
| Calcium | 0.18 | 0.00 | 0.10 | 0.26 | (CaO) | 0.0025 | 27.3 | 139.1 | 72.85 |
| Iron | 13.46 | 0.20 | 5.25 | 17.32 | (FeO) | 0.0844 | 1596.9 | 160.8 | 1694.43 |
| Total | 100.00 | | | 100.00 | | | | | |

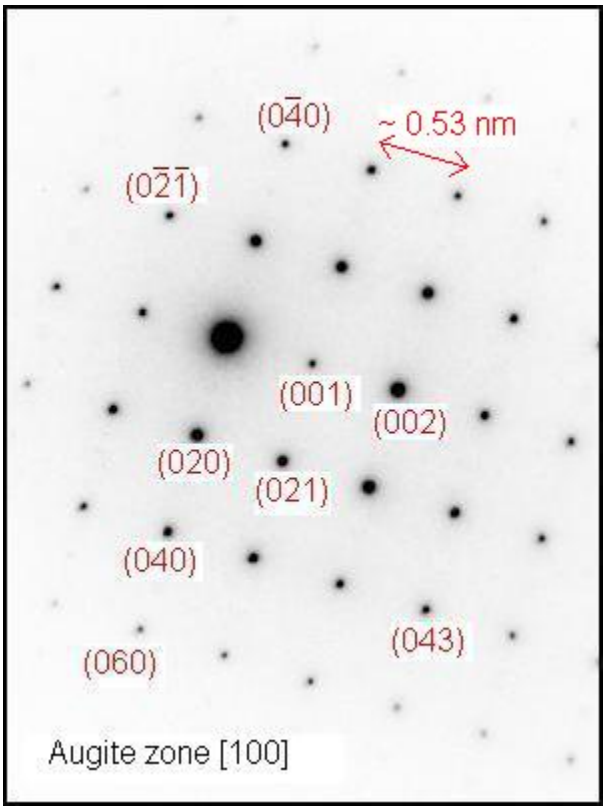
EDS spectrum of an OA, NR structure with a composition consistent with magnesio-riebeckite. Spectrum courtesy of EMSL Analytical, Inc.

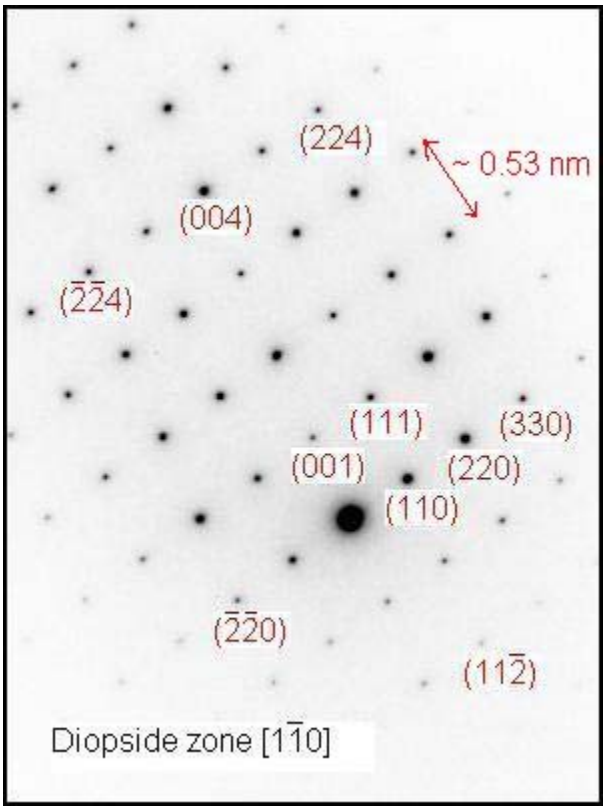


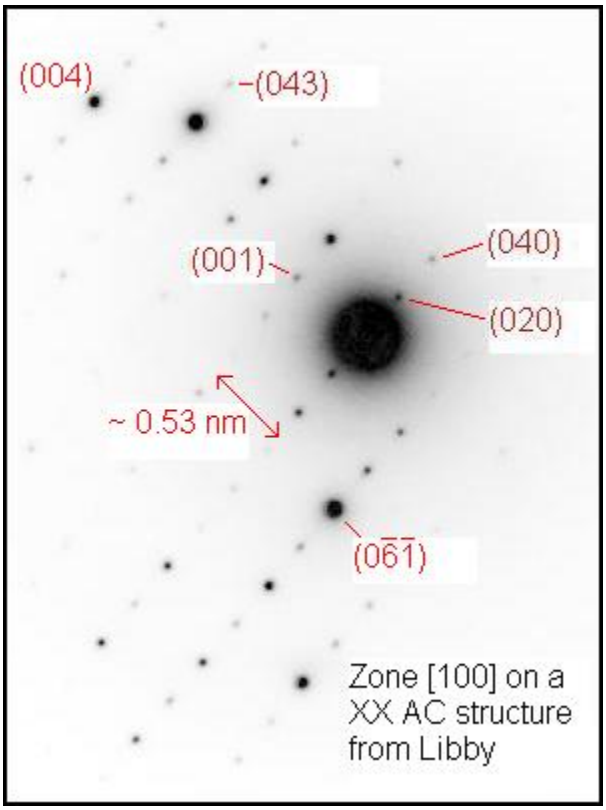

Pyroxenes (more specifically, calcic clinopyroxene, consisting of diopside $CaMgSi_2O_6$, hedenbergite $CaFeSi_2O_6$, and augite $(Ca,Na)(Mg,Fe,Al)(Si,Al)_2O_6$) occur naturally in the Libby Valley and may be confused with tremolite-actinolite. As a general rule pyroxenes are less fibrous than amphiboles. Here are photos of two pyroxene structures found in authentic field samples from Libby and Troy. Pyroxenes also commonly form as single crystals, as shown in the photo on the right. Photos courtesy of ESAT Region 8.

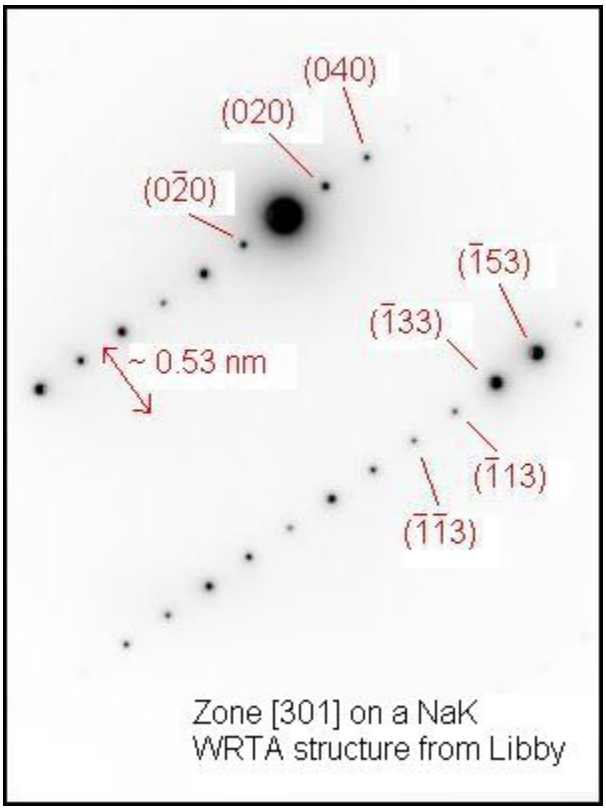
Like amphiboles, pyroxenes show the prominent row spacing of approximately 0.53 nm where the rows are oriented perpendicularly to the c-axis, which is the long axis on the fiber. Examples SAED patterns are provided below.

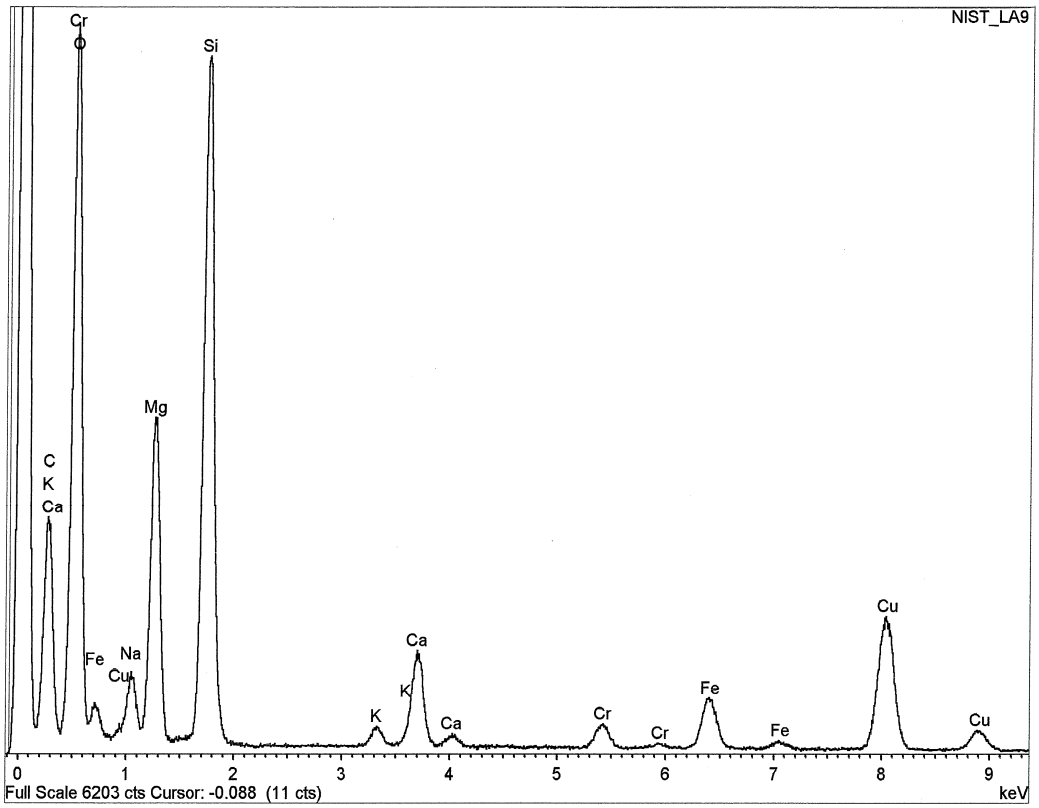


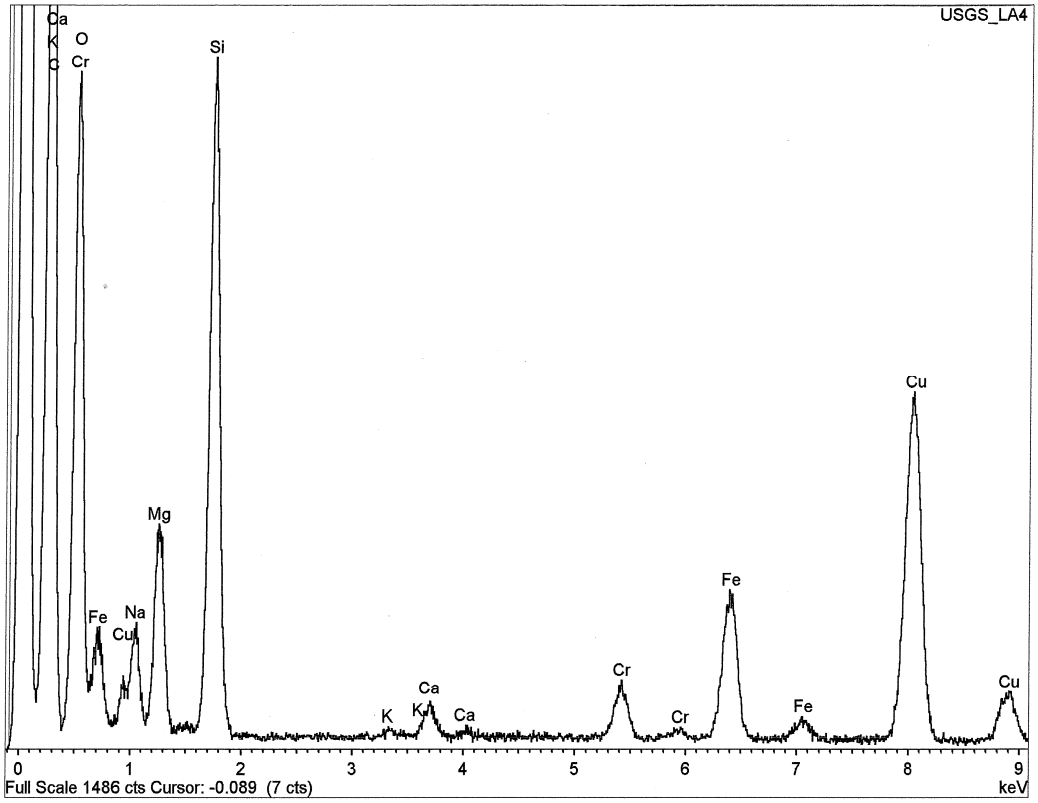

Note that the h and k Miller indices of all spots observed in pyroxene and amphibole SAED patterns must sum to even numbers for crystal symmetry reasons. The b axial length of amphiboles is approximately twice that of pyroxenes. Hence, pyroxenes cannot show the very closely spaced spots along the rows as seen in amphibole SAED patterns that show the (020) spacing as shown in the examples below.



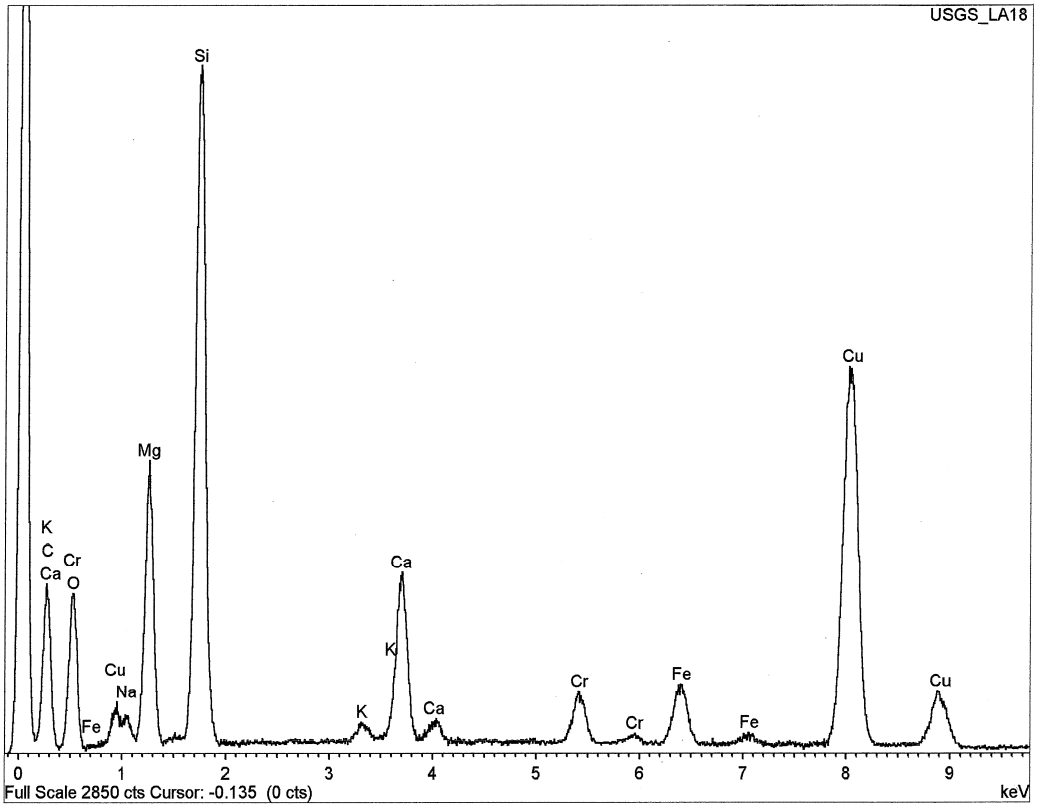





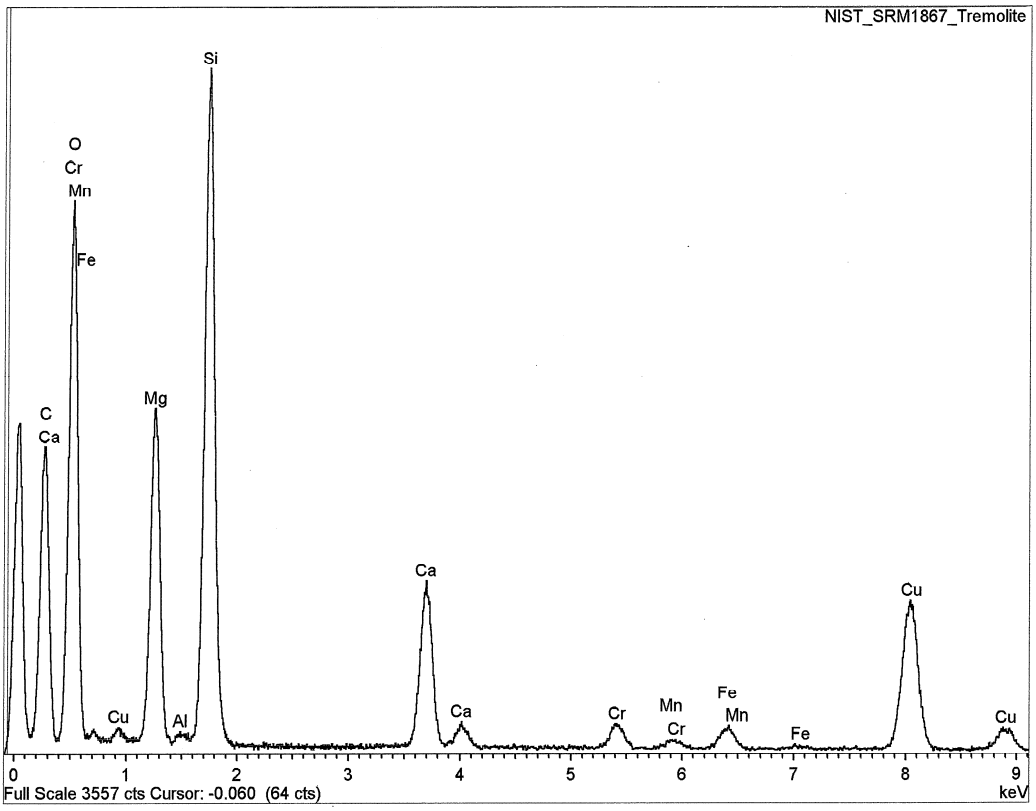

A typical LA structure from the mine (from sample #4 of NIST PLM PE Proficiency Testing Round M12001) classified as a NaK WRTA structure. The chrome in these spectra is an artifact from the specimen holder. Spectra courtesy of ESAT Region 8.



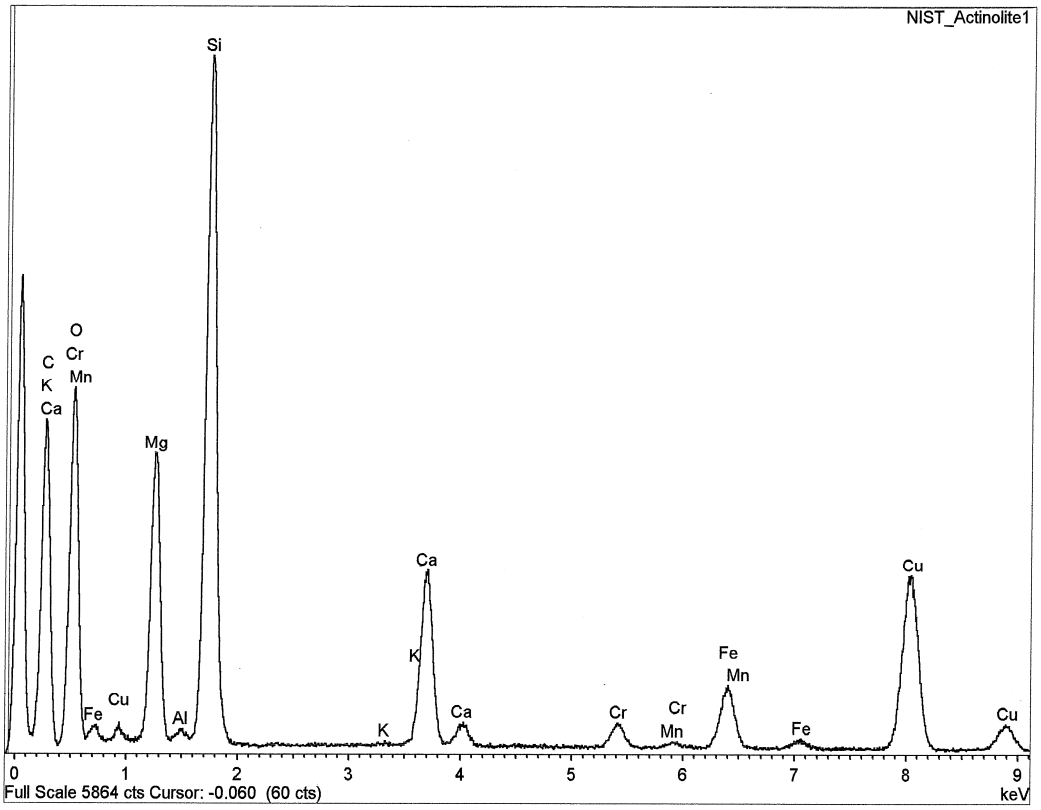

An LA structure from the mine with low Ca and high Na and Fe. This is a NaK WRTA structure from the Dirty 30.



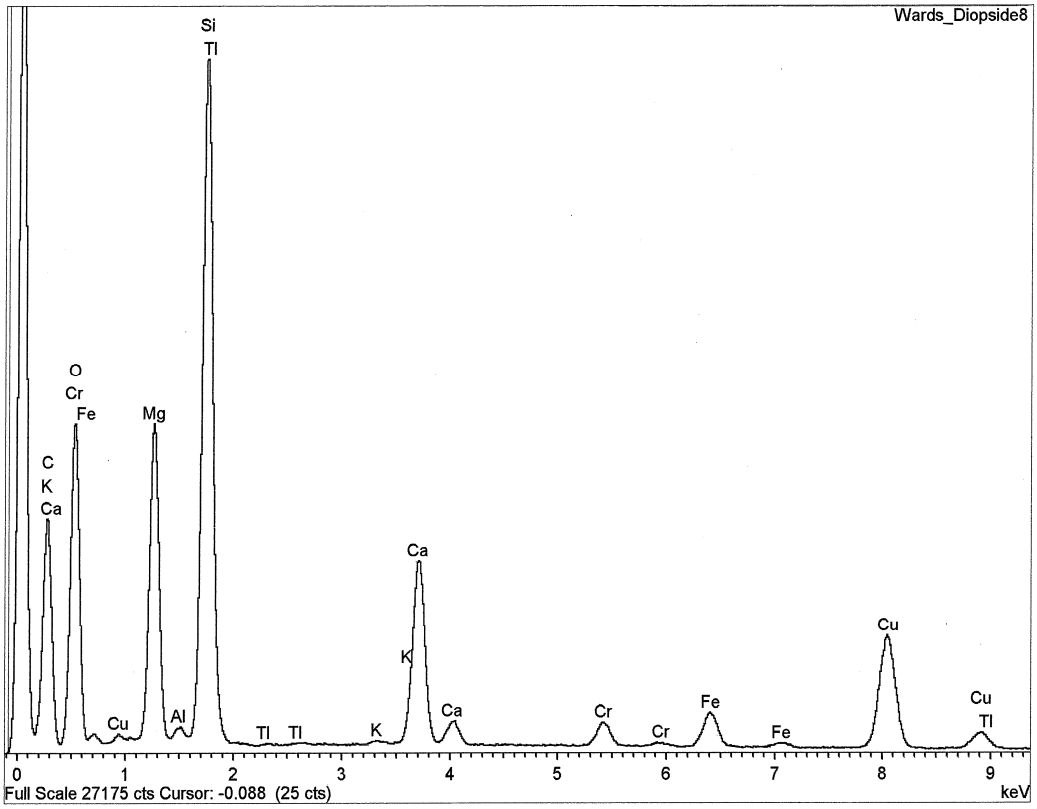

An LA structure from the mine with high Ca and low Na. This is a NaK WRTA structure from the Dirty 30. Quantitative EDS indicates that the calcium content is 2.02 apfu (atoms per formula unit) on the basis of 23 oxygens.




NIST Standard Reference Material 1867a tremolite. Quantitative EDS indicates that the calcium content is 2.01 apfu on the basis of 23 oxygens.




NIST Standard Reference Material 1867a actinolite. Quantitative EDS indicates that the calcium content is 2.06 apfu on the basis of 23 oxygens.




A pyroxene from Herschel, Ontario identified as diopside according to the supplier who provided the mineral specimen. Although the height of the calcium peak looks similar to that of the SRM 1867a actinolite, quantitative analysis of this spectrum indicates that its calcium content is 2.26 apfu on the basis of 23 oxygens. That is permissible for pyroxenes but is a stoichiometric impossibility for an amphibole. Ca cannot exceed 2 apfu (plus or minus a few percent) for any amphibole.

# Libby Asbestos Project
# Project-specific Standard Operating Procedure
# Semi-Quantitative Visual Estimation of Vermiculite in Soils during Removal Activities

SOP No.: CDM-LIBBY-16

## 1.0 Purpose

During response actions at contaminated properties, it is EPA's goal to perform as thorough, consistent, and complete a clean up as possible within the criteria established in the governing documents for the Libby Superfund Site. The purpose of this standard operating procedure (SOP) is to provide guidance to project personnel to properly inspect and identify vermiculite on a property in areas where removal activities (i.e., excavation) are not planned.

## 2.0 Definitions

The following land use designations are used for determining inspection protocol during the investigation phase (e.g., General Property Investigation [GPI]) and will be referenced when conducting the supplemental vermiculite inspections during removal actions.

Specific Use Area (SUA) – Discrete exterior parcels on a property with a designated specific use. Due to the nature of activities typically carried out in SUAs, residents may be especially vulnerable to exposures when Libby amphibole asbestos (LA) contaminated soil becomes airborne. SUAs may be bare or covered with varying amounts of vegetation. SUAs include but are not limited to areas such as:

- Flower Pots
- Flowerbeds
- Gardens
- Stockpiles
- Play Areas
- Dog Pens
- Horse Corrals
- Driveways (non-paved)
- Parking Lots (non-paved)
- Roads (non-paved)
- Alleys (non-paved)

Common Use Area (CUA) – Exterior parcels on a property with varied or generic use. CUAs may be bare or covered with varying amounts of vegetation. CUAs include:

- Walkways
- Yards (front, back, side, etc.)
- Former Gardens
- Former Flowerbeds

Limited Use Area (LUA) – Exterior parcels on a property that are accessed, utilized, and maintained on a very limited basis. LUAs may be bare or covered with varying amounts of vegetation. LUAs include:

- Pastures
- Maintained/Mowed Fields
- Underneath porches/decks[1]
- Overgrown Areas (with trails/footpaths, or between SUAs/CUAs)

Non-Use Area (NUA) – Exterior parcels on a property with no current use (e.g., areas that are not maintained or accessed). NUAs may be bare or covered with varying amounts of vegetation. NUAs include:

- Wooded Lots
- Un-maintained Fields

Point Inspection – Used in SUAs and CUAs. A point inspection (PI) is an intrusive visual inspection of the top portions of the soil at a randomly selected point within a zone. A PI consists of the active displacement of the surface material (e.g., soil, gravel, etc.) with a small shovel or trowel and visual inspection of the displaced material to determine if vermiculite is present. If vermiculite is observed during the PI, the location and a semi-quantitative estimate of vermiculite contamination will be recorded as directed in the governing document.

Zone – A discrete area on the property delineated by the field staff where PIs will occur. The governing document will describe the location and size of each zone based on the investigation data quality objectives. It should be noted that inspection zones will be established based on site features and removal design (i.e., excavation lines) and may not result in "perfect squares." No area type may be combined with any other area type. For example, driveways and flowerbeds are both SUAs but will be separated into unique zones for visual inspection. Similarly, large CUAs such as yards may be subdivided into multiple zones dependent on site conditions. Sectioning properties into

[1] The soils underneath porches and decks will be classified as LUAs depending on ground clearance and accessibility to homeowners and pets. If these areas are not accessible, they will be classified as NUAs.

additional zones will be at the discretion of the field teams but consistent among the teams.

# 3.0 Applicability

This SOP applies to properties within the Libby Superfund Site at varying stages of the removal process. The vermiculite inspection should be performed as early as possible in the removal process to facilitate removal activity planning but will occur before excavation crews leave the area of interest on a property (e.g., back yard, etc.). That is, vermiculite inspections should be performed to coincide with the removal contractor's excavation plan and conducted in areas where the contractor will excavate first and advance to subsequent areas. Timely inspections are imperative so as to minimize moving removal equipment (e.g., excavators, skid-steers, etc.) across excavated areas.

# 4.0 Procedure

The following sections outline the general process of performing the vermiculite inspections:

- Identify areas of interest
- Establish zones
- Perform PI

## 4.1 Identify Areas of Interest

Upon arrival at the property, the field staff will locate all areas requiring the visual inspection as required by the governing document. All property use areas should already be categorized (e.g., SUA, CUA, etc.) during previous investigations (e.g., GPI). If not, categorize land use areas by the definitions provided in Section 2.0. If at any time there are disagreements in identification of inspection areas, the team leaders and/or appropriate government representatives will be consulted.

## 4.2 Establish Zones

Zones will be established throughout the property within the areas of interest. Inspection zones will only be established in CUAs and SUAs as these are the areas where additional excavation during removal activities will occur. Zones will be used to methodically inspect areas of interest as defined in the governing document. Since the data quality objectives of specific investigations vary, zone inspection area requirements will be discussed in the governing document. The field team will use appropriate measuring tools (e.g., measuring wheel, design drawings, etc.) to establish zone boundaries.

## 4.3 Perform Point Inspection

Once zones are established, PIs will commence. The number of PIs to be performed in each zone will be detailed in the guidance document. The location of the PIs will be at

the discretion of the field team but will be randomly selected within the zone. The following process outlines the general sequence of performing a PI:

- Select an area within the zone.
- Visually inspect the PI location using a spade or trowel, carefully removing excess debris (e.g., organic material, grass, etc.) prior to extraction. Extract an amount of material (i.e., soil, crushed rock, etc.) from the depths as outlined below:
    - CUAs – 0 to 3 inches below ground surface (BGS) or to refusal
    - SUAs – 0 to 6 inches BGS or to refusal
- Visually inspect material and record semi-quantitative estimation of vermiculite as described below.
- Replace cover material.
- Repeat as necessary employing procedure outlined in the governing document.

During PIs, field staff will estimate the quantity of vermiculite observed. Each PI will be assigned a semi-quantitative estimate of vermiculite content using a 4-point scale: none (N), low (L), intermediate (M), and high (H). For PI locations where vermiculite is observed, estimates (e.g., L, M, or H) will be recorded as described in the governing document.

# 5.0 Health & Safety/Engineering Controls

All personnel will carry out visual inspections in accord with proper personal protective equipment (PPE) and other requirements as outlined in the governing document. If necessary, the field team should use appropriate engineering controls, such as wetting methods, during the PIs to minimize dust generation.

# 6.0 Equipment Decontamination

Equipment decontamination is not required between PIs as sample collection is not included as part of this SOP. However, field staff should inspect field tools (e.g., shovel, trowel, etc.) between each PI to ensure remnant vermiculite flakes are not remaining. Equipment should be thoroughly decontaminated, using methods outlined in the governing document, before leaving the site.

# 7.0 Documentation

Appropriate documentation will be recorded for findings of the vermiculite inspection per project requirements. This may include logbooks, QARs, drawing edits, etc. The governing document will describe applicable documentation procedures required for inspection activities outlined in this SOP.

# 8.0 Training

Every effort will be made to ensure consistency in the semi-quantitative evaluation of vermiculite in soil to the extent possible. This will include training (e.g., field calibration), use of specimen examples (i.e., jars/photographs of low, intermediate, and

high quantities of vermiculite, etc.), designated field staff, and oversight by project management staff. Figures illustrating none, low, intermediate, and high quantities of vermiculite are attached to this SOP for reference (Attachment 1).

# Attachment 1
# Vermiculite Inspection Standards

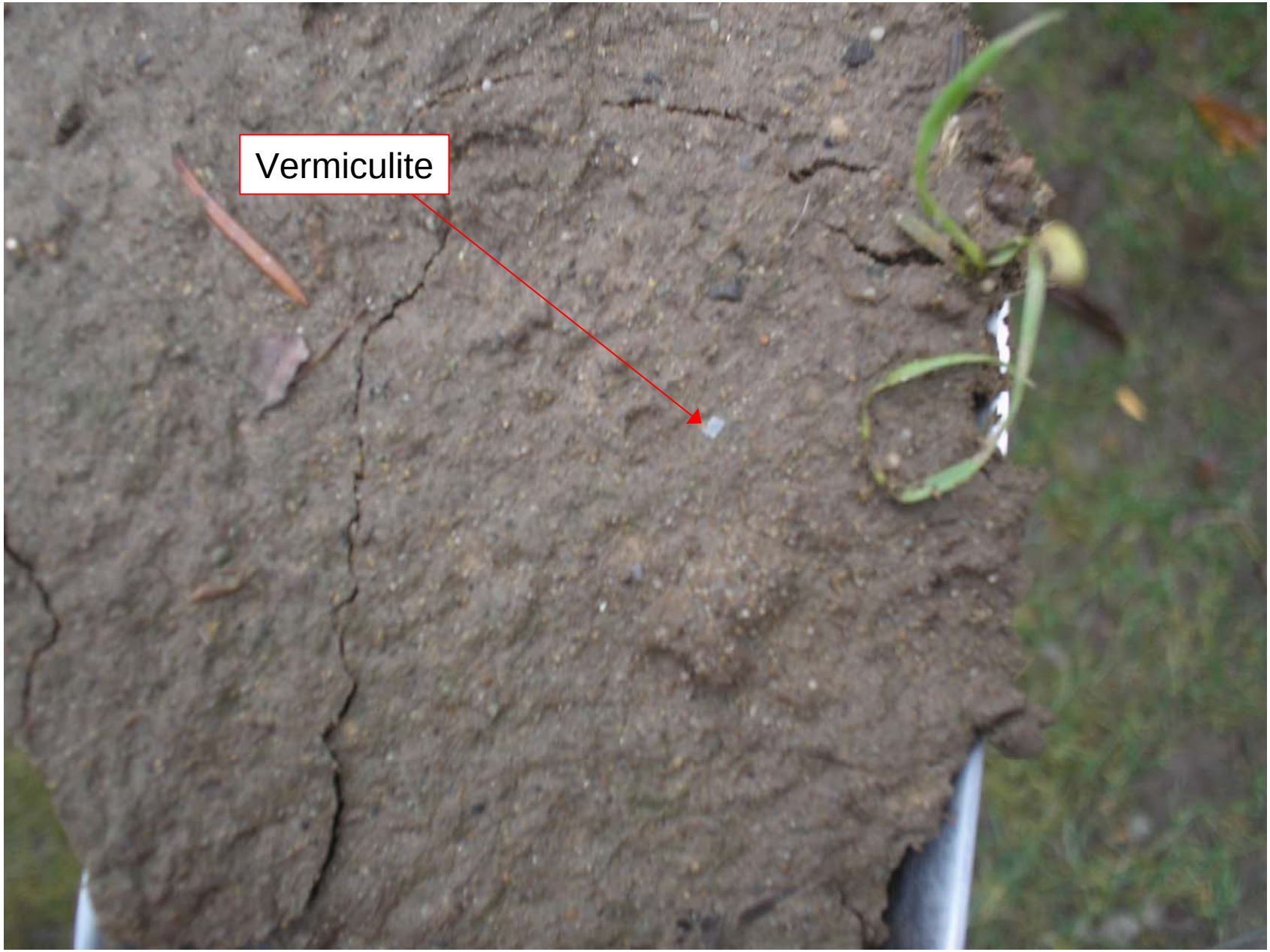



**Figure 1: Low Visible Vermiculite – A maximum of a few flakes of vermiculite observed within a given visual inspection point**

**Figure 2: Intermediate Visible Vermiculite – Vermiculite easily observed throughout visual inspection point, including the surface.**

**Figure 3: Intermediate Visible Vermiculite – Vermiculite easily observed throughout visual inspection point, including the surface.**

**Figure 4: High Visible Vermiculite – Visual inspection point contains Approximately 50% (or greater) vermiculite by volume**

Date: September 4, 2012 SOP No. EPA-LIBBY-09 (Revision 2)

Title: STANDARD OPERATING PROCEDURE FOR TEM DATA REVIEW AND DATA ENTRY VERIFICATION

Author: Erin Formanek (CDM Smith)

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**SYNOPSIS:** This standard operating procedure (SOP) provides a standardized method for review of raw transmission electron microscopy (TEM) data and verification of entry of TEM results into the project database. Steps included in this SOP are: a) selection of TEM analyses for review and verification, b) review of the original laboratory TEM benchsheets, and c) verification of the transfer of results from the benchsheets into the project database. This method is applicable for use only at the Libby Asbestos Superfund Site.

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**APPROVALS:**

| TEAM MEMBER | SIGNATURE/TITLE | DATE |
|---|---|---|
| EPA, Region 8 | Hania Zinner, RPM | 9/4/12 |
| CDM Smith | Erin Formanek | 9/4/12 |

| Revision | Date | Summary of Changes |
|---|---|---|
| 0 | 12/7/06 | -- |
| 1 | 3/5/08 | ▪ Modify selection procedure to exclude: 1) records associated with files uploaded due to error corrections, and 2) samples that will be validated under other review efforts.<br>▪ Modify SOP to include a check of samples with errors to ensure that corrections were made properly.<br>▪ Change review time period from monthly to quarterly.<br>▪ Add consistency review of data entered in accord with LB-000066.<br>▪ Refer to LB-000016 (ISO) and LB-000031 (AHERA/ASTM) for appropriate aspect ratio recording rules. |
| 2 | 9/4/12 | ▪ Added detail for the various roles of personnel involved in the verification process.<br>▪ Added diagram for workflow (Figure 1).<br>▪ Removed reference to the Libby2 Database.<br>▪ Added additional fields to be verified in the project database.<br>▪ Updated the names of the laboratory modifications that are referenced for appropriate aspect ratio recording rules.<br>▪ Remove previously specified required verification frequency because some verification is expected to happen during the quarterly validation process. |

## 1.0 OBJECTIVE

The purpose of this standard operating procedure (SOP) is to provide a standardized method for consistency review of transmission electron microscopy (TEM) data and analytical method(s) in the project database, and verification of results entered in the project database, and the electronic data deliverables (EDDs). **Figure 1** presents a flow diagram of the TEM verification process. Information included in this SOP is organized into the following sections:

2.0 Personnel and Qualifications
3.0 Data Summary Tables
4.0 Selection of TEM Records for Review
5.0 Consistency Review Procedure for TEM ISO 10312
6.0 Consistency Review Procedure for TEM AHERA/ASTM
7.0 Verification of Data Transfer from the Benchsheet to the Project Database
8.0 Reporting
9.0 References

## 2.0 PERSONNEL AND QUALIFICATIONS

*Verification Data Manager*

The verification data manager should be proficient in Microsoft® Access and Microsoft® Excel, as well as be familiar with TEM analytical reporting. The verification data manager is responsible for creating the Data Summary Tables (see **Attachment 1**) by querying the project database. The verification data manager is also responsible for coordinating with the database manager to ensure that discrepancies discovered in the data verification process have been resolved properly.

*Database Manager*

The database manager is responsible for maintaining the project database/laboratory documentation and coordinating with the laboratories to communicate discrepancies discovered during the verification process.

*Data Verifier*

The data verifier must be skilled and/or trained in interpretation of raw laboratory benchsheets and electronic data reporting files in support of TEM analysis. Data verifiers must be well-versed in TEM counting and recording rules as specified in TEM ISO 10312:1995(E) (ISO 1995), TEM AHERA (AHERA 1986), and ASTM D 5755-09 (ASTM 2009).

Data verifiers must also be well-versed in project-specific counting and recording rules as presented in the governing project documents that specify sampling and analysis procedures (e.g., Site Management Plan [SMP], Sampling and Analysis Plan [SAP], Quality Assurance Project Plan [QAPP]) to perform the required consistency reviews.

*Data Verification Coordinator*

The data verification coordinator (DVC) must be skilled in interpretation of raw laboratory benchsheets and electronic data reporting files in support of TEM analysis. DVCs must be well-versed in TEM counting and recording rules, as well as any project-specific counting and recording rules, as presented in the governing project documents that specify sampling and analysis procedures (e.g., SMP, SAP, QAPP). Lastly, DVCs are responsible for the following:

--Ensuring that verification reports are clear and accurate
--Ensuring that the steps outlined in this SOP are followed
--Training of data verifiers

## 3.0 DATA SUMMARY TABLES

Data Summary Tables are to be created by the verification data manager in Microsoft® Excel format and provided to the data verifier. Data Summary Tables will be used by the data verifier to select the TEM analyses for verification and to compare the project database results to the

hand-written laboratory benchsheets. **Attachment 1** presents an example of the Data Summary Tables for TEM. **Attachment 1A** illustrates the analytical and result information that will be verified. **Attachment 1B** illustrates the raw structure information that will also be verified.

## 4.0 SELECTION OF TEM RECORDS FOR REVIEW

The fraction of TEM records selected for review and verification will depend upon project-specific data quality needs. If less than 100% of analyses are to be verified, the goals for selecting a representative subset of TEM results for review and verification are provided below.

Over the course of a project, a minimum of ten percent (10%) of all field samples for which TEM analyses have been performed will be reviewed and verified. Laboratory quality control (QC) analyses, such as recounts and repreparations, will be excluded from the verification process (as their review is conducted as part of data validation). Selections should be made to ensure representation across the laboratory analysts performing TEM analyses. All TEM analyses will be stratified by analyst, with the number of analyses from each analyst selected for verification being in proportion to the total number analyzed. In addition, analyses will be stratified according to detect/non-detect status, with approximately 50% of the analyses selected for verification being detects, and 50% being non-detects. The following table illustrates the selection process if the goal is to select 10% of TEM analyses for review:

| | Number TEM Analyses Completed | | | Number TEM Analyses Selected | | |
|---|---|---|---|---|---|---|
| Analyst | Detect | Non-Detect | Total | Detect | Non-Detect | Total |
| 1 | 14 | 112 | 126 | 11 | 6 | 17 |
| 2 | 20 | 421 | 441 | 16 | 22 | 38 |
| 3 | 2 | 4 | 6 | 2 | 1 | 3 |
| 4 | 0 | 8 | 8 | 0 | 1 | 1 |
| Total | 36 | 545 | 581 | 29 | 30 | 59 |

| | Number to be Selected Based on 10% Goal | Actual Number Selected |
|---|---|---|
| Total | 58 | 59 |
| Detect | 29 | 29 |
| Non-Detect | 29 | 30 |

In this example, there are a total of 581 analyses available (36 detects + 545 non-detects), analyzed by four different analysts. Thus, the total number of analyses to be selected for review is 10% x 581 = 58.1 (rounded to 58). This total is to be split evenly between detects (29) and non-detects (29). The number of detects and non-detects selected for each analyst is calculated by multiplying the target number (29) by the fraction of the total detects and non-detects evaluated by the analyst. For example, for Analyst 1:

Number of detects selected = 29 x (14/36) = 11.3 (rounded to 11)
Number of non-detects selected = 29 **x** (112/545) = 5.9 (rounded to 6)

If an analyst has analyzed at least one analysis in a category (detect or non-detect), the minimum number of analyses to be selected is one. For example, for Analyst 4, the number of detects analyzed is zero, so the number of detects selected is zero. For Analyst 4, the number of non-detects to be selected (computed using the approach above) is:

Number of non-detects selected = 29 **x** (8/545) = 0.4

In this case, the number selected for verification is set to the minimum of 1.

As seen, this procedure will tend to select a higher proportion of detects (29 of 36 analyses, 81%) than non-detects (30 of 545 analyses, 6%). This approach is used because it is considered likely that the incidence of errors will be higher in analyses with one or more detected structures than in analyses with no detected structures.

The analyses that have been selected for analytical result verification should also undergo a verification of the field sample data sheet (FSDS) information. This verification process is outlined in Libby-specific SOP EPA-LIBBY-11.

## 5.0 CONSISTENCY REVIEW PROCEDURE FOR TEM ISO 10312

For each TEM ISO 10312 analysis to be verified, the data verifier will locate the original hand-written laboratory documentation within the appropriate laboratory job[a]. This hand-written documentation includes the laboratory benchsheets, internal laboratory chain of custody, and filter preparation logs. The data verifier will either print hard copies (if laboratory job provided as a PDF) or make photocopies (if laboratory job is provided as a hard copy data package) of the associated laboratory documentation for each TEM analysis selected for verification so that a hard copy is available for use in the verification.

**Figure 2** presents an example laboratory benchsheet. The data verifier will review the original hand-written laboratory benchsheets to determine if the raw structure data were recorded by the analyst in accordance with TEM ISO 10312 counting rules and any project-specific recording rules. The types of information to be reviewed include:

--The recorded structure types are consistent with the TEM ISO 10312 counting rules. Valid structure types include F, B, CC, CD, CF, CFO, CB, CBO, CR, MC, MD, MF, MFO, MB, MBO, and MR.

--Disperse complex structures (i.e., CD, MD) are broken down in accordance with TEM ISO 10312 counting rules and compact complex structures (i.e., CC, MC) are not broken down. For example, a CD43 should provide 4 secondary structures, with three secondary structures having a length greater than 5 um. In this example, the structure

[a] The laboratory job is either a hard copy data package or a scanned copy of the hard copy data package provided as a portable document file (PDF) by the analytical laboratory.

type for each of the recorded secondary structures should begin with the "C" prefix (e.g., CF, CB, CR).

--The primary and total columns on the laboratory benchsheet have been populated with non-zero numbers for all countable structures and a zero for all non-countable structures. If the primary and total columns have not been populated at all, it can be assumed that all structures recorded are countable unless otherwise stated in the comment field.

--If recorded, all non-asbestos mineral (NAM) structures are identified as non-countable structures.

--All recorded fibers (i.e., F, CF, MF) meet the appropriate aspect ratio requirement. [See the governing project documents for aspect ratio recording rules for ISO 10312.]

-- If Libby Laboratory Modification LB-000066 is applicable (as indicated in the project governing documents), the mineral type (e.g., WRTA) and appropriate spectra code (e.g., NaK) is recorded in the appropriate fields for all recorded LA, OA, and NAM structures.

--The mineral class is populated for all structures.

--Structure comments (e.g., < 3:1) are supported by recorded data.

## 6.0 CONSISTENCY REVIEW PROCEDURE FOR TEM AHERA/ASTM

For each TEM AHERA/ASTM analysis to be verified, the data verifier will locate the original hand-written laboratory documentation within the appropriate laboratory job[b]. This hand-written documentation includes the laboratory benchsheets, internal laboratory chain of custody, and filter preparation logs. The data verifier will either print hard copies (if laboratory job provided as a PDF) or make photocopies (if laboratory job is provided as a hard copy data package) of the associated laboratory documentation for each TEM analysis selected for verification so that a hard copy is available for use in the verification.

**Figure 2** presents an example laboratory benchsheet. The data verifier will review the original hand-written laboratory benchsheets to determine if the raw structure data were recorded by the analyst in accordance with AHERA/ASTM counting rules and any project-specific recording rules. The types of information to be reviewed include:

--The recorded structure types are consistent with the counting rules. For AHERA/ASTM, valid structure types include F, B, M, and C.

--The total column has been populated with non-zero numbers for all countable structures and a zero for all non-countable structures.

[b] The laboratory job is either a hard copy data package or a scanned copy of the hard copy data package provided as a portable document file (PDF) by the analytical laboratory.

--If recorded, all non-asbestos mineral (NAM) structures are identified as non-countable structures.

--The recorded structures meet the counting rule requirements. For AHERA/ASTM, all recorded fibers and matrices meet the appropriate aspect ratio requirement. [See the governing project documents for aspect ratio recording rules for AHERA/ASTM.]

--The recorded dimensions for matrices are the protrusion dimensions, not the matrix dimensions (provided sketches will be used to qualitatively assess dimensions).

--The mineral class is populated for all structures.

--If Libby Laboratory Modification LB-000066 is applicable (as indicated in the project governing documents), the mineral type (e.g., WRTA) and appropriate spectra code (e.g., NaK) is recorded in the structure comment field for all recorded LA, OA, and NAM structures.

--Structure comments (e.g., < 5:1) are supported by recorded data.

## 7.0 VERIFICATION OF DATA TRANSFER FROM THE BENCHSHEET TO THE PROJECT DATABASE

The following steps will be performed by the data verifier to ensure that data entered into the project database were entered properly. This data transfer verification can be accomplished by comparing the data in the project database (i.e., the data provided in the Data Summary Tables) to the handwritten benchsheets. If a discrepancy is noted, the verifier should confirm where in the data flow process that the error occurred. This can be achieved by reviewing the EDD that was submitted by the laboratory to confirm whether or not the data entry occurred successfully. If there is an error in the data entry to the EDD, correction to the EDD is needed. Otherwise, it is likely that the data upload procedure resulted in a misrepresentation of the data. In this case, the database manager will need to revise the upload procedure and reload the EDD.

7.1 The data verifier will verify the analysis-specific information provided in the Data Summary Tables (see **Attachment 1A**) against the original laboratory job documentation (i.e., laboratory benchsheets, internal laboratory chain of custody, filter preparation logs). [Note: Whenever possible, verification should be performed against hand-written notations, NOT internal laboratory summary tables prepared from hand-written notes. If hand-written notes are not available, this should be noted in the written report summarizing findings and recommendations that will be created at the end of the data verification process.]
Some examples of analysis-specific information that will be verified are provided below:

--Analysis Method
--Analysis Date
--Analyst Name
--Laboratory Name
--Laboratory Job Number
--Laboratory Sample Number
--Preparation Type (Direct, Indirect, or Indirect with Ashing)

--Effective Filter Area (EFA, $mm^2$) *[If direct preparation, value should match the primary filter area. If indirect preparation, value should match secondary filter area.]*
--Grid Opening Area (Ago, $mm^2$)
--Sample Type
--Sample Quantity [*For air samples, if the air volume on the benchsheet is within 2 liters of the volume on the Data Summary Table, rank this as concordant. Note: it is assumed that the air volume reported on the chain of custody is accurate. Verification of the air volume reported on the chain of custody is performed as part of the FSDS data verification and is outside the scope of this SOP.]*
--Sample Quantity Units
--Analysis Comments[c]
--Stopping and recording rules

Note that the fields presented in Attachment 1A are those that need to be verified for air and dust samples. There are additional fields that will need to be verified for other media types (as appropriate) as summarized below:

--Sample mass
--Sample mass units
--Ash residue total
--Ash residue total units
--Ash residue aliquot
--Ash residue aliquot units
-- FBA flow ratio
--Sample diameter
--Sample diameter units
-- Structures >10 um for LA, OA, and CH

7.2 The data verifier will check the calculation of the F-factor based on hand-written entries on the original hand-written laboratory benchsheet or filter preparation logs as shown in **Table 1.** The calculated F-factor will be compared with the column titled "F-factor" in the Data Summary Table (see **Attachment 1A**).

7.3 Using the original hand-written laboratory benchsheets, the data verifier will count the total number of unique grid openings evaluated. The number of grid openings counted will be compared with the grid openings counted in the Data Summary Table, which are grouped by mineral class and magnification level (see **Attachment 1A**). [Note: If the total number counted by the data verifier does not match the value in the grid openings counted column, this is an indicator that a grid opening was skipped, duplicated, or that a grid opening name was incorrectly entered into the EDD during the laboratory data entry process.]

7.4 Using the original hand-written laboratory benchsheets, the data verifier will count the number of asbestos structures for each mineral class across the grid openings evaluated. This

[c] The analysis comments field is to be reviewed for any applicable information regarding analysis status not captured in the "Filter Status" field which may impact the integrity of the sample results (e.g., Overloaded, Damaged, Missing, Canceled).

number will be compared with the columns titled "STRUCTCONCHIGHMAG" and/or "STRUCTCONCPCME" in the Data Summary Table for the corresponding mineral class (see **Attachment 1A**). Structure count results should be compared to both the "HighMag Structures" and "PCME Structure" columns if the analysis was performed at high magnification (i.e., ~20,000x). Structure count results should be compared only to the "PCME Structure" columns if the analysis was performed at low magnification (i.e., ~5,000x).

If asbestos structures recorded are part of a complex primary structure, only the secondary structures should be counted. For example, for a disperse matrix that consists of a primary matrix particle with 2 component fibers longer than 5 um (MD22), count the two component matrix fibers (MF), but not the primary matrix.

7.5 Using the original hand-written laboratory benchsheets, the data verifier will check the transfer of the raw structure data from the laboratory benchsheet to the Data Summary Table containing the raw structure results (see **Attachment 1B**). The data verifier will check the following fields:

--Grid
--Grid opening name
--Structure type
--Primary structure number
--Total structure number
--Structure type
--Structure dimension (length and width) *[In the event that the structure dimensions are recorded on the laboratory benchsheet as screen units, the recorded structure dimensions should be multiplied by the appropriate length and width screen units provided on the laboratory benchsheet to verify the dimensions present on the Data Summary Table.]*
--Mineral class
--Mineral description
--EDXA observation
--Structure identification
--Chrysotile count (this field records whether chrysotile structures, if observed, would have been recorded for the grid opening)
--Low Mag (this field records if the grid opening was examined under low magnification)
--Structure comments

7.6 The data verifier will ensure that an appropriate stopping rule has been met. As noted above, the governing project documents (e.g., SMP, SAP, QAPP) should specify the applicable TEM stopping rules. The data verifier should check that at least one stopping rule has been met and enter which rule(s) were met in the "Stopping Rule Achieved" column in the Data Summary Table.

7.7 The data verifier will check the calculation of the analysis sensitivity:

Air sensitivity $(cc)^{-1}$ = EFA / [GOx * Ago * F-factor * Air Volume (L)* 1000]
Dust sensitivity $(cm^2)^{-1}$ = EFA / [GOx * Ago * F-factor * Dust Sample Area $(cm^2)$]
Water sensitivity $(L)^{-1}$ = EFA / [GOx * Ago * Volume applied to the filter (L)]

Tree bark sensitivity $(cm^2)^{-1}$ = EFA / [GOx * Ago * F-factor * Bark Surface Area $(cm^2)$]
Duff/Tissue sensitivity $(g)^{-1}$ = EFA / [GOx * Ago * F-factor * Pre-Ashing Sample Mass (g)]
Fluidized bed soil sensitivity $(g)^{-1}$ = EFA / [GOx * Ago * F-factor * Sample Mass (g) * $Q_R$]

where:

EFA = effective filter area, in square millimeters $(mm^2)$
GOx = grid openings counted
Ago = grid opening area $(mm^2)$
$Q_R$ = fluidized bed flow ratio
L = liters
g = grams
$cm^2$ = square centimeters
$mm^2$ = square millimeters

7.8 The data verifier will check the calculation of the sample concentration:

Concentration = Number of Asbestos Structures * Sensitivity

## 8.0 REPORTING

For each field to be verified, if the data in the Data Summary Table matches the information in the hard copy laboratory job documentation, mark the appropriate field on the hard copy with a check mark. If the Data Summary Table does not match the hard copy laboratory job documentation, circle the incorrect entry on the hard copy, and note the specific discrepancy in the Data Summary Table in the "Comment" column (see **Attachment 1**). For example, "Grid openings counted is 40 based on hard copy, but 39 in the EDD/database".

As the verification of each selected analysis is completed, the data verifier will enter their company name and their first initial and last name (e.g., E. Smith) in the Data Summary Table in the appropriate columns.

When the verification is complete for all analyses selected, the data verifier will prepare an electronic data verification package. This package will consist of:

-- A summary report summarizing findings and recommendations. **Attachment 2** provides an example template for reporting TEM data verification results.
--A scanned copy of the hard copy laboratory documentation used in the verification process (which includes all verification check marks for reviewed fields).
--An electronic attachment of the Data Summary Tables (which includes any data verifier comments).

The DVC will review the data verification package for accuracy and completeness. If any deficiencies are noted, the DVC will re-train verification personnel and make any corrections as necessary.

In addition to verifying all issues noted in summary report, the DVC will perform an independent data verification of 5% of the analyses verified to ensure that any potential issues have been identified correctly. The DVC will indicate in **Attachment 1** which analyses were

selected for review. If any deficiencies are noted, the DVC will re-train verification personnel and make any corrections as necessary. If the DVC disagrees with the error noted in the Data Summary Table, the discrepancy will be revised, and the DVC will replace the verifiers name with his/her name for the analysis. The summary report will be revised by the DVC to reflect any changes as needed.

The electronic data verification package will then be provided to the appropriate project database manager, or their designee, to facilitate the correction process for laboratory EDDs and/or the hand-written laboratory benchsheets by the analytical laboratory. The verification data manager, or their designee, will record the resolution date of any corrections in the appropriate column of the Data Summary Table. Note that all of the following criteria must have been met for a TEM analysis to be considered verified:

--All necessary corrections have been made to the laboratory EDD.
--The corrected laboratory EDD has been re-submitted by the analytical laboratory to the appropriate parties (as specified in the governing project documents).
--The corrected laboratory EDD has been uploaded to the project database.
-- All necessary corrections have been made to the hand-written laboratory benchsheet.
--The corrected hand-written laboratory benchsheet has been re-submitted by the analytical laboratory to the appropriate parties.
--Signatures for the data verifier, DVC, and verification data manager have been added to the verification summary report.

## 9.0 REFERENCES

Asbestos Hazardous Emergency Response Act (AHERA). 1986. Title 20, Chapter 52, Sec. 4011. Public Law 99-519.

American Society for Testing and Materials (ASTM). 2009. *Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations.* ASTM D 5755-09. American Society for Testing and Materials. January 2009.

ISO. 1995. *Ambient Air – Determination of asbestos fibres – Direct-transfer transmission electron microscopy method.* International Organization for Standardization, Reference Number ISO 10312:1995(E). http://www.iso.org/iso/catalogue_detail.htm?csnumber=18358

**FIGURE 1**

**TEM VERIFICATION PROCESS**

**FIGURE 1. TEM VERIFICATION PROCESS**

| Personnel | Step |
|---|---|
| ***Verification Data Manager*** | 1. Create Data Summary Tables in Excel and provide to the verifier. |
| ***Verifier*** | 2. Select TEM analyses for review. |
| | 3. Locate hand-written laboratory documentation (laboratory benchsheets, COCs, preparation logs) and print or photocopy hard copies for selected |
| | 4. Perform a consistency review utilizing the Data Summary Tables and hand-written laboratory |
| | 5. Verify that analysis-specific data were correctly transferred from the laboratory documentation to the project database (utilizing the Data Summary Tables and hand-written laboratory |
| | 6. Verify the F-factor, GOx, number of asbestos structures reported, analysis sensitivity, and reported concentration in the Data Summary Tables (utilizing the hand-written laboratory |
| | 7. Verify that an appropriate stopping rule was met; record the stopping rule met in the Data |
| | 8. Verify that raw structure data were correctly transferred from the laboratory benchsheet to the database (utilizing the Data Summary Tables and hardcopy hand-written laboratory benchsheets). |
| | 9. Create electronic data verification package and provide to Data Verification Coordinator. |
| ***Data Verification Coordinator (DVC)*** | 10. Review electronic data verification package |
| | 11. Provide electronic data verification package to database manager. |
| ***Database Manager*** | 12. Correspond with laboratories to ensure appropriate corrections are made to laboratory EDDs/ benchsheets. |
| | 13. Upload corrected laboratory EDDs to project database. |
| | 14. Update project file with revised laboratory |
| ***Verification Data Manager*** | 15. Record issue resolution date in the Data Summary Table. |
| ***Verifier, DVC, and Verification Data Manager*** | 16. Sign and date verification summary report. |

TEM = transmission electron microscopy
COC = chain of custody
EDD = electronic data deliverable
GOx = number of grid openings counted

**FIGURE 2**

**EXAMPLE OF TEM LABORATORY BENCHSHEET**

# FIGURE 2. EXAMPLE TEM LABORATORY BENCHSHEET



**LIBBY**

***TEM Asbestos Structure Count_Air-DustEDD_37b***

| | |
|---|---|
| Laboratory ID: | |
| Instrument ID | |
| Voltage (KV) | |
| Mag. | HIGH / LOW |
| Grid opening area (mm2) | |
| Scale: 1L = | |
| Scale: 1D = | |
| Primary filter area (mm2) | |
| Secondary Filter Area (mm2) | |
| Category (Field, Blank) | |
| Primary filter pore size (um) | |

| | | | |
|---|---|---|---|
| EPA Sample Number: | | Tag: | |
| Matrix (A=Air, D=Dust, DF = Dustfall): | | | |
| Air volume (L), dust area (cm2), or dustfall container area (cm2) | | | |
| Date received by lab | | | |
| Lab Job Number: | | | |
| Lab Sample Number: | | | |
| Number of grids prepared | | | |
| Prepared by | | | |
| Preparation date | | | |
| EPA COC Number: | | | |
| Secondary filter pore size (um) | | | |

| | |
|---|---|
| Analyzed by: | |
| Analysis date | |
| Method (D=Direct, I=Indirect, IA=Indirect-ashed) | |
| If sample type = air, is there loose material or debris in the cowl? (Yes, No) | |
| Analysis Method (TEM-ISO, TEM-AHERA, TEM-ASTM) | |
| Grid storage location | |
| Archive filter(s) storage location | |
| Lab QC Type (Not QC, Recount Same, Recount Different, Re-prep, Verified Analysis, Reconciliation, Lab Blank, Interlab) | |
| Estimated Particulate Loading (%) | |

F-Factor Calculation (Indirect Preps Only):

*Enter data in appropriate cells provided to the right----->*

Recording Rules:

Minimum Aspect Ratio (circle one):

none ≥ 3:1 ≥ 5:1

| | |
|---|---|
| Minimum Length (um): | |
| Minimum Width (um): | |

Stopping Rules:

| | |
|---|---|
| Target Sensitivity: | |
| Max Area Examined: | |
| Target # of Structures: | |

| Grid | Grid Opening | Structure Type | No. of Structures | | Dimensions | | Identification | Mineral Class (see below) | | | | Mineral Desc | EDXA | Sketch/ Comments | 1 = yes, blank = no | | | CH Not Counted |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Primary | Total | Length | Width | | LA | OA | CH | NAM | | | | Sketch | Photo | EDS | |
| | | | | | | | | | | | | | | | | | | |
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F-factor Calculation:

Indirect Prep Inputs

| | |
|---|---|
| | Fraction of primary filter used for indirect prep or ashing *[For dust and dustfall, enter 1.0]* |
| | First resuspension volume or rinsate volume (mL) |
| | Volume applied to secondary filter (mL) or used for serial dilution |

Inputs for Serial Dilutions

| | |
|---|---|
| | Second resuspension volume (mL) |
| | Volume applied to secondary filter (mL) or used for serial dilution |
| | Third resuspension volume (mL) |
| | Volume applied to secondary filter (mL) |

Input for Ashing of Secondary Filter

| | |
|---|---|
| | Fraction of secondary filter used for ashing |

LA = Libby-type amphibole OA = Other (non-Libby type) amphibole CH = Chrysotile NAM = Non-asbestos material

If sample was analyzed by more than one analyst or across multiple analysis dates, enter analysis details below.

| | Analyst #2 | Analyst #3 |
|---|---|---|
| Analyzed by: | | |
| Analysis date: | | |
| Instrument: | | |

Grid opening traverse direction (circle one):

H Horizontal

V Vertical

Are prepped grids acceptable for analysis? (circle one) Yes No

If No, explain:

**TABLE 1**

**F-FACTOR EQUATIONS**

## TABLE 1. F-FACTOR EQUATIONS

| Media | Filter Preparation | F-Factor Equation |
|---|---|---|
| Air/Dust | Direct | F-factor = 1 |
| | Indirect | F-factor = Fraction of primary filter used * (Volume applied to secondary filter / Total re-suspension volume) |
| | Indirect-Ashed | F-factor = Fraction of primary filter used * (Volume applied to filter for ashing / Total re-suspension volume, pre-ashing) * Fraction of filter that was ashed * (Volume applied to secondary filter / Total re-suspension volume, post-ashing) |
| Water | | F-factor = [1 / ((Total volume after first dilution (mL) / Volume used from primary sample (mL)) * (Total volume after second dilution (mL) / Volume used from 1st dilution (mL))] |
| Tree Bark | | F-factor = [(Ashed residue mass (g), aliquot used in dilution / Ashed residue mass (g), total) * (Volume applied to secondary filter (mL) or used for serial dilution / First resuspension volume or rinsate volume (mL)) * (Volume applied to secondary filter (mL) or used for serial dilution / Second resuspension volume (mL)) * (Volume applied to secondary filter (mL) or used for serial dilution / Third resuspension volume (mL))] |
| Duff/Tissue | | F-factor = [(Ashed residue mass (g), aliquot used in dilution / Ashed residue mass (g), total) * (Volume applied to filter (mL) / Resuspension volume (mL)] |
| Fluidized Bed Soil | Direct | F-factor = 1 |
| | Indirect | F-factor = [Fraction of original filter used * (Volume applied to secondary filter (mL) / Total suspension volume (mL))] |

# ATTACHMENT 1

# EXAMPLE OF DATA SUMMARY TABLES

**ATTACHMENT 1A. DATA SUMMARY OF ANALYTICAL AND RESULT INFORMATIO!**

| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | Recording Rules | | | | | | Stopping Rules | | | | Grid Openings Counted | | | | STRUCTCNTHIGHMAG | | | STRUCTCNTPCME | | | Sensitivity | | STRUCTCONCHIGHMAG | | | STRUCTCONCPCME | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Samp No | File Revision No | Lab ID | Instrument | Mag High | Mag Low | GO Size | EFA | Tag | Analysis Quantity | Analysis Quantity Units | Receipt Date | Lab Job Number | Lab SampleID | Number Grid Prep | Preparer Name | Prep Date | Analyst Name | Analysis Date | Prep Method | Loose Material | Analysis Method | Est Filter Loading | Indirect Fraction Primary Filter | Aliquot 1 | Volume 1 | Aliquot 2 | Volume 2 | Aliquot 3 | Volume 3 | Indirect Fraction Secondary Filter | F Factor | Analysis Comments | Min AR High | Min AR Low | Min Length High | Min Length Low | Min Width High | Min Width Low | Target Sens | Maximum GOs | Max Area Examined | Target N Strucs | Chrys High | Chrys Low | LA/OA High | LA/OA Low | LA | OA | CH | LA | OA | CH | LA/OA | CH | LA | OA | CH | LA | OA | CH | Stopping Rule Achieved | Verifier's Company | Verifier's Name | Comment | Correction Date |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

## ATTACHMENT 1B. DATA SUMMARY OF STRUCTURE INFORMATION

| Samp No | StructureID | Row Index | Grid | Grid Opening | Structure Type | Primary | Total | Length | Width | AR | Mineral Class | Mineral Desc | EDXA Observation | Structure Identification | Chrysotile Count | Low Mag | Structure Comment | Verifier's Company | Verifier's Name | Comment | Correction Date |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | | | | | | | | | |
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# ATTACHMENT 2

# EXAMPLE OF TEM DATA VERIFICATION SUMMARY REPORT

# TEM CONSISTENCY REVIEW AND DATA TRANSFER VERIFICATION REPORT

Project/Dataset Description: _____

***SUMMARY OF FINDINGS AND DATA QUALITY IMPLICATIONS***

_____

_____

_____

_____

_____

_____

_____

Recommendations for future review and verification: _____

_____

_____

_____

_____

***Data Verifier:*** _____ ***Date:*** _____

***Data Verification Coordinator:*** _____ ***Date:*** _____

***Verification Data Manager*:***_____ ***Date:*** _____

****The Verification Data Manager acknowledges that all issues discovered during the verification process have been resolved and that the following criteria have been met:***

--All necessary corrections have been made to the laboratory EDD.
--The corrected laboratory EDD has been re-submitted by the analytical laboratory to the appropriate parties (as specified in the governing project documents).
--The corrected laboratory EDD has uploaded to the project database.
-- All necessary corrections have been made to the hand-written laboratory benchsheet.
--The corrected hand-written laboratory benchsheet has been re-submitted by the analytical laboratory to the appropriate parties.

***TEM ISO 10312 SELECTION AND CONSISTENCY REVIEW RESULTS***

| Analyst, Lab | Number of TEM ISO 10312 Analyses | | | Number of TEM ISO 10312 Analyses Selected for Review | | |
|---|---|---|---|---|---|---|
| | Detect | Non-Detect | Total | Detect | Non-Detect | Total |
| Analyst #1, Lab Name | | | | | | |
| Analyst #2, Lab Name | | | | | | |
| … | | | | | | |
| | | | | | | |
| Total | | | | | | |

| | Goal | Actual |
|---|---|---|
| Selected Total | _____ | _____ |
| Selected Detects | _____ | _____ |
| Selected Non-Detects | _____ | _____ |

Detailed summary of bench sheet consistency review –

Number of analyses reviewed: _____ (_____% of total analyses selected)

If not all analyses could be reviewed, provide a brief explanation for why: _____

_____

Number of analyses with recording issues identified: _____ (_____% of total analyses reviewed)

Types of recording issues identified (indicate the number of analyses):

_____ Reported structure types are inconsistent with ISO 10312

_____ Primary and/or total columns are not populated correctly

_____ NAM structures are recorded and not identified as non-countable

_____ Fibers recorded as countable do not meet counting rules

_____ Mineral class designation is missing or inconsistent

_____ Structure comments are inconsistent with recorded data

Do the recording issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

_____

***TEM AHERA/ASTM SELECTION AND CONSISTENCY REVIEW RESULTS***

| Analyst, Lab | Number of TEM 100.2 Analyses | | | Number of TEM 100.2 Analyses Selected for Review | | |
|---|---|---|---|---|---|---|
| | Detect | Non-Detect | Total | Detect | Non-Detect | Total |
| Analyst #1, Lab Name | | | | | | |
| Analyst #2, Lab Name | | | | | | |
| … | | | | | | |
| | | | | | | |
| Total | | | | | | |

| | Goal | Actual |
|---|---|---|
| Selected Total | _____ | _____ |
| Selected Detects | _____ | _____ |
| Selected Non-Detects | _____ | _____ |

Detailed summary of bench sheet consistency review –

Number of analyses reviewed: _____ (_____% of total analyses selected)

If not all analyses could be reviewed, provide a brief explanation for why: _____

_____

Number of analyses with recording issues identified: _____ (_____% of total analyses reviewed)

Types of recording issues identified (indicate the number of analyses):

_____ Reported structure types are inconsistent with TEM 100.2

_____ Fibers recorded as countable do not meet counting rules

_____ Mineral class designation is missing or inconsistent

_____ Structure comments are inconsistent with recorded data

Do the recording issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

_____

***DATA TRANSFER VERIFICATION RESULTS***

Number of analyses verified: _____ (_____% of total analyses selected)

Number of analyses with data transfer issues identified: _____ (_____% of total analyses verified)

Types of data transfer issues identified:

_____ Incorrect/missing information on analysis details (e.g., lab job number, analysis date, filter status)

_____ Incorrect/missing information on raw structure details (e.g., length, width, mineral class)

_____ F-factor calculation is incorrect or inputs are missing

_____ Air volume, dust area, sample mass, etc. reported by laboratory is inconsistent with field value

_____ Number of grid openings counted is incorrect

_____ Sensitivity calculation is incorrect or inputs are missing

_____ Total number of countable structures is incorrect

Do the data transfer issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

_____

Comments: _____

_____

_____

_____

Date: August 23, 2012 SOP No. EPA-LIBBY-10 (Revision 0)

Title: STANDARD OPERATING PROCEDURE FOR PLM DATA REVIEW AND DATA ENTRY VERIFICATION

Author Natalie Ross (CDM Smith)

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**SYNOPSIS:** This standard operating procedure (SOP) provides a standardized method for review of raw polarized light microscopy (PLM) data and verification of entry of PLM results into the project database. Steps included in this SOP are: a) selection of PLM analyses for review and verification, b) review of the original laboratory PLM benchsheets, and c) verification of the transfer of results from the benchsheets into the project database. This method is applicable for use only at the Libby Asbestos Superfund Site.

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**APPROVALS:**

| TEAM MEMBER | SIGNATURE/TITLE | DATE |
|---|---|---|
| EPA, Region 8 | Jamia Zimmer, RPM | 9/4/12 |
| CDM Smith | Natalie Ross | 9.4.12 |

| Revision | Date | Reason for Revision |
|---|---|---|
| 0 | 8/23/12 | -- |
| | | |
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## 1.0 OBJECTIVE

The purpose of this standard operating procedure (SOP) is to provide a standardized method for consistency review of polarized light microscopy (PLM) data and analytical method(s) in the project database, and verification of results entered in the project database, and the electronic data deliverables (EDDs). **Figure 1** presents a flow diagram of the PLM verification process. Information included in this SOP is organized into the following sections:

2.0 Personnel and Qualifications
3.0 Data Summary Tables
4.0 Selection of PLM Records for Review
5.0 Consistency Review Procedure for PLM-VE
6.0 Consistency Review Procedure for PLM-9002
7.0 Consistency Review Procedure for PLM-Grav
8.0 Verification of Data Transfer from the Benchsheet to the Project Database
9.0 Reporting
10.0 References

## 2.0 PERSONNEL AND QUALIFICATIONS

*Verification Data Manager*

The verification data manager should be proficient in Microsoft® Access and Microsoft® Excel as well as be familiar will PLM analytical reporting. The verification data manager is responsible for creating the Data Summary Tables (see **Attachment 1**) by querying the project database. The verification data manager is also responsible for coordinating with the database manager to ensure that discrepancies discovered in the data verification process have been resolved properly.

*Database Manager*

The database manager is responsible for maintaining the project dataset/laboratory documentation and coordinating with the laboratories to communicate discrepancies discovered during the verification process.

*Data Verifier*

The data verifier must be skilled and/or trained in interpretation of raw laboratory benchsheets and electronic data reporting files in support of PLM analysis. Data verifiers must be well-versed in data reporting requirements and recording rules as specified in the National Institute for Occupational Safety and Health (NIOSH) Method 9002 (PLM-9002) and the most recent versions of site specific SOPs SRC-LIBBY-03 (referred to as PLM visual area estimation or "PLM-VE") and SRC-LIBBY-01 (referred to as PLM gravimetric analysis or "PLM-Grav").

Data verifiers must also be well-versed in project-specific recording rules as presented in the governing project documents that specify sampling and analysis procedures (e.g., Site

Management Plan [SMP], Sampling and Analysis Plan [SAP], Quality Assurance Project Plan [QAPP]) to perform the required consistency reviews.

*Data Verification Coordinator*

The data verification coordinator (DVC) must be skilled in interpretation of raw data laboratory benchsheets and electronic data reporting files in support of PLM analysis. DVCs must be well-versed in PLM methods and recording rules, as well as any in project-specific analytical requirements, as presented in the governing project documents that specify sampling and analysis procedures (e.g., SMP, SAP, QAPP). Lastly, DVCs are responsible for the following:

--Ensuring that verification reports are clear and accurate
--Ensuring that the steps outlined in this SOP are followed
--Training of data verifiers

## 3.0 DATA SUMMARY TABLE

Data Summary Tables are to be created by the verification data manager in Microsoft® Excel format and provided to the data verifier. Data Summary Tables will be used by the data verifier to select the PLM analyses for verification and to compare the project database results to the hand-written laboratory benchsheets. **Attachment 1** presents an example of the Data Summary Table that illustrates the analytical and result information for PLM that will be verified.

## 4.0 SELECTION OF PLM RECORDS FOR REVIEW

The fraction of PLM records selected for review and verification will depend upon project-specific data quality needs. If less than 100% of analyses are to be verified, the goals for selecting a representative subset of PLM results for review and verification are provided below.

Over the course of a project, a minimum of ten percent (10%) of all field samples for which PLM analyses have been performed will be reviewed and verified. Laboratory quality control (QC) analyses will be excluded from the verification process (as their review is conducted as part of data validation). Selections should be made to ensure representation across the laboratory analysts performing PLM analyses. All PLM analyses will be stratified by analyst, with the number of analyses from each analyst selected for verification being in proportion to the total number analyzed. In addition, analyses will be stratified according to detect/non-detect status, with approximately 50% of the analyses selected for verification being detects, and 50% being non-detects. The following table illustrates the selection process if the goal is to select 10% of PLM analyses for review:

| | Number PLM Analyses Completed | | | Number PLM Analyses Selected | | |
|---|---|---|---|---|---|---|
| Analyst | Detect | Non-Detect | Total | Detect | Non-Detect | Total |
| 1 | 14 | 112 | 126 | 11 | 6 | 17 |
| 2 | 20 | 421 | 441 | 16 | 22 | 38 |
| 3 | 2 | 4 | 6 | 2 | 1 | 3 |

| Analyst | Number PLM Analyses Completed | | | Number PLM Analyses Selected | | |
|---|---|---|---|---|---|---|
| | Detect | Non-Detect | Total | Detect | Non-Detect | Total |
| 4 | 0 | 8 | 8 | 0 | 1 | 1 |
| Total | 36 | 545 | 581 | 29 | 30 | 59 |

| | Number to be Selected Based on 10% Goal | Actual Number Selected |
|---|---|---|
| Total | 58 | 59 |
| Detect | 29 | 29 |
| Non-Detect | 29 | 30 |

In this example, there are a total of 581 analyses available (36 detects + 545 non-detects), analyzed by four different analysts. Thus, the total number of analyses to be selected for review is 10% x 581 = 58.1 (rounded to 58). This total is to be split evenly between detects (29) and non-detects (29). The number of detects and non-detects selected for each analyst is calculated by multiplying the target number (29) by the fraction of the total detects and non-detects evaluated by the analyst. For example, for Analyst 1:

Number of detects selected = 29 x (14/36) = 11.3 (rounded to 11)
Number of non-detects selected = 29 x (112/545) = 5.9 (rounded to 6)

If an analyst has analyzed at least one analysis in a category (detect or non-detect), the minimum number of analyses to be selected is one. For example, for Analyst 4, the number of detects analyzed is zero, so the number of detects selected is zero. For Analyst 4, the number of non-detects to be selected (computed using the approach above) is:

Number of non-detects selected = 29 x (8/545) = 0.4

In this case, the number selected for verification is set to the minimum of 1.

As seen, this procedure will tend to select a higher proportion of detects (29 of 36 analyses, 81%) than non-detects (30 of 545 analyses, 6%). This approach is used because it is considered likely that the incidence of errors will be higher in analyses with detected asbestos than in analyses that are non-detect.

The analyses that have been selected for analytical result verification should also undergo a verification of the field sample data sheet (FSDS) information. This verification process is outlined in Libby-specific SOP EPA-LIBBY-11.

## 5.0 CONSISTENCY REVIEW PROCEDURE FOR PLM-VE

For each PLM-VE analysis to be verified, the data verifier will locate the original hand-written laboratory benchsheets within the appropriate laboratory job[a]. The data verifier will either print hard copies (if laboratory job provided as a PDF) or make photocopies (if laboratory job is provided as a hard copy data package) of the associated laboratory documentation for each PLM analysis selected for verification so that a hard copy is available for use in the verification.

**Figure 2** presents an example laboratory benchsheet for PLM-VE. The data verifier will review the original hand-written laboratory benchsheets to determine if the reported results are in accordance with PLM-VE reporting rules and any project-specific recording rules. The types of information to be reviewed include:

--Asbestos is classified into one of three categories; Libby amphibole (LA), other amphibole asbestos (OA), and chrysotile (CH)

--The reported qualifier codes for LA, OA and CH are consistent with the PLM-VE reporting rules. Valid qualifier codes for LA include "ND" (not detected), "Tr" (trace) and "<" (less than). Valid qualifier codes for OA and CH include "ND" and "<".

--When the reported qualifier is "<", the concentration for LA, OA and CH should be recorded as 1%.

--When the reported concentration for LA, OA, and CH is equal to or greater than 1%, a numeric integer (1, 2, 3, etc.) should be recorded in the concentration column without an entry in the qualifier column.

*Important Notice: Data reporting requirements have changed over time. Concentration values recorded as mass fraction (MF) percent, area fraction (AF) percent, or FRAC% on the benchsheet are equivalent to the "Conc" CharacteristicID in the project database.*

--For LA, results are assigned to one of four "bins" with the associated qualifiers and concentrations, as follows:

| Bin | Qualifier | Concentration |
|---|---|---|
| A | ND | |
| B1 | Tr | |
| B2 | < | 1 |
| C | | 1,2,3, etc. |

--If recorded, OA structure types are identified as "AMOS" (amosite), "ANTH" (anthophyllite), "CROC" (crodidolite), or "MULTI" (multiple).

--When asbestos fibers are positively identified in a sample, optical properties should be recorded for each asbestos type present.

[a] The laboratory job is either a hard copy data package or a scanned copy of the hard copy data package provided as a portable document file (PDF) by the analytical laboratory.

--When non-asbestos material (NAM) fibers are observed, at least one optical property should be recorded on the benchsheet that distinguishes the fiber from asbestos. This field is not required to be entered into the EDD.

> *Important Notice: Data reporting requirements have changed over time. Historical analyses may not have met this requirement.*

--If optical properties are recorded on the benchsheet, the ambient temperature of the laboratory is also recorded.

> *Important Notice: Data reporting requirements have changed over time. Historical analyses may not have met this requirement.*

## 6.0 CONSISTENCY REVIEW PROCEDURE FOR PLM-9002

For each PLM-9002 analysis to be verified, the data verifier will locate the original hand-written laboratory benchsheets within the appropriate laboratory job[b]. The data verifier will either print hard copies (if laboratory job provided as a PDF) or make photocopies (if laboratory job is provided as a hard copy data package) of the associated laboratory documentation for each PLM-9002 analysis selected for verification so that a hard copy is available for use in the verification.

**Figure 3** presents an example laboratory benchsheet for PLM-9002. The data verifier will review the original hand-written laboratory benchsheets to determine if the reported results are in accordance with PLM-9002 reporting rules and any project-specific recording rules. The types of information to be reviewed include:

--Asbestos is classified into one of three categories: Tremolite-Actinolite (TREM-ACTN), OA, and CH

--The reported qualifier codes for TREM-ACTN, OA and CH are consistent with the method reporting rules. Valid qualifier codes include "ND" (not detected) and "<" (less than).

--When the reported qualifier is "<", the concentration for TREM-ACTN, OA and CH should be recorded as 1%.

--When the reported concentration for TREM-ACTN, OA, and CH is equal to or greater than 1%, a numeric integer (1, 2, 3, etc.) should be recorded in the concentration column without an entry in the qualifier column.

> *Important Notice: Data reporting requirements have changed over time. Concentration values recorded as mass fraction (MF) percent, area fraction (AF) percent, or FRAC% on the benchsheet are equivalent to the "Conc" CharacteristicID in the project database.*

[b] The laboratory job is either a hard copy data package or a scanned copy of the hard copy data package provided as a portable document file (PDF) by the analytical laboratory.

--Results are not assigned to bins.

--If recorded, OA structure types are identified as "AMOS" (amosite), "ANTH" (anthophyllite), "CROC" (crodidolite), or "MULTI" (multiple).

--When asbestos fibers are positively identified in a sample, optical properties should be recorded for each asbestos type present.

--When non-asbestos material (NAM) fibers are observed, at least one optical property should be recorded on the benchsheet that distinguishes the fiber from asbestos. This field is not required to be entered into the EDD.

*Important Notice: Data reporting requirements have changed over time. Historical analyses may not have met this requirement.*

-- If optical properties are recorded on the benchsheet, the ambient temperature of the laboratory is also recorded.

*Important Notice: Data reporting requirements have changed over time. Historical analyses may not have met this requirement.*

## 7.0 CONSISTENCY REVIEW PROCEDURE FOR PLM-GRAV

For each PLM-Grav analysis to be verified, the data verifier will locate the original hand-written laboratory benchsheets within the appropriate laboratory job[c]. The data verifier will either print hard copies (if laboratory job provided as a PDF) or make photocopies (if laboratory job is provided as a hard copy data package) of the associated laboratory documentation for each PLM analysis selected for verification so that a hard copy is available for use in the verification.

**Figure 4** presents an example laboratory benchsheet for PLM-Grav. The data verifier will review the original hand-written laboratory benchsheets to determine if the reported results are in accordance with PLM-Grav reporting rules and any project-specific recording rules. Examples of information to be reviewed include:

--The reported qualifier codes for LA, OA, and CH are consistent with the method reporting rules. Valid qualifier codes include "ND" (not detected) and "Tr" (trace).

--If recorded, OA structure types are identified as "AMOS" (amosite), "ANTH" (anthophyllite), "CROC" (crocidolite) or "MULTI" (multiple).

--If a concentration value is reported in the project database, the data verifier will check that the following calculations are accurate based on the hand-written results recorded on the laboratory benchsheet:

[c] The laboratory job is either a hard copy data package or a scanned copy of the hard copy data package provided as a portable document file (PDF) by the analytical laboratory.

Asbestos concentration (%) = [(mass of asbestos/1000) /mass of sample] x 100

where:

Mass of asbestos (LA, OA or CH) in milligrams (mg) = [*weight of sample container and asbestos – weight of empty sample container]*

1000 = conversion factor (covert from mg to g)

Mass of sample in grams (g) = [*weight of sample container and total sample – weight of empty sample container]*

100 = conversion factor (convert from fraction to percentage)

## 8.0 VERIFICATION OF DATA TRANSFER FROM THE BENCHSHEET TO THE PROJECT DATABASE

The data verifier will ensure that data entered into the project database were entered properly. This data transfer verification can be accomplished by comparing the data in the project database (i.e., the data provided in the Data Summary Tables) to the handwritten benchsheets.

The data verifier will verify the analysis-specific information provided in the Data Summary Tables (see **Attachment 1**) against the original laboratory documentation (e.g., laboratory benchsheets). [Note: Whenever possible, verification should be performed against hand-written notations, NOT internal laboratory summary tables prepared from hand-written notes. If hand-written notes are not available, this should be noted in the written report summarizing findings and recommendations that will be created at the end of the verification process.] Some examples of analysis-specific information that will be verified are provided below:

--Laboratory Name
--Laboratory Job Number
--SOP Name/Revision
--Instrument ID
--Sample Number
--Tag
--QC Type
--Lab Sample ID
--Date Analyzed
--Analyst Name
--Sample Appearance (e.g., tan, non-fibrous, homogeneous)
--Deviation (Y/N)
--Comments
--Non-Fibrous Matrix Materials (if reported)
--Temperature (if reported in the project database)

For PLM-VE Results:

--LA Bin

--Concentration result for LA, OA, and CH (Note: this is reported as "Conc" in the project database).

For PLM-9002 Results:

--Concentration result for TREM-ACTN, OA, and CH (Note: this is reported as "Conc" in the project database).

For PLM-Grav Results:

--Concentration result for LA, OA, and CH (Note: this is reported as "Conc" in the project database).

Optical Property Data for Detected Samples *(for PLM-VE and PLM-9002 only):*

--Habit (acicular, fiber bundles, prismatic, straight, tapered)
--Fiber Color (blue, brown, colorless, gray, green, red, tan yellow)
--Sign of Elongation (+/-)
--Pleochroism (Y/N)
--Extinction Angle (parallel, inclined)
--Refractive Index (α)
--Refractive Index (γ)
--Birefringence (high, low, medium, none)

If a discrepancy is noted, the verifier should confirm where in the data flow process that the error occurred. This can be achieved by reviewing the EDD that was submitted by the laboratory to confirm whether or not the data entry occurred successfully. If there is an error in the data entry to the EDD, correction to the EDD is needed. Otherwise, it is likely that the data upload procedure resulted in a misrepresentation of the data. In this case, the database manager will need to revise the upload procedure and reload the EDD.

## 9.0 REPORTING

For each field to be verified, if the data in the Data Summary Table matches the information in the hard copy laboratory job documentation, mark the appropriate field on the hard copy with a check mark. If the Data Summary Table does not match the hard copy laboratory job documentation, circle the incorrect entry on the hard copy, and note the specific discrepancy in the Data Summary Table in the "Comment" column (see **Attachment 1**). For example, "Analysis date is 1/1/11 based on hard copy, but 1/2/11 in the EDD/database".

As the verification of each selected analysis is completed, the data verifier will enter their company name and their first initial and last name (e.g., E. Smith) in the Data Summary Table in the appropriate columns.

When the verification is complete for all analyses selected, the data verifier will prepare an electronic data verification package. This package will consist of:

--A written report summarizing findings and recommendations. **Attachment 2** provides an example template for reporting PLM data verification results.
--A scanned copy of the hard copy documentation used in the verification process (which includes all verification check marks for reviewed fields).
--An electronic attachment of the Data Summary Table (which includes any data verifier comments).

The DVC will review the data verification package for accuracy and completeness. If any deficiencies are noted, the DVC will re-train verification personnel and make any corrections as necessary.

In addition to verifying all issues noted in summary report, the DVC will perform an independent data verification of 5% of the analyses verified to ensure that any potential issues have been identified correctly. The DVC will indicate in **Attachment 1** which analyses were selected for review. If any deficiencies are noted, the DVC will re-train verification personnel and make any corrections as necessary. If the DVC disagrees with the error noted in the Data Summary Table, the discrepancy will be revised, and the DVC will replace the data verifiers name with his/her name for the analysis. The summary report will be revised by the DVC to reflect any changes as needed.

The electronic data verification package will then be provided to the appropriate project database manager, or their designee, to facilitate the correction process for laboratory EDDs and/or the hand-written laboratory benchsheets by the analytical laboratory. The project database manager, or their designee, is also responsible for ensuring that any database upload issues are resolved. The verification data manager, or their designee, will record the resolution date of any corrections in the appropriate column of the Data Summary Table. Note that all of the following criteria must have been met for a PLM analysis to be considered verified:

--All necessary corrections have been made to the laboratory EDD.
--The corrected laboratory EDD has been re-submitted by the analytical laboratory to the appropriate parties (as specified in the governing project documents).
--The corrected laboratory EDD has been uploaded to the project database.
--All necessary corrections have been made to the hand-written laboratory benchsheet.
--The corrected hand-written laboratory benchsheet has been re-submitted by the analytical laboratory to the appropriate parties.
--Signatures for the data verifier, DVC, and verification data manager have been added to the verification summary report.

## 10.0 REFERENCES

ESAT (Environmental Services Assistance Team). 2012. SOP SRC-LIBBY-03: *Analysis of Asbestos Fibers in Soil by Polarized Light Microscopy.* Prepared by ESAT, Region 8.

ESAT (Environmental Services Assistance Team). 2012. SOP SRC-LIBBY-01: *Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy.* Prepared by ESAT, Region 8.

NIOSH Method 9002: Asbestos (bulk) by PLM. Manual of Analytical Methods, 4th Edition. August 1994.

**FIGURE 1**

**PLM VERIFICATION PROCESS**

**FIGURE 1. PLM VERIFICATION PROCESS**

| Personnel | Step |
|---|---|
| ***Verification Data Manager*** | 1. Create Data Summary Tables in Excel and provide to the verifier. |
| ***Verifier*** | 2. Select PLM analyses for review. |
| | 3. Locate hand-written laboratory documentation (laboratory benchsheets, point count worksheets) and print or photocopy hard copies for selected analyses. |
| | 4. Perform a consistency review utilizing the Data Summary Tables and hand-written laboratory |
| | 5. Verify that analysis-specific data were correctly transferred from the laboratory documentation to the project database (utilizing the Data Summary Tables and hand-written laboratory |
| | 6. Verify the assigned bin (if applicable ) and asbestos percent values reported in the Data Summary Tables (utilizing the hand-written laboratory documentation). |
| | 7. Verify the optical properties were correctly transferred from the laboratory benchsheet to the database (utilizing the Data Summary Tables and hardcopy hand-written laboratory benchsheets). |
| | 8. Create electronic data verification package and provide to Data Verification Coordinator. |
| ***Data Verification Coordinator (DVC)*** | 9. Review electronic data verification package and |
| | 10. Provide electronic data verification package to |
| ***Database Manager*** | 11. Correspond with laboratories to ensure appropriate corrections are made to laboratory EDDs/ benchsheets. |
| | 12. Upload corrected laboratory EDDs to project database. |
| | 13. Update project file with revised laboratory |
| ***Verification Data Manager*** | 14. Record issue resolution date in the Data Summary Table. |
| ***Verifier, DVC, and Verification Data Manager*** | 15. Sign and date verification summary report. |

PLM = polarized light microscopy
COC = chain of custody
EDD = electronic data deliverable

**FIGURE 2**

**EXAMPLE OF PLM-VE LABORATORY BENCHSHEET**

# FIGURE 2. EXAMPLE PLM-VE LABORATORY BENCHSHEET



Lab ID _____ Date Received _____ Instrument ID _____

Lab Job Number _____ Analytical Method SOP Name/Revision _____

| EPA Index ID | Tag | QC Type (NOT QC, LDS, LDC) | Lab Sample ID | Date Analyzed | Analyst Name | Stereomicroscopy Examination | | | | | Libby Amphibole | | Other Amphibole | | | Chrysotile | | Deviation (Yes/No) | Comments (see below) | Optical Properties for Libby Amphibole (see key for appropriate data inputs) | | | | | | | | Type and % of NAM Fibers (w/Optical Properties) | | | | | | | | | Non-Fibrous Matrix Materials[3] | Temp (°C) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Sample Appearance | Est. % LA | | Est. % OA and CH | | Qual (ND, Tr, <) | Conc (%) | Qual (ND, <) | Conc (%) | OA Type (AMOS, ANTH, CROC, MULTI) | Qual (ND, <) | Conc (%) | | | Habit | Fiber Color | Sign Elong. (+/-) | Pleoch. (Yes/No) | Angle Extinct. (I/P) | Ref. Index α | Ref. Index γ | Biref. | Cellulose | | Fiberglass | | Synthetic | | Other | | | | |
| | | | | | | | Qual (ND, Tr, <) | Conc (%) | Qual (ND, <) | Conc (%) | | | | | | | | | | | | | | | | | Conc (%) | OP[1] | Conc (%) | OP[1] | Conc (%) | OP[1] | Type[2] | Conc (%) | OP[1] | | |
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**Comments (Use back if needed)**

**Notes:**

1) Optical property that distiguishes fibrous material from asbestos (I = Isotropic, H = Habit, B = High Birefringence, U = Undulatory Extinction, RI = Refractive Index, O = Opaque, S = Sign of Elongation, P = Parallel Extinction)

2) T = Talc, W = Wollastonite, K = Kyanite, Ho = Hornblende, CC = Calcic Clinopyroxene, Ha = Hair, R = Rutile

3) S = Sand, C = Clay, O = Opaques, Q = Quartz, F = Feldspar, M = Mica

**FIGURE 3**

**EXAMPLE OF PLM-9002 LABORATORY BENCHSHEET**

# FIGURE 3. EXAMPLE PLM NIOSH 9002 LABORATORY BENCHSHEET



Lab ID _____ Date Received _____ Instrument ID _____

Lab Job Number _____ Analytical Method SOP Name/Revision _____

| EPA Index ID | Tag | Matrix (Bulk, Soil, Sediment) | QC Type (NOT QC, LDS, LDC) | Lab Sample ID | Date Analyzed | Analyst Name | Stereomicroscopy Examination | | | | | Tremolite-Actinolite | | Other Amphibole | | | Chrysotile | | Deviation (Yes/No) | Comments (list below) | OPTICAL PROPERTIES FOR TREMOLITE-ACTINOLITE (see key for appropriate data inputs) | | | | | | | | Temp (°C) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | Sample Appearance | Est. % Trem-Actn | | Est. % OA and CH | | | | | | | | | | | Habit | Fiber Color | Sign Elong. (+/-) | Pleoch. (Yes/No) | Angle Extinct. | Ref. Index α | Ref. Index γ | Biref. | |
| | | | | | | | | Qual (ND, <) | Conc (%) | Qual (ND, <) | Conc (%) | Qual (ND, <) | Conc (%) | Qual (ND, <) | Conc (%) | OA Type (AMOS, ANTH, CROC, UNK) | Qual (ND, <) | Conc (%) | | | | | | | | | | | |
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**Comments (Use back if needed)**

**FIGURE 4**

**EXAMPLE OF PLM-GRAV LABORATORY BENCHSHEET**

# FIGURE 4. EXAMPLE PLM-GRAV LABORATORY BENCHSHEET

Stereomicroscopic and Gravimetric Analysis of Coarse Soil

| | | | | | |
|---|---|---|---|---|---|
| **Lab ID:** | | **Instrument ID:** | | **Analysis Method SOP Version:** | |
| **Lab Job No.:** | | | | **Date Received:** | |

Calculated automatically in the "Electronic Data Entry" form. Do not enter data here.

**Page** _____ **of** _____

| EPA Index ID | Tag | Matrix (Soil, Sediment) | QC Type (Not QC, LDS, LDC) | Lab Sample ID | Date Analyzed | Analyst Name | Status 1 = Analyzed 2 = Lost 3 = Damaged 4 = Cancelled | Total Sample Weight (**g**) | | | Mass of Asbestos Particles (**mg**) | | | | | | | | | | | | | | | | Comments (see Notes below) | Temp (°C) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | Libby Amphibole (LA) Asbestos | | | | Other Amphibole (OA) Asbestos | | | | | Chrysotile (CH) Asbestos | | | | | | | | |
| | | | | | | | | Tare Weight (g) Empty Container | Mass of Sample + Container (g) | Mass of Sample (g) | LA Qual* (ND, Tr) | Tare Weight - Container (mg) | Mass of LA + Container (mg) | Mass (mg) LA | OA Qual* (ND, Tr) | OA Type** (AMOS, ANTH, CROC, MULTI) | Tare Weight - Container (mg) | Mass of OA + Container (mg) | Mass (mg) OA | CH Qual* (ND, Tr) | Tare Weight - Container (mg) | Mass of CH + Container (mg) | Mass (mg) CH | % LA | % OA | % CH | | |
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**Notes:**

*Qualifier codes: ND = No asbestos observed.
Tr = Trace levels observed but not quantified.

**OA Type codes: AMOS = Amosite
ANTH = Anthophyllite
CROC = Crocidolite
MULTI = Multiple Types

Comment Codes (user-defined):

# ATTACHMENT 1

## EXAMPLE OF DATA SUMMARY TABLES

ATTACHMENT 1A. DATA SUMMARY TABLE FOR PLM-VE VERIFICATION

| Analysis Lab ID | Lab Job Number | Analysis Method | Method SOP | Instrument ID | Samp. No | Tag | Matrix | QC Type | Lab Sample ID | Analysis Date | Analyst Name | Sample Appearance | Deviation | Comments | LA Bin | LA Conc. | OA Conc. | CH Conc. | Optical Property Data for Detected Samples | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | | | | | | | FBRCOLOR | ELONG | PLEOCH | EXTINCT | RIALPHA | RIGAMMA | BIREF | HABIT |
| | | | | | | | | | | | | | | | | | | | | | | | | | | |

ATTACHMENT 1B. DATA SUMMARY TABLE FOR PLM 9002 DATA VERIFICATION

| Analysis Lab ID | Lab Job Number | Analysis Method | Method SOP | Instrument ID | Samp. No | Tag | Matrix | QC Type | Lab Sample ID | Analysis Date | Analyst Name | Sample Appearance | Deviation | Comments | LA Conc. | OA Conc. | CH Conc. | Optical Property Data for Detected Samples | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | | | | | | FBRCOLOR | ELONG | PLEOCH | EXTINCT | RIALPHA | RIGAMMA | BIREF | HABIT |
| | | | | | | | | | | | | | | | | | | | | | | | | | |

ATTACHMENT 1C. DATA SUMMARY TABLE FOR PLM-GRAV DATA VERIFICATION

| Analysis Lab ID | Lab Job Number | Analysis Method | Method SOP | Instrument ID | Samp. No | Tag | Matrix | QC Type | Lab Sample ID | Analysis Date | Analyst Name | LA Conc. | OA Conc. | CH Conc. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | | |

# ATTACHMENT 2

## EXAMPLE OF PLM DATA VERIFICATION SUMMARY REPORT

PLM CONSISTENCY REVIEW AND DATA TRANSFER VERIFICATION REPORT

Project/Dataset Description:_____

***SUMMARY OF FINDINGS AND DATA QUALITY IMPLICATIONS***

_____

_____

_____

_____

_____

_____

_____

Recommendations for future review and verification: _____

_____

_____

_____

_____

***Data Verifier:*** _____ ***Date:*** _____

***Data Verification Coordinator:*** _____ ***Date:*** _____

***Verification Data Manager*:***_____ ***Date:*** _____

****The verification data manager acknowledges that all issues discovered during the verification process have been resolved and that the following criteria have been met:***

- All necessary corrections have been made to the laboratory EDD.
- The corrected laboratory EDD has been re-submitted by the analytical laboratory to the appropriate parties (as specified in the governing project documents).
- The corrected laboratory EDD has uploaded to the project database.
- All necessary corrections have been made to the hand-written laboratory benchsheet.
- The corrected hand-written laboratory benchsheet has been re-submitted by the analytical laboratory to the appropriate parties.

***PLM-VE SELECTION AND CONSISTENCY REVIEW RESULTS***

Summary of available analyses for date range specified –

| Analyst, Lab | Number of PLM-VE Analyses | | | Number of PLM-VE Analyses Selected for Review | | |
|---|---|---|---|---|---|---|
| | Detect | Non-Detect (Bin A) | Total | Detect | Non-Detect (Bin A) | Total |
| Analyst #1, Lab Name | | | | | | |
| Analyst #2, Lab Name | | | | | | |
| … | | | | | | |
| | | | | | | |
| Total | | | | | | |

| | Goal | Actual |
|---|---|---|
| Selected Total | _____ | _____ |
| Selected Detects | _____ | _____ |
| Selected Non-Detects | _____ | _____ |

Detailed summary of bench sheet consistency review –

Number of analyses reviewed: _____ (_____% of total analyses selected)

If not all analyses could be reviewed, provide a brief explanation for why: _____

_____

Number of analyses with recording issues identified: _____ (_____% of total analyses reviewed)

Types of recording issues identified (indicate the number of analyses):

_____ Qualifier codes are inconsistent with the method reporting rules

_____ Reported value does not use correct binning category

_____ If recorded, OA structure types are not identified

_____ Optical properties are not recorded for reported LA fibers

_____ At least one optical property was not recorded for non-asbestos fibrous material

Do the recording issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

_____

***PLM-9002 SELECTION AND CONSISTENCY REVIEW RESULTS***

Summary of available analyses for date range specified –

| Analyst, Lab | Number of PLM-9002 Analyses | | | Number of PLM-9002 Analyses Selected for Review | | |
|---|---|---|---|---|---|---|
| | Detect | Non-Detect | Total | Detect | Non-Detect | Total |
| Analyst #1, Lab Name | | | | | | |
| Analyst #2, Lab Name | | | | | | |
| … | | | | | | |
| | | | | | | |
| Total | | | | | | |

| | Goal | Actual |
|---|---|---|
| Selected Total | _____ | _____ |
| Selected Detects | _____ | _____ |
| Selected Non-Detects | _____ | _____ |

Detailed summary of bench sheet consistency review –

Number of analyses reviewed: _____ (_____% of total analyses selected)

If not all analyses could be reviewed, provide a brief explanation for why: _____

_____

Number of analyses with recording issues identified: _____ (_____% of total analyses reviewed)

Types of recording issues identified (indicate the number of analyses):

_____ Qualifier codes are inconsistent with the method reporting rules

_____ If recorded, OA structure types are not identified

_____ Optical properties are not recorded for reported TREM-ACTN fibers

_____ At least one optical property was not recorded for non-asbestos fibrous material

Do the recording issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

_____

***PLM-GRAV SELECTION AND CONSISTENCY REVIEW RESULTS***

Summary of available analyses for date range specified –

| Analyst, Lab | Number of PLM-Grav Analyses | | | Number of PLM-Grav Analyses Selected for Review | | |
|---|---|---|---|---|---|---|
| | Detect | Non-Detect | Total | Detect | Non-Detect | Total |
| Analyst #1, Lab Name | | | | | | |
| Analyst #2, Lab Name | | | | | | |
| … | | | | | | |
| | | | | | | |
| Total | | | | | | |

| | Goal | Actual |
|---|---|---|
| Selected Total | _____ | _____ |
| Selected Detects | _____ | _____ |
| Selected Non-Detects | _____ | _____ |

Detailed summary of bench sheet consistency review –

Number of analyses reviewed: _____ (_____% of total analyses selected)

If not all analyses could be reviewed, provide a brief explanation for why: _____

_____

Number of analyses with recording issues identified: _____ (_____% of total analyses reviewed)

Types of recording issues identified (indicate the number of analyses):

_____ Qualifier codes are inconsistent with the method reporting rules

_____ If recorded, OA structure types are not identified

_____ Asbestos concentration calculation is incorrect or inputs are missing

Do the recording issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

_____

***DATA TRANSFER VERIFICATION RESULTS***

Number of analyses verified[1]: _____ (_____% of total analyses selected)

Number of analyses with data transfer issues identified: _____ (_____% of total analyses verified)

Examples of data transfer issues identified:

_____ Incorrect/missing information on analysis details (e.g., lab job number, analysis date)

_____ Results for PLM-9002 are not reported as TREM-ACTN

_____ Results for PLM-VE and PLM-Grav are not reported as LA

_____ Reported value for PLM-VE does not use correct binning category

_____ Optical properties PLM-VE and PLM-9002 are missing or inconsistent

Do the data transfer issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

_____

Comments: _____

_____

_____

_____

***ISSUE RESOLUTION AND STATUS***

_____

_____

_____

_____

_____

_____

[1] Only those analyses that have passed the bench sheet consistency review are included in the data transfer verification.

# Libby Asbestos Superfund Site
# Standard Operating Procedure
# Air Sample Collection

**Prepared by:** _____ Date: 4/9/12
CDM Smith

**Approved by:** _____ Date: 4/9/12
EPA Region 8

| Revision No. | Date | Reason for Revision |
|---|---|---|
| 0 | 4/6/12 | Includes elements from previous Libby SOPs EPA-LIBBY-01 (*Sampling of Asbestos Fibers in Air*) and CDM-LIBBY-14 (*Stationary Air Sample Collection*) to present a unified, updated approach to collecting Libby Site air samples. |

## 1.0 Objective

The objective of this standard operating procedure (SOP) is to establish baseline requirements, procedures, and responsibilities for performing air sampling by the U.S. Environmental Protection Agency or its contractors in support of the Libby Asbestos Superfund Site (Libby Site). Modifications to this SOP may be detailed in governing documents referencing this SOP.

## 2.0 Background

### 2.1 Definitions

DryCal® – A type of air flow meter used at the Libby Site as a primary flow standard for calibrating rotameters.

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

Inert tubing – Inert tubing (e.g., Tygon®) is used in the sampling train to connect the outflow end of the sample cassette to the sampling pump.

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

Personal air sample – An air sample collected within the breathing zone of the individual being sampled.

Phase contrast microscopy (PCM) sample cassette – A 25-millimeter (mm), three-piece cassette with a 50mm electronically conductive extension cowl loaded with a 0.8 micron pore size, mixed cellulose ester (MCE) filter.

Rotameter – A type of air flow meter.

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

Sampling pump – A device used to pull air through a filter medium at an established rate. Common pumps used at the Libby Site are the SKC Airchek Sampler, Model 224-PCXR4 (low-volume battery-powered sampling pump) and the Gast 1532 rotary vane pump (high-volume alternating current sampling pump).

Sampling stands – Telescoping tripods designed to hold sample cassettes at a desired height and isolate them from the vibrations of the sampling pump.

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

Stationary (ambient) air sample – An air sample collected from a fixed location.

### 2.2 Discussion

Air sampling generally consists of using sampling pumps to draw air over a sample filter for a pre-determined volume in order to measure airborne quantities or concentrations of asbestos fibers. Air sample data serves many purposes at the Libby Site – samples may be investigatory in nature, be used to determine compliance with Occupational Safety and Health Administration requirements, or measure attainment of action levels or cleanup criteria established by EPA to evaluate response actions. Documents governing air sampling activities shall specify the intended use of the air sampling data.

## 3.0 Responsibilities

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

Team Leader (TL) – The TL is responsible for overseeing air sample collection processes as described in this SOP. The TL is also responsible for checking all work performed and verifying that the work satisfies the objectives of the data collection effort. It is also the responsibility of the TL to communicate the need for any deviations from the SOP with the appropriate personnel, and document the deviation using a Libby Field Record of Modification Form.

Field Team Members – Field team members performing air sampling are responsible for adhering to the procedures contained in this SOP. The field team members should have limited discretion with regard to collection procedures but should exercise judgment regarding the exact locations of samples within the specified sampling area. Field team members are also responsible for communicating any sample collection issues (e.g., equipment failure) to the TL for the purpose of troubleshooting and information-sharing with other field team members.

# 4.0 Equipment

The following equipment is required for Libby Site air sampling:

- Sampling pump
- PCM sample cassettes
- Sampling stands
- Inert tubing
- Rotameter
- DryCal®
- Sample labels
- Pint-sized plastic zip-top bags
- Cooler or other rigid container
- Custody Seals
- Small standard screwdriver (used to adjust the flow rate in low-volume pumps)
- Field logbooks
- FSDSs
- Indelible blue or black ink pens

# 5.0 Procedures

Prior to conducting work at any Libby work site, health and safety procedures, as specified in the governing health and safety plan, will be reviewed and the appropriate personal protective equipment donned.

## 5.1 Preparation

### 5.1.1 Calibrating the Rotameter with an Electronic Calibrator

Rotameters used for pump calibration are calibrated to a primary flow standard on a quarterly basis. The primary flow standard in use at the Libby Site is the DryCal® DC-Lite, manufactured by Bios International Corporation. Procedures for rotameter calibration with the DC-Lite flow meter are as follows:

1. Obtain the actual temperature and pressure in Libby, Montana from the local National Oceanic and Atmospheric Administration (NOAA) weather station. Record actual temperature and pressure in the fields provided on the Precision Rotameter Calibration Data Sheet (Attachment 1).
2. Set up the calibration train as shown in EPA SOP #2015 (EPA 1994), Figure 4, with the sampling pump, rotameter, and primary flow meter (Attachment 2).
3. The rotameter will be held perpendicular to the plane of the table in a vertical position.
4. Turn the DC-Lite and sampling pump on.
5. Turn the flow adjustment screw or knob on the pump until the desired flow rate is attained.
6. Calibrate rotameter to desired ball reading, as read from the middle of the flow ball, with a sampling pump and sample cassette in-line. The cassette used for calibration must be the same type and from the same lot of sample cassettes that will be used for sampling. Record value in the ball reading column on the rotameter calibration data sheet.

7. Check adjusted flow rate of sampling pump to the DC-Lite flow calibrator primary flow standard. Ten repetitive flow measurements will be averaged and that result recorded in the flow rate column for the selected interval.
8. Repeat this process at 10 intervals over the range of the precision rotameter.
9. Input data into rotameter calculation sheet to generate the corrected flow rate.

### 5.1.2 Flow Rates and Sample Volume

In general, air samples will be collected using flow rates ranging between 1.0 and 10.0 liters per minute (L/min), with a minimum total sample volume of 1,200 liters. Flow rates will be set at the discretion of the field team member in order to capture, at a minimum, 80 percent (%) of the workday. The sampling pump will provide a non-fluctuating air flow through the filter, and will maintain the initial volume flow rate to within ±10% throughout the sampling period. If at any time the measurement indicates that the flow rate has increased or decreased by more than 10% of the set flow rate, sample collection will cease and the sample will be voided.

In no case will a sample be collected at a flow rate lower than 1.0 L/min, since the linear flow velocity would fall below 4 centimeters per second (cm/sec), which is the minimum velocity specified by the International Organization for Standardization (ISO) method 10312 (ISO 1995) that is used for Libby project air samples.

As samples are initially collected during the sampling event and analyzed, flow rates and sample times may be adjusted to ensure the loading on the sample filter facilitates reaching the required sensitivity goals (i.e., to prevent filter overloading). Filter loading is discussed in more detail in Section 5.2.2 of this SOP.

### 5.1.3 Calibrating the Sampling Pump with a Rotameter

Each sampling pump will be calibrated before and after each sampling event with a primary or secondary calibration device as described below. This is to ensure that each sampling pump is operating to project requirements as stated in Section 5.2.

The procedures used for sampling pump calibration are as follows:

1. Set up the calibration chain as shown in EPA SOP #2015 (EPA 1994), Figure 5 (Attachment 3) using a rotameter, sampling pump, and a representative sample cassette. The sample cassette to be used for sampling is installed between the pump and the calibrator.
2. To set up the calibration train, attach one end of tubing to the sample cassette base, then attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the sample cassette cap to the rotameter.
3. The flow meter should be held in a vertical position.
4. Turn the sampling pump on.
5. Turn the flow adjustment screw or knob on the manifold regulating air flow to the samples until the middle of the float ball on the rotameter is lined up with the pre-calibrated flow rate value.

Each rotameter used for field calibration will be transported to and from each sampling location in a sealed zip-top plastic bag.

### 5.1.4 Collecting Multiple Samples using One Pump

A sampling pump equipped with more than one manifold may be used to collect more than one sample at a time. In the case two samples will be collected from one pump, calibration must be checked after each alteration of the flow regulators. For example: Turn the knob on (manifold A) until the middle of the float ball on the rotameter is at the desired flow rate value. Turn the knob on (manifold B) until the middle of the float ball on the rotameter is at the desired flow rate value. Verify the calibration of (manifold A), adjust as required. This process must be repeated until both (manifold A) and (manifold B) are at the desired flow rate.

## 5.2 Operation

### 5.2.1 Air Sampling Locations

If not specifically discussed in the governing document referencing this SOP, the location of each stationary air sample will be determined by field personnel based on site-specific conditions (e.g., personnel breathing zone, near equipment, soil excavation boundaries, etc.).

### 5.2.2 Sample Collection Protocol

Each air sample will be collected according to the following procedures:

1. Place a sample label on the sample cassette. Place the corresponding sample label on the FSDS.
2. Determine proper sample location. For a fixed air monitor, this will generally be at a height that represents the breathing zone of the potentially exposed population (e.g., 4-6 feet above the ground or floor). For personal air monitoring, the cassette will typically be placed on the lapel just below the face of the individual being monitored.
3. Set up the sampling train and attach the air intake hose to the sample cassette base. Follow calibration procedures listed in Section 5.1.3. The sample cassette will be positioned such that it is held facing downwards at an approximate 45 degreee angle to avoid any particles entering the filter by precipitation. Remove the sample cassette cap and turn the sampling pump on.
4. Record all pertinent information on the FSDS.
5. Unless otherwise specified in the governing document referencing this SOP, check the sampling pump at a minimum of every 4 hours. If the sample filter darkens in appearance or if loose dust is observed inside the cassette, the sample period will be terminated and the remaining steps below followed to complete collection of the sample. The loading observations will be noted on the FSDS in the comment section.
6. At the end of the sampling period, attach the sample cassette cap with the flow meter device inserted according to the appropriate sampling train. Do not remove the sampling cassette from the sampling train.
7. Collect the post-sampling flow rate with the rotameter. Turn the pump off.
8. Record the stop date and time.
9. Record the post-sampling flow rate.

10. Remove the tubing from the sample cassette. Holding the sample cassette upright, replace the inlet plug on the sample cassette cap and the outlet plug on the sample cassette base. Do not put sample cassettes in shirt or coat pockets as the filters can pick up fibers.
11. Sign and date custody seal and wrap it around both ends of the sample cassette. The custody seal should not cover the sample label.
12. Place each sample cassette in a half-quart sized plastic zip-top sample bag. Each bag should be marked with indelible ink indicating the sample number.

#### 5.2.3 Pump Failure Procedures

If a sampling pump faults prior to the total desired run time, the following procedures will be used:

1. Record the time of the observed pump fault in the comments section of the FSDS.
2. If using a SKC low-volume pump, record the total sample time (in minutes) from the pump counter and note accordingly in the comments section of the FSDS sheet, then add total minutes collected to the start time and document the actual stop time in the stop time section of the FSDS.
3. If no minutes appear on the pump counter, void the sample and recollect as specified by the governing document referencing this SOP, or as directed by the TL or health and safety representative.
4. If time allows, change out the pump and restart sampling. Turn the sampling pump back on and calibrate as required (Section 5.1.3) until desired sample volume requirements are met.

### 5.3 Post-operation

Non-disposable air sampling equipment will be decontaminated according to instructions provided in the governing document referencing this SOP. In general, sampling pumps and tubing will be wet-wiped prior to and following sample collection. Dispose of the wipes as IDW.

Sample custody, and any packaging and shipping requirements, for Libby Site samples will be specified in the governing document referencing this SOP. Project documentation (field logbook, FSDS) will be retained according to data management requirements and/or in the project file.

## 6.0 Restrictions/Limitations

High levels of dust or other suspended particulates in air may clog or overload the sample filter and reduce the ability to observe and characterize asbestos fibers on the filter. Precautions should be taken to avoid unnecessary sources of dust emissions or use of aerosol sprays. Sampling requirements (e.g., flow rate, sampling time) may need to be adjusted accordingly to avoid filter overload.

## 7.0 Quality Assurance/Quality Control

Quality assurance/quality control (QA/QC) for activities described in this SOP will be attained through a variety of processes, including, but not limited to, the items discussed below. Additional QA/QC requirements, such as audits or field assessments, will be addressed in the governing document referencing this SOP.

### 7.1 Training

Every effort will be made to ensure consistency in collecting air samples in support of the Libby Site. Consistency will be achieved to the extent possible through proper training, using designated field staff, and providing TL oversight. Any deficiencies or inconsistencies in implementing this SOP noted by the TL will require re-training of the field team.

### 7.2 Equipment Maintenance

The manufacturer's instructions regarding operating procedures and maintenance will be reviewed prior to equipment use. Equipment and instrumentation will be utilized in accordance with manufactures instructions.

### 7.3 Field Quality Control Samples

The field quality control (QC) samples for air sampling at the Libby Site typically consist of lot blanks and field blanks. Refer to the governing document referencing this SOP for field QC sample collection requirements and acceptance criteria.

## 8.0 References

U.S. Environmental Protection Agency. 1994. Asbestos Sampling, Standard Operating Procedure #2015, Revision 0.0. November 17.

International Organization for Standardization. 1995. Ambient Air – Determination of Asbestos Fibers – Direct Transfer Transmission Electron Microscopy Method. ISO 10312:1995(E).

# Attachment 1

## Libby Asbestos Project
## Precision Rotameter Calibration Data Sheet

Project Number: _____

Calibration Date: _____ Calibrated By: _____

Odometer ID: _____ Primary Standard ID: _____

Actual Temp. (ºF): _____ Actual Pressure (in. Hg): _____

°F = degrees Fahrenheit
in. Hg = inches mercury

| | Ball Reading =Y (mid-ball) | Flow Rate = $X_1$ (L/min) |
|---|---|---|
| **1.** | _____ | _____ |
| **2.** | _____ | _____ |
| **3.** | _____ | _____ |
| **4.** | _____ | _____ |
| **5.** | _____ | _____ |
| **6.** | _____ | _____ |
| **7.** | _____ | _____ |
| **8.** | _____ | _____ |
| **9.** | _____ | _____ |
| **10.** | _____ | _____ |

**Rotameter Calibration Procedure:**

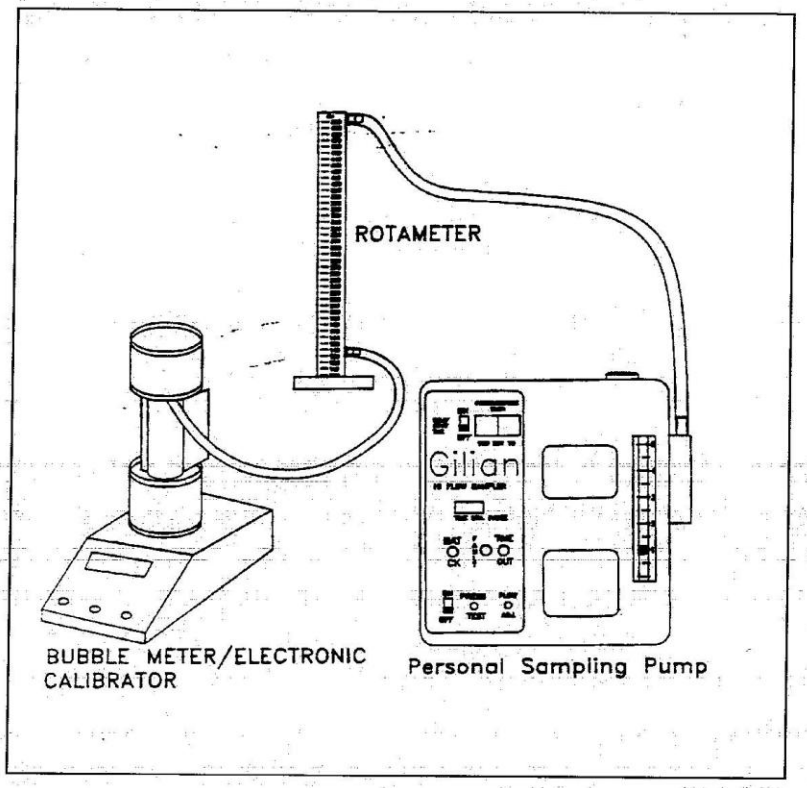
1. Obtain the actual temperature and actual pressure in Libby, MT from the local NOAA weather station. Record the actual temperature and actual pressure in the fields provided above.
2. Calibrate rotameter to desired ball reading with a sampling pump and cassette in-line. The cassette must be the same type and from the same lot of cassettes that will be used for sampling. Record the value in the “Ball Reading” column above.
3. Check the adjusted flow rate of the sample pump to the DryCal® primary flow standard. Ten repetitive flow measurements will be averaged and that result recorded in the “Flow Rate” column for the selected interval.
4. Repeat this process at 10 intervals over the range of the precision rotameter. Input data into rotameter calculation sheet to generate the corrected flow rate.

# Attachment 2

**APPENDIX B (Cont'd)**

Figures

FIGURE 4. Calibrating a Rotameter with a Bubble Meter




16

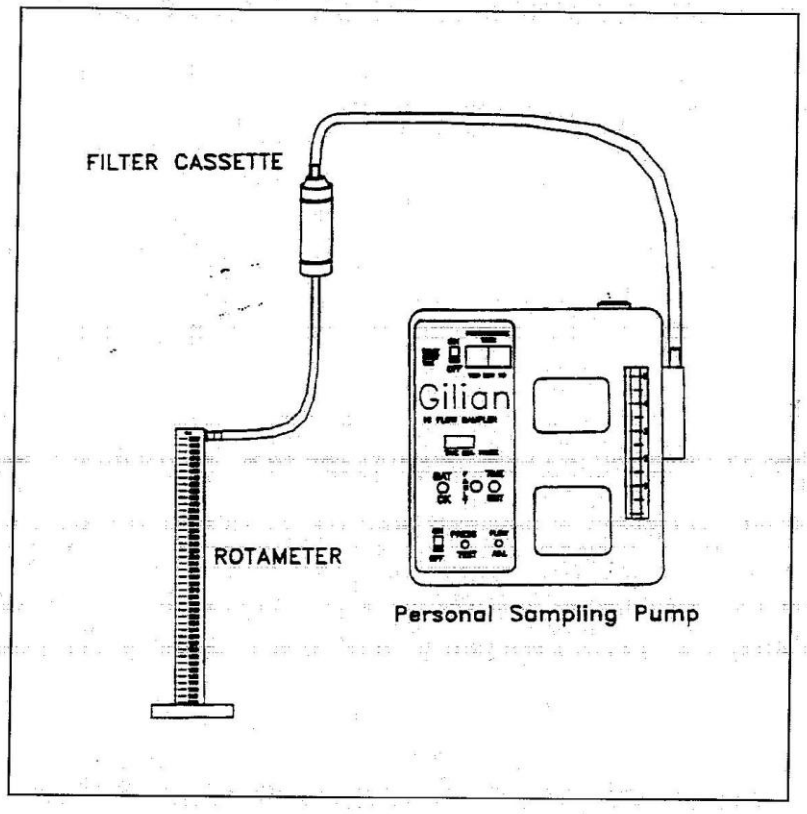
Figure obtained from EPA SOP #2015 (EPA 1994).

# Attachment 3

**APPENDIX B (Cont'd)**

Figures

FIGURE 5. Calibrating a Sampling Pump with a Rotameter




17

Figure obtained from EPA SOP #2015 (EPA 1994).

# SOIL SAMPLE PREPARATION

**SYNOPSIS:** This is a standardized method for the preparation of fine and coarse-grain soil samples collected at the Libby Asbestos Superfund Site for asbestos analysis at an approved laboratory.

**APPROVALS:**

USEPA Region 8

Dania Zinner — Signature

12-6-12 — Date

Dania Zinner — Print Name

RPM — Title

ESAT Region 8

Signature

12-5-12 — Date

Mark McDaniel — Print Name

Team Lead — Title

## RECORD OF CHANGES

| Revision | Date | Principal Changes and Author |
|---|---|---|
| 0 | Unknown | This SOP was originally prepared by ISSI Consulting Group. ISSI is no longer in existence, and finalization of the SOP was performed by William Brattin at Syracuse Research Corporation (SRC). |
| 1 | 01/07/2000 | Incorporated sieving to the sample preparation |
| 2 | 07/12/2000 | Revision in sieve size and other minor edits |
| 3 | 05/07/2002 | Incorporated minor edits |
| 4 | 08/01/2002 | Modified sieving procedure and added grinding step |
| 5 | 03/06/2003 | Incorporated modifications to the procedure and documentation requirements |
| 6 | 03/24/2003 | Incorporated modifications to the log sheets to conform with electronic data storage requirements and added grinder blank requirements |
| 7 | 08/05/2003 | Incorporated modifications to drying and sample storage procedures |
| 8 | 05/04/2004 | Incorporated modifications to drying batch size and recording of preparation information |
| 9 | 05/14/2007 | Incorporated modifications so as to expand use to other Operable Units (removed references to OU4/CSF, changed Index ID to Sample ID). Repaired formatting. Removed reference to missing Figure 1. Added optional use of electronic logs. Oven temperature set to 90 ± 10 degrees C. Lowered inventory batch size from ~120 to ~50 samples so that one inventory batch can fit in one tub. Designated drying batch as one batch per oven (~20 samples). Allowed for optional use of disposable drying pans. Removed direction to NOT move grinding plates during decontamination (new BICO design allows plates to be separated for decontamination without adjusting gap). Ovens will be calibrated daily. *[Note: Revision 9 was as unsigned version that reflects changes made at the Troy Sample Preparation Facility. Some of the changes in Revision 9 are retained in Revision 10, below].* |
| 10 | 12/06/2007 | Incorporated modifications so as to expand use to other Operable Units. Designated drying batch as ~20 samples. Allowed for optional use of disposable drying pans. Allowed alternative methods for decontamination of plate grinder. Clarified and modified QC requirements. General editing for clarity. |
| 11 | 07/27/2012, 11/15/2012, 12/05/2012 | Entire SOP review and update provided by ESAT Region 8, which includes procedures for equipment calibration. |

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## LIST OF ATTACHMENTS

Attachment 1: Sample Drying Bench Sheet
Attachment 2: Sample Preparation Bench Sheet

## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to standardize the methods used to prepare soil samples collected at the Libby Asbestos Superfund Site (referred to as the Libby Site from this point forward) for the analysis of asbestos content by an approved laboratory. This SOP is specifically intended for application at the Libby Site and focuses on homogenizing soils and soil-like materials in order to produce equal fractions of the original sample with reproducible results.

## 2.0 SCOPE AND APPLICATION

Soil samples from the Libby Site are processed at the Environmental Services Assistance Team (ESAT) Region 8 Troy Sample Preparation Facility (SPF) before submittal to the laboratory for analysis. This process separates the coarse fraction of the soil from the fine fraction. The fine fraction constitutes all material passing through a ¼-inch sieve. The fine fraction is homogenized and ground to a maximum particle size of approximately 250 microns (µm). This fine fraction is further sub-divided into four fractions using a riffle splitter. One or more of these fractions is then submitted to an approved and accredited laboratory for polarized light microscopy (PLM) analysis.

## 3.0 RESPONSIBILITIES

3.1 It is the responsibility of the SPF supervisor to ensure that all preparation, quality assurance (QA) and quality control (QC) procedures are performed in accordance with this SOP and to identify and take appropriate corrective action to address any deviations that may occur during sample preparation.

3.2 The ESAT Team Manager, QA Coordinator (QAC), and/or SPF Lead will communicate with project managers at the United States Environmental Protection Agency (EPA; also referred to as the client), or their designate, any situations where a modification to or deviation from the SOP may be useful or necessary. ESAT must receive approval from the EPA for any deviation or modification from the SOP before incorporating any such deviation or modification into the sample preparation process (refer to Section 8.2).

3.3 All SPF personnel are responsible for reading and understanding the SPF-specific Health and Safety Plan (HASP) and performing all tasks in accordance with the requirements of the HASP.

## 4.0 METHOD DESCRIPTION

Soil samples received at the SPF are dried in a laboratory oven, and then split into a preparation sample and an archive sample. The preparation sample is sieved to separate coarse material (>¼-inch) from fine material (<¼-inch). The fine material is ground to a particle size of less than 250 µm, and this fine ground material is split into several aliquots. This grinding step is needed to achieve a reasonable degree of homogeneity in the sample, and to allow for preparation of slides for PLM analysis. The coarse fraction (if any) and one aliquot of the fine ground material are then sent to an approved analytical laboratory for PLM analysis. Fine-ground samples are analyzed according to the current version of SOP SRC-LIBBY-03, *Analysis of Asbestos Fibers in Fine Soil*

*by Polarized Light Microscopy*, and coarse samples are analyzed according to the current version of SOP SRC-LIBBY-01, *Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy*. Sample fractions that are not sent to an analytical lab for analysis are stored in an archive facility currently maintained by SPF personnel. Fractions are tracked both on paper and electronically.

## 5.0 ACRONYMS

| | |
|---|---|
| ACM | Asbestos Containing Material |
| EPA | United States Environmental Protection Agency |
| ESAT | Environmental Services Assistance Team |
| HASP | Health and Safety Plan |
| HAZWOPER | Hazardous Waste Operations and Emergency Response |
| HEPA | High Efficiency Particulate Air |
| LA | Libby Amphibole |
| NIST | National Institute of Standards and Technology |
| OSHA | Occupational Safety and Health Administration |
| PE | Performance Evaluation |
| PLM | Polarized Light Microscopy |
| PPE | Personal Protective Equipment |
| QA | Quality Assurance |
| QAC | Quality Assurance Coordinator |
| QC | Quality Control |
| QMP | Quality Management Plan |
| SOP | Standard Operating Procedure |
| SPF | Troy Sample Preparation Facility |
| SRC | Syracuse Research Corporation |
| USGS | United States Geological Survey |

## 6.0 HEALTH AND SAFETY

6.1 Follow general laboratory health and safety policies and regulations in the HASP, Chemical Hygiene Plan, or equivalent.

6.2 All sample handling and preparation activities (drying, splitting, sieving, grinding, etc.) are performed within the SPF Work Zone, which is fitted with a negative pressure, ventilated hood with an operating High Efficiency Particulate Air (HEPA) filtration system. A sample container should only be open within the Work Zone at the following times: when the sample is inside of the sample preparation hood, when transferring a sample from the ventilation hood to the oven, or when transferring a sample from the oven to the grinding hood. Appropriate personal protective equipment (PPE) must be worn at all times within the Work Zone.

## 7.0 CAUTIONS

After processing each sample, thoroughly decontaminate all equipment and work surfaces in order to prevent cross-contamination between samples.

## 8.0 GENERAL LABORATORY PRACTICES

8.1 QA Program

8.1.1 The SPF operates under a QA program appropriate to the type, range, and volume of work it performs.

8.1.2 It is the responsibility of SPF personnel to read, understand, and follow the ESAT Region 8 Quality Management Plan (QMP). Additional QA/QC requirements specific to the SPF are described in Section 18.0.

8.1.3 All work is performed at a permanent location. The SPF is able to carry out all preparation, calibration, and daily QA/QC activities independently, and at one location. There are no remote or sub-facilities where preparation work is performed.

8.2 Documenting SOP Modifications

8.2.1 Any deviation from the SOP shall be documented in a laboratory modification form. Additionally, when there is reason to suspect a departure from the SOP has affected the result or validity of data provided to the client, the client must be notified of the nature of the departure from the SOP and informed about the possible effect on the result or validity of the analysis. The course of action taken to keep the departure from recurring must also be discussed with the client.

## 9.0 PERSONNEL QUALIFICATIONS

9.1 Personnel performing sample preparation activities must read and understand the HASP and all associated SOPs. In addition, personnel must complete the 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training and the required OSHA 8-hour Refresher courses. Additional training may be identified prior to project implementation and will be administered prior to any individual beginning work at the SPF.

9.2 The health and safety records for all personnel, including HAZWOPER 40-hour and 8-hour certificates must be kept in a central location and available at all times.

## 10.0 EQUIPMENT AND SUPPLIES

The SPF must be equipped with the following instrumentation, hardware, software, and all other materials and supplies required to perform this SOP. All equipment must be properly maintained and calibrated (as appropriate) prior to use.

- General purpose laboratory oven capable of maintaining a constant temperature of approximately 90$^{o}$C
- Analytical balance capable of measuring in a range of 0.1 g to at least 2,000 g
- Weight set, traceable to National Institute Standards and Technology (NIST)
- Riffle splitter with ¾-inch chutes
- Plate grinder with plates adjustable from ¼-inch to approximately 250 µm

- Wet/dry vacuum with HEPA filtration
- Compressed air
- Metal scoop or spoon (plastic scoops or spoons are not acceptable)
- ¼-inch metal sieve and catch pan (plastic sieves and pans are not acceptable)
- Shims used for grinder plate calibration
- NIST traceable certified micrometer
- 60-mesh (250 µm) sieve and 200-mesh (74 µm) sieve
- Asbestos-free quartz sand
- Drying pans (disposable aluminum baking pans)
- HEPA-filtered hood, a class 1 biohazard hood, or glove box with continuous airflow (negative pressure)
- Vaneometer
- Re-sealable Poly Bags, 4-mil – sizes 4x6 inch and 10x13 inch (these two sizes are standard; however, a larger 4-mil poly bag may used if a larger master sample bag is needed)
- Disposable, powder-free examination gloves (nitrile or latex)
- Half-face respirator with disposable P100 cartridges
- Safety glasses or goggles (Z-87 rated) with side shields
- Tyvek coveralls with attached hood/boots
- Additional PPE required by the SPF-specific HASP
- Sample Drying bench sheets (provided in Attachment 1)
- Sample Preparation bench sheets (provided in Attachment 2)
- Equipment maintenance/calibration logbooks, document controlled
- Self-adhesive sample labels
- Asbestos containing material (ACM) waste bags
- Indelible marking pen
- Water in spray bottles and paper towels (for wet wiping)

## 11.0 SOIL STORAGE

Upon receipt at the SPF, samples are grouped into an inventory batch and assigned an inventory batch number. This number is an identifier in the following format: 12-1014, where 12 = two-digit calendar year (as in 2012) and 1014 = four-digit consecutive number, starting with 0001. Whenever soil samples are not being processed, they are stored in plastic bins or shipping boxes/coolers. The samples do not require refrigeration but must be kept in an orderly, clean fashion. All bins will be assigned a bin identification number, or Bin ID, which is a four-digit consecutive number starting with 0001. The Bin ID is displayed on a prominent hanging tag. Bins will be arranged on labeled shelves by the Bin ID for easy retrieval. All bins will also be labeled with one or more inventory batch numbers. Bin information is tracked by the sample coordinator in an Excel file, which indicates the Bin ID, bin contents, and its physical location within the SPF.

## 12.0 BULK SOIL DRYING

### 12.1 Equipment Verification Checks

12.1.1 Samples will be weighed prior to and following drying activities. A verification check of the analytical balance is performed each day when samples are loaded into or unloaded from the oven (note that a verification check is performed on the balance for other soil processing purposed as well, even if samples are not being dried on a particular day). Before weighing samples, perform the verification check using Class 6 weights (equivalent to Class S-1 weights) and record the results, any required maintenance, and the balance number in the Analytical Balance Verification and Maintenance Logbook.

12.1.2 All drying activities will be performed in a HEPA-filtered hood, a class 1 biohazard hood, or glove box with continuous airflow (negative pressure). Prior to loading the oven, use a vaneometer to verify that the hood's ventilation system is operating properly, and record the results and any required maintenance in the Ventilation Hood Verification and Maintenance Logbook. Verification of the hood's ventilation system is performed daily.

12.1.3 A HEPA vacuum will be used to decontaminate the oven following the removal of dried samples. A verification check is performed on the HEPA vacuum daily prior to drying activities. All system checks, required maintenance and the vacuum number will be recorded in the HEPA Vacuum Verification and Maintenance Logbook.

12.1.4 An oven temperature verification check will be performed daily during periods of operation. Oven temperature verification checks and any required maintenance will be documented in the Oven Temperature Verification and Maintenance Logbook.

### 12.2 Drying Procedure

12.2.1 Group samples into drying batches of approximately 20 samples per batch. Assign each batch a drying batch number, and record this number on the Sample Drying bench sheet, as well as the number of the oven used to dry the samples.

12.2.2 Include one preparation blank per oven. See Section 18.1 for more details regarding preparation blanks.

12.2.3 Set the oven temperature to 90°C (±l0$^{o}$C). For every drying batch, check the oven temperature to verify that proper temperature has been reached and document the start date/time and temperature on the Sample Drying bench sheet.

12.2.4 Transfer each sample to be dried from its 4-mil poly bag into a clean drying pan.

12.2.4.1 Prior to drying each sample, record the starting sample mass to the nearest 0.1 g on the Sample Drying bench sheet (Attachment 1).

12.2.4.1.1 Place the empty drying pan on the analytical balance and tare the balance to zero. Then, pour the sample into the drying pan and record the weight to the nearest 0.1 g. By taring the balance to zero, the recorded weight is only that of the sample and not the sample plus the drying pan.

12.2.4.2 Each sample must be transferred to its respective drying pan under the HEPA-filtered hood.

12.2.4.3 Label each drying pan with the Sample ID of the sample. Place each sample in the oven.

12.2.5 Leave the samples in the oven until completely dry (potentially 24-48 hours). Verify that each sample is dry by squeezing a portion of the soil with a freshly gloved thumb and forefinger to test the cohesiveness.

12.2.6 Turn off the oven and allow the samples to cool in the oven.

12.2.7 Once the samples are cooled, unload each sample and transfer to a clean, disposable aluminum pan labeled with the Sample ID.

12.2.7.1 The weight of each dried sample must be recorded to the nearest 0.1 g on the Sample Drying bench sheet (Attachment 1).

12.2.7.1.1 Place the empty aluminum pan on the analytical balance and tare the balance to zero. Then, transfer the sample into the new aluminum pan and record the weight to the nearest 0.1 g. By taring the balance to zero, the recorded weight is only that of the sample and not the sample plus the aluminum pan.

12.2.7.2 All samples transfers should be made in the HEPA-filtered hood to prevent potential exposure to asbestos fibers that might be released from the sample.

12.2.8 Once all information on the Sample Drying bench sheet is completed (including the technician's initials and the date) a second technician must perform a QC check of the information, and initial and date the bench sheet.

12.2.9 Then, each sample can either be poured into a clean 4x6 inch 4-mil poly bag (identified with the Sample ID) or personnel can proceed directly with sample processing (see Sections 13 through 16).

12.3 Decontamination

Decontaminate all equipment used (drying pans, the inside of the hood, and the inside of the drying oven) using the HEPA vacuum and wet wiping all surfaces before loading a new batch for drying.

## 13.0 DIVISION OF ARCHIVE AND PREPARATION SAMPLES

All dried samples are mixed and split into two portions: one portion is held in archive, and the second portion is prepared for shipment to an approved analytical laboratory for asbestos analysis. Additional splitting may be necessary to reduce the portion size to a size appropriate for grinding. The sections below describe the sample splitting procedure.

13.1 Equipment Calibration

All splitting, sieving, and grinding activities will be performed in a HEPA-filtered hood, a class 1 biohazard hood, or glove box with continuous airflow (negative pressure). Prior to any splitting, sieving, or grinding activities, use a vaneometer to verify that the hood’s ventilation system is operating properly, and record the results and any required maintenance in the Ventilation Hood Verification and Maintenance Logbook.

13.2 Procedure for Sample Splitting

Splitting must be performed in the HEPA-filtered hood to prevent potential exposure to asbestos fibers that might be released from the sample. Samples will be divided using the following procedures:

13.2.1 Place the cooled, re-bagged samples in the hood, and knead the contents of the bag to break up any soil clumps.
13.2.2 Place one collection pan on each side of the riffle splitter. Pour the sample through the splitter in order to divide the sample into two equal sub-parts.
13.2.3 After splitting, set aside one portion for sample preparation (preparation procedures described below). If the mass of the portion for preparation is larger than about 200 grams, split the preparation sample again so that ¾ of the original sample will be archived and ¼ will be set aside for processing.
13.2.4 Place the remaining portion(s) into a clean, 4-mil poly bag, re-bag the sample in another clean, 4-mil poly bag, and store as an archive sample in the event additional analyses are required in the future. Identify the archive sample with the Sample ID and the suffix "A" (for archive fraction). Record the technician's initials and date in the Sample Preparation bench sheet (Attachment 2). Store the archive portion in the numbered inventory box noted in the Sample Preparation bench.

13.3 Preparation Duplicate Samples

One preparation duplicate sample will be prepared for every 20 field samples (or 5%) processed. A preparation duplicate is generated by using the riffle splitter to divide the preparation fraction into two equivalent portions ("parent" and "duplicate"). The duplicate portion is assigned an independent Sample ID, and both the parent sample and the duplicate sample are then processed in an identical fashion. Each sample is submitted to the laboratory blind, meaning that the laboratory is not made aware of which sample is the parent or the duplicate. For further information on preparation and processing of preparation duplicates, refer to Section 18.4.

13.4 Performance Evaluation Samples

Performance Evaluation (PE) samples are used to assess the accuracy of the analytical laboratory and to check for any potential contamination or loss of asbestos during processing. For further information on preparation and processing of PE samples, refer to Section 18.3.

13.5 Decontamination

13.5.1 The splitter need not be decontaminated following this step if the next use of the splitter will be the division of the fine-ground fraction of the same samples into four fractions (see Section 16.0). If for any reason the next use of the splitter is division of material from a different sample, the riffle splitter must be decontaminated as follows.
13.5.2 Use the HEPA vacuum and compressed air to decontaminate the splitter and

brush or wipe off any visible material that is not removed by the air blast. The splitter is now ready to process the next sample.

## 14.0 SIEVING THE PREPARATION SAMPLE

All preparation samples are sieved prior to grinding to separate the coarse fraction from the fine fractions. The sample sieving procedure is described in the sections below.

### 14.1 Equipment Calibration

14.1.1 All sieving activities will take place in the HEPA-filtered hood. Prior to any splitting, sieving, or grinding activities, use a vaneometer to verify that the hood's ventilation system is operating properly, and record the results and any required maintenance in the Ventilation Hood Verification and Maintenance Logbook.

14.1.2 Samples are weighed during sieving activities. A verification check of the analytical balance is performed each day when samples are sieved. Before weighing samples, perform the verification check using Class 6 weights (equivalent to Class S-1 weights) and record the results, any required maintenance, and the balance number in the Analytical Balance Verification and Maintenance Logbook.

### 14.2 Sample Sieving Procedure

14.2.1 Pour the sample onto a clean ¼-inch stainless-steel sieve with a clean pre-weighed catch pan. Shake the screen until all particles <¼-inch in size have passed through the screen into the pan. When needed, a pestle may be used to gently break up any remaining soil clumps to ensure all particles <¼-inch in size pass through the screen.

14.2.2 Pour all material which does not pass through the screen (>¼-inch) into a clean 4x6 inch 4-mil poly bag. This is the Coarse Fraction sample.

14.2.2.1 The weight of the coarse fraction must be recorded to the nearest 0.1 g on the Sample Preparation bench sheet (Attachment 2).

14.2.2.1.1 Place the clean poly bag on the analytical balance and tare the balance to zero. Then, transfer the coarse fraction into the poly bag and record the weight to the nearest 0.1 g. By taring the balance to zero, the recorded weight is only that of the coarse fraction and not the coarse fraction plus the poly bag.

14.2.3 If all of the material passes through the screen, such that there is no coarse fraction, record a mass of zero for the coarse fraction on the Sample Preparation bench sheet.

14.2.4 Double-bag the coarse fraction sample in a 4x6 inch 4-mil poly bag, and identify the sample with the Sample ID and "C" suffix on the sample bag. Coarse fraction samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed.

14.2.5 All material that passes through the ¼-inch screen is the Fine Fraction. Weigh and record the mass of the fine fraction to the nearest 0.1 g on the Sample Preparation Log bench sheet.

14.2.5.1 The weight of the fine fraction must be recorded to the nearest 0.1 g on the Sample Preparation bench sheet.

14.2.5.1.1 Place the clean poly bag on the analytical balance and tare the balance to zero. Then, transfer the fine fraction into the poly bag and record the weight to the nearest 0.1 g. By taring the balance to zero, the recorded weight is only that of the fine fraction and not the fine fraction plus the poly bag.

14.2.6 Whenever possible, immediately process the fine fraction material according to the procedures described in Sections 15.0 and 16.0. If processing cannot occur immediately, pour the fine fraction material into a new 4-mil poly bag and identify the fine sample material with the Sample ID and the suffix "F" (for "fine fraction"). Double-bag the sample and identify the sample with the Sample ID and suffix on the outside of the bag.

14.3 Decontamination

All non-disposable pans and sieves must be decontaminated between samples. Decontaminate sieves and pans (and the pestle, if used) in the HEPA-filtered hood using compressed air. Wipe or brush off any visible material that is not removed from the air blast. A HEPA vacuum may also be used to remove any residual material.

## 15.0 GRINDING THE FINE-FRACTION SOIL SAMPLES

The fine fraction of each preparation sample will be ground to produce a material of approximately 250 µm[1]. The procedure for grinding the fine fraction is outlined below.

15.1 Equipment Calibration

15.1.1 All grinding activities must take place in the HEPA-filtered hood. Prior to any splitting, sieving, or grinding activities, use a vaneometer to verify that the hood's ventilation system is operating properly, and record the results and any required maintenance in the Ventilation Hood Verification and Maintenance Logbook.

15.1.2 A plate grinder will be used to grind fine-fraction samples to the particle size appropriate for asbestos analysis. Verification checks of the plate grinder will be performed weekly to verify proper particle size (approximately 250 µm) and to demonstrate that samples are not being over-processed. A traceable certified micrometer will be used as a standard to perform the weekly verification checks that the shim being used for calibration is within tolerance (+/-5%). Eventually, shims will fail due to wear, bends, cracks, etc., at which time they will be replaced with a new shim that meets the requirements. The micrometer will be calibrated annually by a third party.

15.1.3 If the required particle size cannot be achieved even after plate adjustment, other grinder maintenance such as plate replacement may be required. Grinding of field samples cannot resume until the desired particle size is achieved. Document the

[1] Note that the particle size is cited as "approximately 250 µm ". This is due to the nature of grinding asbestos material. Some material that is longer than 250 µm may pass through the grinder if its longest side is parallel with the vertical grinder plates. The material that comes in contact more nearly perpendicular to the vertical grinder plates will be ground to <250 µm.

grinder number, verification of acceptable adjustment, and any observations in the Grinder Verification and Maintenance Logbook.

15.1.4 Samples will be weighed following grinding activities. A verification check of the analytical balance is performed each day when samples grinding activities take place. Before grinding samples, perform the verification check using Class 6 weights (equivalent to Class S-1 weights) and record the results, any required maintenance, and the balance number in the Analytical Balance Verification and Maintenance Logbook.

15.2 Grinding Blanks

One grinding blank per grinder will be prepared daily, and will be associated with all samples prepared by that grinder on that day. For further information on grinding blanks refer to Section 18.2.

15.3 Grinding Fine-Fraction Soil Samples

15.3.1 The sample portion that was sieved to <¼-inch will be ground to a particle size of approximately 250 µm. Set up a catch pan under the grinder to collect all the ground material. Take the fine-fraction preparation sample set aside in Section 13.2, load the grinder hopper, and allow the fine-fraction sample to pass through the plate grinder into the catch pan. Note the technician's initials, date of grinding, and grinder number on the Sample Preparation bench sheet.

15.3.2 The net recovery of fine ground material must not be less than 90% of the mass of fine material placed into the grinder. If recovery is less than 90%, soil grinding must be stopped and the grinder re-adjusted until the mass recovery of test sand and/or soil samples exceeds 90%.

15.4 Decontamination of the Plate Grinder

15.4.1 If the plate grinder can be readily disassembled for cleaning without altering its grinding properties, disassemble the grinder and clean the chutes and plates with the HEPA vacuum and compressed air. Then, if needed, wet wipe to ensure decontamination. If wet wiping is used, the plates and chutes must be thoroughly dried before reassembly.

15.4.2 If the grinder is not easily disassembled, clean the grinder with the HEPA vacuum and several blasts of compressed air, paying special attention to areas where dust from the grinding process is known to accumulate (e.g., between the plates and areas adjacent to the catch pan clamps).

15.4.3 Pass an aliquot of approximately 20 g of asbestos-free quartz sand through the grinder to clean out any residual soil.

15.4.4 Discard the quartz sand and re-clean the grinder with the vacuum and another round of high pressure air blasts. After this decontamination procedure, the grinder is ready to process the next sample.

15.4.5 In general, all soil containers, hoppers and catch pans associated with use of the grinder should be decontaminated using the HEPA vacuum and/or wet wiping, followed by a blast of high pressure air.

15.5 Decontamination of the Calibration Sieves

15.5.1 The stacked sieves used to calibrate the plate grinder will be decontaminated using the HEPA vacuum and compressed air between calibration uses.

## 16.0 SPLITTING FINE-GROUND SOIL SAMPLES

Fine-ground soil samples should be distributed into four approximately equal sub-samples using a splitter. All splitting activities will be performed in the HEPA-filtered hood. Prior to any splitting activities, use a vaneometer to verify that the hood's ventilation system is operating properly, and record the results and any required maintenance in the Ventilation Hood Verification and Maintenance Logbook.

16.1 Splitting Procedure for Fine-Ground Sample

The following method for splitting a soil sample was adapted from EPA Method 540-R-97-028 (EPA, 1997).

16.1.1 Set up receiving pans on each side of the splitter. Load the soil from the grinder catch pan into the splitter, collecting the sample in two receiving pans.

16.1.2 Tap the catch pan vigorously several times to free any remaining material. Tap the splitter to facilitate the flow of all material through the chutes into the receiving pans.

16.1.3 Empty one receiving pan into the grinder catch pan and the other receiving pan into the sieve catch pan. Set the sieve catch pan aside; this portion of fine ground sample will be split again later.

16.1.4 Replace the receiving pans under the splitter. Take the grinder catch pan, containing half of the fine ground sample, and re-load the contents into the splitter as detailed above.

16.1.5 Repeat the process of dispersing the sample material by shaking the catch pan and tapping the splitter to uniformly distribute the sample. The resulting splits are the "FG1" and "FG2" portions on the Sample Preparation bench sheet.

16.1.6 Take these two portions and carefully transfer each into a clean 4x6 inch 4-mil poly bag labeled with the Sample ID.

16.1.6.1 The weight of each fine-ground sample portion must be recorded to the nearest 0.1 g on the Sample Preparation bench sheet (Attachment 2).

16.1.6.2 Place the clean poly bag on the analytical balance and tare the balance to zero. Then, transfer the sample into the poly bag and record the weight to the nearest 0.1 g. By taring the balance to zero, the recorded weight is only that of the fine-ground sample portion and not the sample plus the clean 4x6 inch 4-mil poly bag.

16.1.7 Re-bag each fine-ground sample portion in another clean, 4x6 inch 4-mil poly bag. Identify each fine-ground sample with the Sample ID, the suffix "FG" (for "fine fraction, ground"), and the fraction number (e.g., CS-12345-FG1 for fine ground fraction #1). Set aside the FG1 and FG2 fractions of the sample.

16.1.8 Place the two empty receiving pans from the FG1 and FG2 fractions next to the splitter. Repeat the splitting procedure using the other fine ground portion set aside in the sieve pan and split the remaining sample material to create the "FG3"

and "FG4" portions.

16.1.9 Take the remaining "FG3" and "FG4" portions and repeat the procedures described in Sections 16.1.6 and 16.1.7.

16.1.10 Combine all of the bagged coarse and fine portions of the sample into one large clean, 4-mil poly bag (10x13 inch).

16.1.11 Coarse and fine-ground samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed. When samples are requested for shipment, randomly select one of the fine-ground fractions to send to the analytical laboratory. If further analyses are required of the fine-ground fractions, the remaining fractions will be double bagged and sent. All archived fine-ground fractions will be filed in the appropriate inventory archive box noted on the Sample Preparation bench sheet.

16.2 Decontamination

The splitter must be decontaminated between each sample. Use the HEPA vacuum and/or wet wiping followed by a blast of compressed air to decontaminate the splitter, and brush or wipe off any visible material that is not removed by the vacuum or air blast. The splitter is now ready to process the next sample.

## 17.0 DOCUMENTATION

17.1 Sample ID numbers are recorded on the Sample Drying bench sheet, Sample Preparation bench sheet, and on all sample containers. Once all information on the Sample Drying bench sheet and Sample Preparation bench sheet is completed, a second technician must perform a QC check of the information, and initial and date the bench sheet. Sample Drying bench sheets and Sample Preparation bench sheets will be filed or archived according to their associated dry batch and preparation batch number. If revisions to the Sample Drying bench sheets and/or Sample Preparation bench sheets are necessary, the appropriate parties will be notified of the changes; however, these changes will not necessitate revision to the current SOP. Instead, a modification form will be filled out to document the revisions.

17.2 Equipment verification and maintenance logbooks are completed each day equipment is used.

17.2.1 An additional logbook must be completed by SPF personnel each day sample preparation activities take place (Daily Activities Logbook). The Daily Activities Logbook must contain the following information:

- Date
- Time
- Personnel initials who worked that day
- PPE used
- SOP and any other laboratory-specific governing plan being followed
- Descriptions of any deviations from the SOP, including the reason for the deviation, and/or any modification forms being followed

- Summary of the daily activities (including number of samples prepared, and equipment used)
- Any additional comments

17.3 A single-line strikeout (initialed and dated) is required for any corrections/changes made to bench sheets, logbooks, or any other form used at the SPF.

## 18.0 QUALITY CONTROL

QC samples are inserted into the sample processing train to monitor for potential contamination introduced during the preparation process and to assess accuracy of analyses that may be affected by the preparation procedures. If sample results indicate the occurrence of contamination or inconsistent results, the ESAT Team Manager, QAC, and SPF Lead will be notified. The ESAT Team Manager or QAC will then notify the client in order to review laboratory procedures and identify any changes in the SOP that may be necessary. Any such reviews and resultant changes will be documented accordingly by the SPF Lead.

18.1 Preparation Blanks

18.1.1 A preparation blank is a sample of approximately 200g of asbestos-free quartz sand that is treated identically to a field soil sample. That is, the preparation sample is dried in the oven along with the field soil samples, split into archive and preparation fractions using a riffle splitter, screened through a ¼-inch sieve (even though there are no particles larger than ¼-inch), and ground by passing through the plate grinder. This type of sample is intended to detect contamination that may occur at any stage of the soil preparation procedure.

18.1.2 At least one preparation blank will be processed with each drying batch of 20 field samples. Preparation blanks will be assigned a random, unique Sample ID and will be submitted to the laboratory blind. The Sample ID assigned to each preparation blank must follow the numbering system specified in the program-specific project plan.

18.1.3 Detection of asbestos fibers in any preparation blanks at a level greater than non-detect by PLM-Visual Estimation should be taken as a sign of potential cross-contamination, and all field samples associated with the preparation batch for the preparation blank having detectable asbestos will be reviewed and qualified appropriately if detectable levels of asbestos are also found in any of the corresponding field samples. If the overall fraction of preparation blanks that contains detectable asbestos exceeds 1%, a review of laboratory procedures should be undertaken to identify and address the source of the contamination.

18.2 Grinding Blanks

18.2.1 A grinding blank consists of 100-200 grams of asbestos-free quartz sand that is passed through the plate grinder. The purpose of this type of sample is to evaluate the effectiveness of decontamination procedures for the plate grinder.

18.2.2 One grinding blank per grinder will be prepared for each day that field samples are being ground. Each grinder used in the laboratory will be assigned a number and all samples processed will be associated with the grinder used for preparation.

The grinder number used for each sample will be recorded on the Sample Preparation bench sheet. Grinding blanks will not be dried, split for archive, or sieved. Rather, a grinding blank will only be ground and split into four fine-ground samples. The grinding blank is assigned a random and unique Sample ID and is submitted to the laboratory
blind. The Sample ID assigned to each grinding blank must follow the numbering system specified in the program-specific project plan.

18.2.3 Detection of asbestos fibers in any grinding blank at a level greater than non-detect should be taken as a sign of potential cross-contamination, and all field samples associated with the grinding blank that reports detectable asbestos will be reviewed and qualified appropriately if detectable levels of asbestos are also found in any of the corresponding field samples. If the overall fraction of grinding blanks that contains detectable asbestos in a soil preparation facility exceeds 1%, steps should be taken to develop an improved method for grinder decontamination.

18.3 PE Samples

18.3.1 PE samples consist of asbestos-free soil that is spiked with a known quantity of Libby Amphibole (LA) asbestos. These samples were created by the United States Geological Survey (USGS) for use at the Libby Site by spiking uncontaminated soil from Libby with a known mass of LA collected at the mine site, and then grinding the sample to a particle size of ≤250 µm as described above. Several different concentration values of PE samples were prepared, ranging from < 0.1% to 2%.

18.3.2 PE samples will be utilized in the two following ways:

18.3.2.1 First, the SPF will insert unprocessed PE samples into the sample processing train of samples being sent to the laboratory for PLM analysis. This type of PE sample is intended to evaluate the performance of the analytical laboratory (rather than the SPF).

18.3.2.2 Second, the soil preparation laboratory will process PE samples in the same way that field soil samples are processed, with the exception of splitting the samples due to the limited quantity of each PE sample. This type of PE sample is intended to determine if there is any loss of asbestos during sample processing. In addition, considered in conjunction with a grinding blank that is passed through the decontaminated grinder immediately following the PE sample, the PE sample will also be used to facilitate assessment of grinder decontamination procedures.

18.3.3 The frequency of PE samples will be determined by the client (typically one per quarter). The asbestos concentration of the PE sample to be used, the type of PE sample (processed or unprocessed), and the analytical laboratory that will receive the PE sample will also be at the direction of the client.

18.3.4 Results of PE samples processed by the SPF are evaluated by the client by comparing the reported results for LA to the nominal values. Deviations from nominal values may be the result of variations either in soil processing procedures and/or in the analytical procedure. If the frequency of strongly discordant results exceeds 10%, then the source of the inconstancy should be investigated and remedied.

18.4 Preparation Duplicates

18.4.1 A preparation duplicate is prepared by using a riffle splitter to divide a field soil sample into two approximately equal portions, creating a parent and duplicate sample. Both samples are then processed in the same fashion. The preparation duplicate is assigned a unique Sample ID, and is submitted to the laboratory blind. The Sample ID assigned to each preparation duplicate must follow the numbering system specified in the program-specific project plan.

18.4.2 Preparation duplicate samples will be processed at a rate of 5% of the field samples processed (approximately one preparation duplicate for every 20 field samples prepared). Results from duplicate samples serve to evaluate the precision of the combined sample preparation process and the laboratory analysis. Inconsistent results between parent and duplicate samples may be due to variability in sample preparation, sample analysis, and/or to small scale variability in the sample that is not fully controlled by mixing and splitting. If the overall frequency of strongly discordant results (i.e., the results for the parent sample and duplicate are different by more than one bin category according to the current version of Libby-specific SOP SRC-LIBBY-03) is greater than 10%, steps should be taken to identify and address the source of the variability in the sample preparation procedure.

18.4.3 The rate of preparation duplicate samples should be tracked in a spreadsheet to ensure the rate is 5% of field samples processed.

18.5 Additional QA/QC

18.5.1 Periodic (at least annually) audits of the SPF will be conducted to ensure this SOP is being implemented and to identify any corrective actions or changes that need to be made to the SOP. Audits may be conducted internally by ESAT personnel, TechLaw, Inc., or by the client.

## 19.0 DECONTAMINATION

19.1 All non-disposable equipment used during soil sample preparation must be decontaminated prior to use. Scoops, spoons, splitters, sieves, and drying pans that are re-used must be decontaminated with the HEPA vacuum, compressed air, wet-wiping, and/or by brushing off any residual material.

19.2 The walls and counter top of the grinding station hood and any other area of sample handling and preparation, including floors, must be wet-wiped and HEPA-vacuumed at the end of each work day.

19.3 The sample drying oven will be HEPA-vacuumed and wet-wiped after each batch of samples.

19.4 If soil particles are visible on any of the equipment, repeat the decontamination procedure until the equipment is clean.

*Note: To reduce the potential for human exposure to asbestos in the SPF, compressed air*

*should be used carefully according to the manufacturer's instructions and only in the HEPA-filtered hood.*

## 20.0 REFERENCES

20.1 American Society for Testing and Materials. 1998. Standard Practice for Reducing Samples of Aggregate to Testing Size. ASTM Designation: C 702 - 98, 4 p.

20.2 United States Environmental Protection Agency. 1997. Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials. Method 540-R-97-028.

# ATTACHMENT 1

## Libby Asbestos Superfund Site Sample Drying Bench Sheet

# Sample Drying Bench Sheet

TechLaw, Inc. ESAT Region 8
Troy Sample Preparation Facility

SOP: ISSI-LIBBY-01, Rev. 11
Oven No.: _____

| | | |
|---|---|---|
| Batch ID:<br>(YY-####) | COC: | Due Date: |
| Begin Date:<br>(MM/DD/YY) | Begin Time:<br>(HH:MM) | Begin Temp:<br>C ° |
| End Date:<br>(MM/DD/YY) | End Time:<br>(HH:MM) | End Temp:<br>C ° |

| | |
|---|---|
| Beginning Technician(s): | Ending Technician(s): |

| Sample ID: | Wet Weight (g) | Dry Weight (g) | Drying Comments |
|---|---|---|---|
| 1. | | | |
| 2. | | | |
| 3. | | | |
| 4. | | | |
| 5. | | | |
| 6. | | | |
| 7. | | | |
| 8. | | | |
| 9. | | | |
| 10. | | | |
| 11. | | | |
| 12. | | | |
| 13. | | | |
| 14. | | | |
| 15. | | | |
| 16. | | | |
| 17. | | | |
| 18. | | | |
| 19. | | | |
| 20. | | | |

* Each dried sample will be temporarily stored in a plastic tote identified by a Batch ID in the format "YY-# # # #" (e.g.,10-1000)

Comments: _____

_____

QC: _____ CK COC: _____ Date: _____ Loaded: _____ Shipped: _____

# ATTACHMENT 2

## Libby Asbestos Superfund Site Sample Preparation Bench Sheet

# Sample Preparation Bench Sheet

TechLaw, Inc. ESAT Region 8

SOP: ISSI-LIBBY-01, Rev. 11

Troy Sample Preparation Facility

Preparation Batch: _____

| | Sample ID | Inventory ID | Drying Batch ID | Archive Sample | Duplicate Sample | Sieving | | | Sample Grinding | | Sample Splitting | | | | | Comments (indicate if Grinder Blank, Prep Blank, or Duplicate Pair; for Duplicate Pair, enter the parent ID) | QC |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Sample Mass (g) | | | | | Sample Mass (g) | | | | | | |
| | | | | Initials and Date | Initials and Date | Coarse Fraction (> 1/4") | Fine Fraction (< 1/4") | Initials and Date | Initials and Date | Grinder # | FG1 | FG2 | FG3 | FG4 | Initials and Date | | Initials and Date |
| 1 | | | | | | | | | | | | | | | | | |
| 2 | | | | | | | | | | | | | | | | | |
| 3 | | | | | | | | | | | | | | | | | |
| 4 | | | | | | | | | | | | | | | | | |
| 5 | | | | | | | | | | | | | | | | | |
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| 19 | | | | | | | | | | | | | | | | | |
| 20 | | | | | | | | | | | | | | | | | |

# 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 |

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LIST OF ATTACHMENTS

Attachment 1: Libby Asbestos Superfund Site Analysis Bench Sheet (PLM-Grav)

## 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 ($\alpha$ and $\gamma$)

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.

# ATTACHMENT 1

## Libby Asbestos Superfund Site Analysis Bench Sheet (PLM-Grav)

# LIBBY ASBESTOS SUPERFUND SITE ANALYSIS BENCH SHEET (PLM-Grav)

Laboratory Name: _____

Work Order Number: _____

Date Received: _____

SOP Name/Revision: _____

| | | | | | |
|---|---|---|---|---|---|
| Client Sample Number | | | | | |
| Tag | | | | | |
| Lab Sample ID | | | | | |
| Date Analyzed | | | | | |
| Analyst Initials | | | | | |

*TOTAL SAMPLE WEIGHT (g):*

| | | | | | |
|---|---|---|---|---|---|
| Wt of Empty Container | | | | | |
| Wt of Sample + Container | | | | | |

*STEREOMICROSCOPIC EXAMINATION:*

| | | | | | |
|---|---|---|---|---|---|
| Homogeneity (Y/N) | | | | | |
| Sample Color | | | | | |
| Sample Type/Texture | | | | | |
| Type and % of Non-Asbestos Fibers | | | | | |
| Non-Fibrous Matrix Materials (if known) | | | | | |

*MASS OF ASBESTOS PARTICLES (g):*

| | | | | | |
|---|---|---|---|---|---|
| LA Qual (ND, Tr) | | | | | |
| Wt of Empty Container | | | | | |
| Wt of LA + Container | | | | | |
| OA Qual (ND, Tr) | | | | | |
| OA Type | | | | | |
| Wt of Empty Container | | | | | |
| Wt of OA + Container | | | | | |
| CH Qual (ND, Tr) | | | | | |
| Wt of Empty Container | | | | | |
| Wt of CH + Container | | | | | |

*ASBESTOS OPTICAL PROPERTIES BY PLM:*

| | | | | | |
|---|---|---|---|---|---|
| Habit | | | | | |
| Fiber Color | | | | | |
| Sign of Elongation (+/-) | | | | | |
| Pleochroism (Y/N) | | | | | |
| Extinction Angle | | | | | |
| Refractive Index ($\alpha$) | | | | | |
| Refractive Index ($\gamma$) | | | | | |
| Birefringence | | | | | |
| Becke Line or CSDS | | | | | |
| Temperature (°C) | | | | | |

*OTHER:*

| | | | | | |
|---|---|---|---|---|---|
| QC Type | | | | | |
| Deviation (Y/N) | | | | | |
| Comments | | | | | |

# 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. |

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Attachment 3: Analytical Test Report Standard Laboratory Data Package Checklist
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Attachment 7: Photomicrographs of Representative Fields of View of 0.2% and 1.0% LA Controlled PE Reference Materials
Attachment 8: Flow Chart for Determining Asbestos Content by Complementary Use of Stereomicroscopy and PLM Visual Estimation
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## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standard approach for semi-quantitative analysis of asbestos in samples of soil or other soil-like materials using the visual area estimation technique by Polarized Light Microscopy (PLM). This SOP is specifically intended for application at the Libby Asbestos Superfund Site (referred to as the Libby Site from this point forward) and has been refined to focus testing on Libby Amphibole (LA) asbestos at levels below 1%.

## 2.0 SCOPE AND APPLICATION

This method is intended for analysis of asbestos in soil or other similar soil-like media in which the soil has been taken through the preparation process described in Section 4.0. This method is appropriate for the analysis of all types of asbestos fibers (chrysotile and amphiboles), including LA. For the purposes of this SOP, the term 'asbestos' will refer to the six regulated asbestos minerals (chrysotile, amosite, crocidolite, anthophyllite, tremolite, and actinolite), as well as LA.

## 3.0 RESPONSIBILITIES

3.1 It is the responsibility of the laboratory supervisor to ensure that all analyses and quality control (QC) procedures are performed in accordance with this SOP and to identify and take appropriate corrective action to address any deviations that may occur during sample preparation or analysis.

3.2 The Laboratory Manager, Quality Assurance Coordinator (or equivalent), and/or Analytical Lead will communicate with the client, any situations where a modification to or deviation from the SOP may be useful and/or required. The laboratory supervisor must receive approval from the client for any modification to or deviation from the SOP before incorporating any such modification or deviation into the sample preparation and analysis process (refer also to Section 8.2).

3.3 It is the responsibility of the laboratory to maintain a PLM SOP for Bulk Asbestos Materials, Quality Assurance Manual (QAM), or an equivalent document(s) that meets all the requirements of the National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150 and Handbook 150-3. It is also the responsibility of the laboratory to ensure its testing activities stay in compliance with the requirements of NVLAP Handbooks 150 and 150-3 and the regulatory and accrediting agencies that provide oversight of the laboratory's operations and all Libby Site project-specific requirements.

## 4.0 METHOD DESCRIPTION

4.1 The test method describes a semi-quantitative analysis of asbestos in samples of soil or other soil-like materials using the visual area estimation technique by PLM, referred to as PLM-VE. The test method used for analyzing PLM asbestos samples specific to the Libby Site is based on the National Institute of Occupational Safety and Health (NIOSH) Method 9002, United States Environmental Protection Agency (EPA) Method 600/R-93/116, and the State of California Air Resources Board (CARB) Method 435, with project-specific modifications provided in this SOP.

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, accurate to 1mg (0.001g)

10.3.7 Libby Asbestos Data Tool (LADT) or other computer software capable of generating a project-specific Electronic Data Deliverable (EDD) that meets the current client data reporting requirements

10.3.8 Mortar and pestle (agate or porcelain)

10.3.9 Vaneometer

10.3.10 Wet/dry vacuum with HEPA filtration

10.3.11 Decontamination equipment (e.g. disposable lint-free wipes, wet mop with bucket, etc.)

## 11.0 STANDARDS, REAGENTS AND SUPPLIES

11.1 High Dispersion RI Liquid(s) from 1.620 to 1.640

11.2 1.550 High Dispersion RI Liquid

11.3 1.680 to 1.700 RI Liquid(s)

11.4 Solid RI Standards (precision optical glass, RI from 1.48 to 1.72, in gradations of 0.01, 25 standards)

11.5 National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 1866b - Common Commercial Asbestos consisting of chrysotile, amosite, and Crocidolite

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

.

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 Initial Stereomicroscopic Examination

13.1.1 All sample preparation activities, including stereomicroscopic examination, slide mounts, etc., must be performed in a HEPA-filtered hood, class 1 biohazard hood, or glove box with continuous airflow (negative pressure).

13.1.2 Due to the sample preparation requirements described in the current revision of SOP ISSI-LIBBY-01, *Soil Sample Preparation*, samples should never be wet. If the sample is wet, contact the client.

13.1.3 The stereomicroscope is a low magnification microscope (approximately 10X-50X) used for visual examination of specimens at a coarse scale. Stereomicroscopic examination is especially useful for soil samples where fibers may be unevenly or thinly distributed throughout the sample.

13.1.4 Begin the analysis by pouring the entire sample out of its sample bag onto a clean, asbestos-free substrate, such as an agate mortar, ceramic evaporating dish, watchglass, weighing dish, etc.

13.1.4.1 For fine-ground soil samples, the mass of the sample will ideally be 20 to 50 grams; however, some samples submitted to the laboratory may be smaller or larger.

13.1.5 With the stereomicroscope, visually examine the entire sample for homogeneity, sample color and texture, and the presence of any suspect fibers.

13.1.6 If individual fibers suspected of being asbestos are observed during this initial examination, pick out one or more of these fibers with fine forceps (or other appropriate utensil) and mount them on a glass microscope slide in an appropriate RI liquid. These sample preparations are referred to as fiber-picks in this SOP.

13.1.6.1 Each microscope slide must be wiped with disposable lint-free wipes prior to use to avoid contamination.

13.1.6.2 Mount individual fibers in 1.550 RI liquid if chrysotile is suspected, 1.620 to 1.640 RI liquid if LA or anthophyllite is suspected, or 1.680 to 1.700 RI liquid if amosite or crocidolite is suspected.

13.1.6.3 Only one drop of RI liquid is necessary to prepare a fiber-pick slide.

13.1.6.4 Cover this preparation with a glass cover slip and identify the fibers using PLM analysis techniques (see Section 13.5).

13.1.7 Record all stereomicroscopic findings, including homogeneity, sample appearance (color and texture), an initial estimated percent LA, and an initial estimated percent other asbestos (chrysotile and other amphibole), in the appropriate fields on the analytical bench sheet.

13.1.7.1 Stereomicroscopic examination does not provide positive identification of asbestos fibers. Later analysis by PLM will confirm, deny, or refine the preliminary estimated percent and type of asbestos.

13.1.7.2 The procedure for performing a calibrated visual estimate using both

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 Secondary Stereomicroscopic Evaluation

13.4.1 After the sub-samples have been taken from the original sample, aggressively agitate or tap the sample substrate containing the original sample to cause the particulate to settle and the asbestos fibers to sort to the surface.

13.4.1.1 Re-examine the entire sample using the stereomicroscope, and repeat the fiber-pick procedures described in Section 13.1.6.

*Note: If a fiber-pick was prepared during the initial stereomicroscopic examination, it is not required that another fiber-pick be prepared after agitating the substrate. However, regardless of whether or not a fiber-pick was performed during the initial stereomicroscopic examination, each sample substrate must be agitated and the sample re-examined using stereomicroscopy following the procedures in this section. Additional fiber-picks can be prepared at the analyst’s discretion.*

13.4.1.2 Agitating the substrate should only be used as a qualitative technique following random slide mount preparation and not as a quantitative technique because it tends to make the sample inhomogeneous.

*Note: Agitating the substrate and re-examining the sample using stereomicroscopy can be done prior to preparing the five slide mounts. However, do not agitate the substrate until all the sub-samples have been taken from the original sample and placed in the mortar in order to avoid collecting inhomogeneous sub-sample material.*

13.4.2 Avoid contamination by maintaining a clean work space.

13.4.2.1 After preparing each sample, clean all work surfaces, sample substrates, utensils, and any other items that came into contact with the sample, using water and paper towels.

13.4.2.2 Dispose of gloves once they become excessively dirty.

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 $\alpha$) 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 $\gamma$) 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 ($\delta$; expressed numerically as the difference between $\alpha$ and $\gamma$) 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 ($\alpha$) 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 ($\gamma$) 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 ($\alpha$) for crocidolite is 1.680 to 1.698 (EPA, 1993).

13.5.9.5 Normally, the range for the higher RI ($\gamma$) 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 ($\alpha$) should be measured with the fiber oriented in the E-W direction (parallel to the lower polar), and the higher RI ($\gamma$) 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 ($\alpha$) for anthophyllite is 1.593 to 1.694 (Deer et al., 1997). The commercial-grade anthophyllite in SRM 1867a has an $\alpha$ of 1.615.

13.5.10.5 The range for the higher RI ($\gamma$) for anthophyllite is 1.613 to 1.722 (Deer et al., 1997). The commercial-grade anthophyllite in SRM 1867a has a $\gamma$ 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 $\lambda_0$. 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 $\lambda_0$. Record the predominant dispersion staining color and corresponding $\lambda_0$ 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 $\lambda_0$ 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 $\lambda_0$ 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 $\alpha$, 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 $\lambda_0$, 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 ($\lambda_0$) that corresponds to the observed color.

13.6.3.11 When measuring $\alpha$ 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 $\lambda_0$.

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 $\lambda_0$; record the corresponding RI value.

13.6.3.14 To measure $\gamma$, 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 $\gamma$.

13.6.3.16 Observe the dispersion staining colors and find the corresponding $\lambda_0$. When measuring $\gamma$, choose the color that produces the highest RI, i.e., the color that corresponds to the shortest $\lambda_0$.

13.6.3.17 Consult the appropriate chart for the asbestos type and liquid being used; record the RI value for the temperature and $\lambda_0$.

*Note: There are two charts for each mineral and liquid combination - one for $\alpha$ and one for $\gamma$. Be sure to use the appropriate chart when measuring $\alpha$ or $\gamma$.*

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 $\alpha$, 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 ($\alpha$') that is between true $\alpha$ and beta ($\beta$) when attempting to measure $\alpha$.

13.6.5.3 True $\alpha$ 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 $\alpha$ and $\gamma$ 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 $\alpha$ and $\gamma$. 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 $\beta$ 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 $\alpha$ 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 $\alpha$ for those that display the lowest RI.

13.6.5.7 Similarly, it is easy to measure a RI called gamma prime ($\gamma$') that is between $\beta$ and true $\gamma$ when attempting to measure $\gamma$. True $\gamma$ can only be observed when a fiber is oriented in exactly the correct position.

13.6.5.8 When measuring $\gamma$, 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 $\gamma$ for those that display the highest RI.

13.6.5.9 Biaxial Optics of Anthophyllite

13.6.5.9.1 When measuring $\alpha$ (the lower RI) for anthophyllite, the fiber should be oriented in the N-S direction. At this orientation, they can show either $\alpha$ or $\beta$, or anywhere in between. It is therefore necessary to examine a number

of fibers oriented in the N-S position to find true $\alpha$, which will be observed for fibers that display the lowest RI).

13.6.5.9.2 When measuring $\gamma$ (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 $\gamma$, not $\gamma'$, 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 ($\alpha$ = 1.538 to 1.554, $\gamma$ = 1.575 to 1.602), but both are lower than those of other asbestos minerals.

13.8.3.4 Talc has higher birefringence than chrysotile, in the range of 0.03 to 0.05, which gives relatively fine fibers of talc first order white to yellow interference colors under crossed polars. Chrysotile fibers of comparable size would have low first order gray interference colors.

13.8.3.5 Grains of talc display parallel extinction.

13.8.4 Wollastonite $CaSiO_3$

13.8.4.1 Wollastonite is one of the pyroxenoid minerals and has a characteristically bladed or prismatic habit.

13.8.4.2 Wollastonite is colorless in plane light.

13.8.4.3 The RIs of wollastonite ($\alpha$ = 1.616 to 1.645, $\gamma$ = 1.631 to 1.656) are very close to that of tremolite; however, wollastonite has a lower birefringence (0.013 to 0.017).

13.8.4.4 Wollastonite has an extinction angle of up to approximately five degrees, which makes it easy to confuse with tremolite.

13.8.4.5 Grains of wollastonite can be spun about their long axis until they change from length slow to length fast or vice versa; whereas, grains of tremolite will always remain length slow regardless of their optical orientation.

13.8.4.6 To spin a wollastonite grain about its long axis, agitate the mixture of RI liquid and sample material by repeatedly tapping the cover slip

with the point of a ball point pen. Unless the grains are lying flat on one crystal face, they should rotate as the RI liquid is agitated.

13.8.5 Kyanite $Al_2SiO_5$

13.8.5.1 Kyanite is an orthosilicate mineral that is commonly used in refractory materials and often has a bladed or columnar habit.

13.8.5.2 Kyanite is colorless to light blue in plane light and may display weak pleochroism. Both its color and pleochroism are much more subdued than that of crocidolite.

13.8.5.3 Kyanite's RIs are higher than those of both crocidolite and amosite ($\alpha$ = 1.710 to 1.718, $\gamma$ = 1.724 to 1.734).

13.8.5.4 Birefringence for kyanite ranges from 0.012 to 0.016.

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

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 ($\alpha$ = 1.60 to 1.70, $\gamma$ = 1.62 to 1.73), but most hornblende has $\alpha$ = 1.645 to 1.665 and $\gamma$ = 1.660 to 1.690.

13.8.6.5 The birefringence of hornblende is moderate, ranging from 0.014 to 0.034, but most falls within 0.018 to 0.028.

13.8.6.6 Hornblende can have parallel or inclined extinction depending on optical orientation. When extinction is inclined, the extinction angle is usually 14 to 25 degrees.

13.8.7 Calcic Clinopyroxene

13.8.7.1 The calcic clinopyroxene group includes Augite, $(Ca,Na)(Mg,Fe,Al)(Si,Al)_2O_6$, 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 $\alpha$ = 1.66 to 1.75 and $\gamma$ = 1.69 to 1.77.

13.8.7.5 The birefringence of calcic clinopyroxene is moderate, as with the majority of other pyroxenes, ranging from 0.018 to 0.034.

13.8.7.6 Calcic clinopyroxene can have a very high extinction angle, up to 48 degrees. In grains with high extinction angles, the sign of elongation

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 ($\alpha$ = 2.6 to 2.7, $\gamma$ = 2.8 to 2.9).

13.8.13.5 Needles of rutile have high birefringence, are length slow, and show parallel extinction.

## 14.0 RECORDING DATA AND RESULTS

14.1 Analytical Bench Sheets

14.1.1 Analysts record, by hand, on analytical bench sheets, analytical results at the time the observations are made. Refer to Attachment 1 for one example of a PLM-VE bench sheet.

14.1.1.1 Additional bench sheets may be created by the laboratory as long as all of the required fields are included.

14.1.2 Completed bench sheets are the original, hard-copy records on which test data on client samples is stored.

14.2 Stereomicroscopic Examination Reportables

14.2.1 Homogeneity (Yes or No)

14.2.2 Sample appearance, including color and texture

14.2.3 Estimated percent LA

14.2.4 Estimated percent other asbestos (other amphibole and chrysotile)

14.3 Reporting Positive Asbestos Results

14.3.1 If asbestos is positively identified in the sample during PLM analysis, record the following data for each asbestos type that is present in the sample on the bench

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 ($\alpha$ and $\gamma$)

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:

Laboratory ID_Work Order Number_Analytical Method_ Correction Number
Example: ESATR8_0920120002_PLM-VE_C0

15.1.3 The EDD serves as an electronic version of the test report submitted to the client.

15.1.3.1 Only one EDD is produced for each chain of custody (COC) received by the laboratory.

15.1.3.2 A hardcopy of the test report is also delivered to the client (see Section 15.2 for further details about hardcopy test reports).

15.1.3.3 The laboratory retains all original records until otherwise instructed by the client.

## 15.2 Test Report Generation

15.2.1 Hardcopy test reports of the raw analytical data are submitted to the client for archival.

15.2.2 A completed test report consists of a cover sheet signed and dated by an approved signatory, as well as the following information and documentation:

15.2.2.1 The laboratory work order number, COC number, number of samples received, and copies of the signed COCs.

15.2.2.1.1 A work order number is a unique number assigned by the laboratory to a set of samples from a single COC. Work order numbers are never duplicated.

15.2.2.2 The date of sample receipt and condition of samples.

15.2.2.3 A Case Narrative, including any opinions and interpretations; deviations, modifications, additions to, or exclusions from the test method; descriptions of any problems encountered in the analysis; or any specific conditions that could affect the results. Also include the following disclaimer: “This test report relates only to items tested.”

15.2.2.4 PLM-VE Analysis Results, as presented in the EDD and containing the analytical data (including all LDC and LDS analyses performed on any samples in the work order).

15.2.2.5 Copies of the handwritten bench sheets containing the analyst’s original data and observations.

15.2.3 Refer to Attachment 3, the Analytical Test Report Standard Laboratory Data Package Checklist, for a complete list of items required for each test report.

15.2.4 When opinions and interpretations are provided in a test report, the laboratory will:

15.2.4.1 Document the basis on which the opinions and interpretations were made.

15.2.4.2 Clearly indicate on the test report which items are opinions and interpretations.

15.2.5 Once the test report is complete, all pages must be paginated prior to delivery to the client.

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.

## 17.0 REFERENCES

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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 _____ |
| | | | | | 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

| $\lambda_m$ (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 |

| $\lambda_m$ (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 |

| $\lambda_m$ (nm) | **1.635** (Cargille Series E) | | | | | **1.640** (Cargille Series E) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Cargille Glass 1.64 (Lot B) | | | | | 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 |

| $\lambda_m$ (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) | | | | |
|---|---|---|---|---|---|
| $\lambda_m$ (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<br>1.604-1.612<br>1.599-1.612<br>1.6063 | 1.625-1.655<br>1.627-1.635<br>1.625-1.637<br>1.6343 | 0.017-0.028 | Oblique (up to 21 °); | + (length slow) |
| Actinolite[7] | | 1.600-1.628<br>1.612-1.668<br>1.613-1.628<br>1.6126 | 1.625-1.655<br>1.635-1.688<br>1.638-1.655<br>1.6393 | 0.017-0.028 | | + (length slow) |
| Winchite | Straight to curved fibers or bundles. Colorless to pale blue<br>Pleochroism weak to moderate: X-colorless, Y=light blue-violet, Z=light blue[3] | **1.618-1.626**[1]<br>**1.618-1.621**[2]<br>1.629[3]<br>1.636[4] | **1.634-1.642**[1]<br>**1.634-1.637**[2]<br>1.650[3]<br>1.658[4] | **0.008-0.019**[1]<br>**0.016**[2]<br>0.021[3]<br>0.022[4] | Oblique, **22°**[1]<br>**15.8°**[2]<br>Oblique, 7-29°[8] | + (length slow) |
| Richterite | Straight to curved fibers or bundles. Colorless, pale yellow, brown, pale to dark green, or violet[8]<br>Pleochroism weak to strong in pale yellow, orange, and red[5] | **1.622-1.623**[1]<br>1.605-1.624[5]<br>1.615[6] | **1.638-1.639**[1]<br>1.627-1.641[5]<br>1.636[6] | **0.012-0.017**[1]<br>0.017-0.022[5] | Oblique, **21-22°**[1]<br>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] |

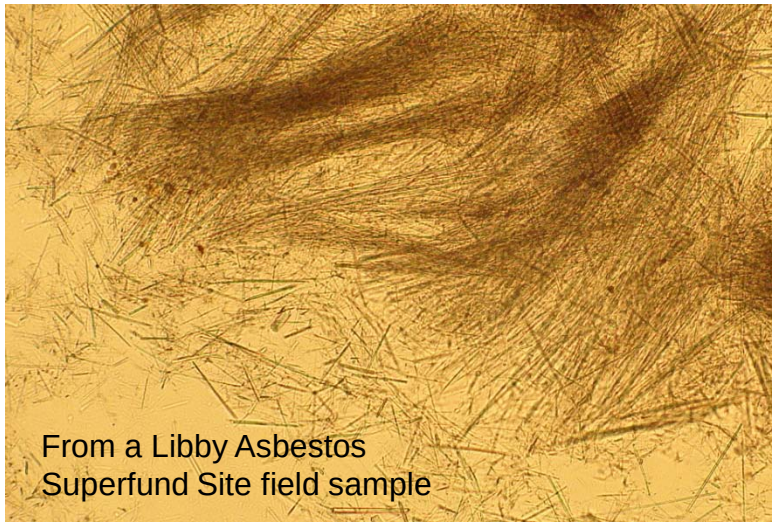
A. This table is adapted for use in SOP SRC-LIBBY-03 (Revision 3) from: Su, Shu-Chun, 2005. White paper: *Tables to Facilitate the Determination of Refractive Indices of Winchite and Richterite, (Libby, Montana) by Dispersion Staining*, August 8, 2005. Data on this table were compiled from data of amphiboles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

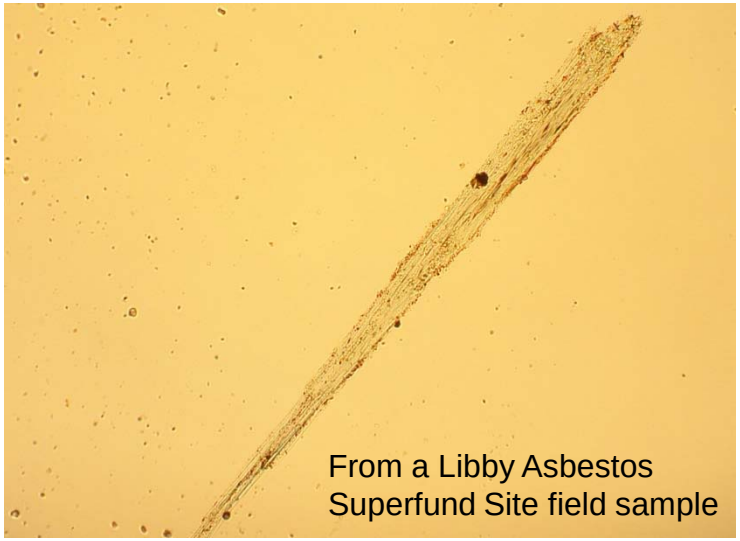
1. Bandli, B.R. et al. (2003) *Optical, compositional, morphological, and X-ray data on eleven particles of amphibole from Libby, Montana, U.S.A.* Canadian Mineralogist, 41, 1241-1253.
2. Wylie, A.G. and Verkouteren, J.R. (2000) *Amphibole asbestos from Libby, Montana: Aspects of nomenclature.* American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

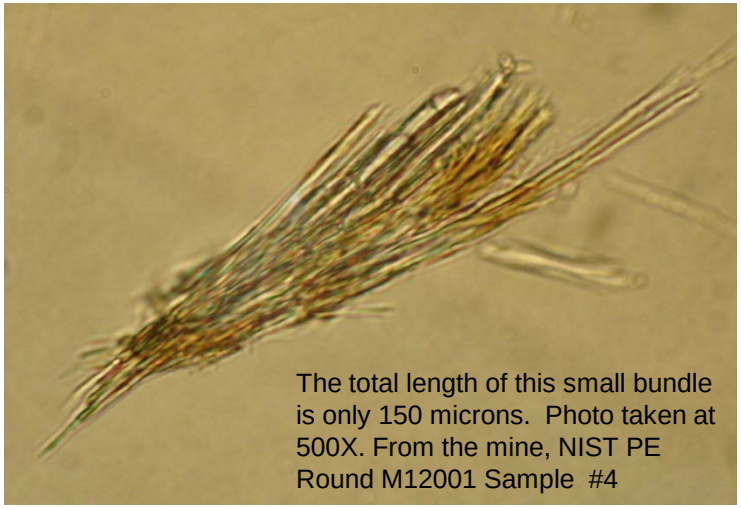
# ATTACHMENT 5

## PLM Photomicrographs Demonstrating a Wide Range of LA Habits

# PLM Photomicrographs Demonstrating a Wide Range of Libby Amphibole Habits

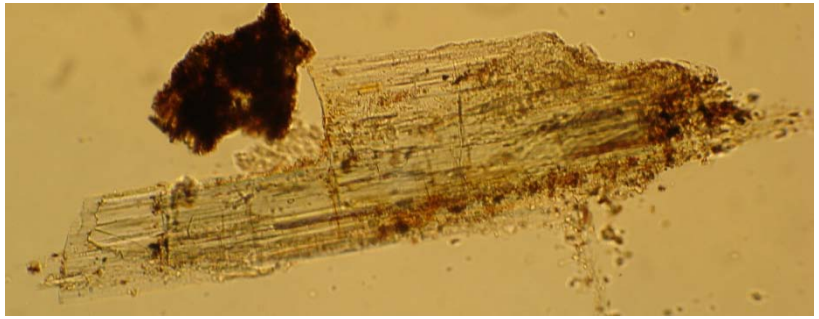




# Prismatic Libby Amphibole

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.



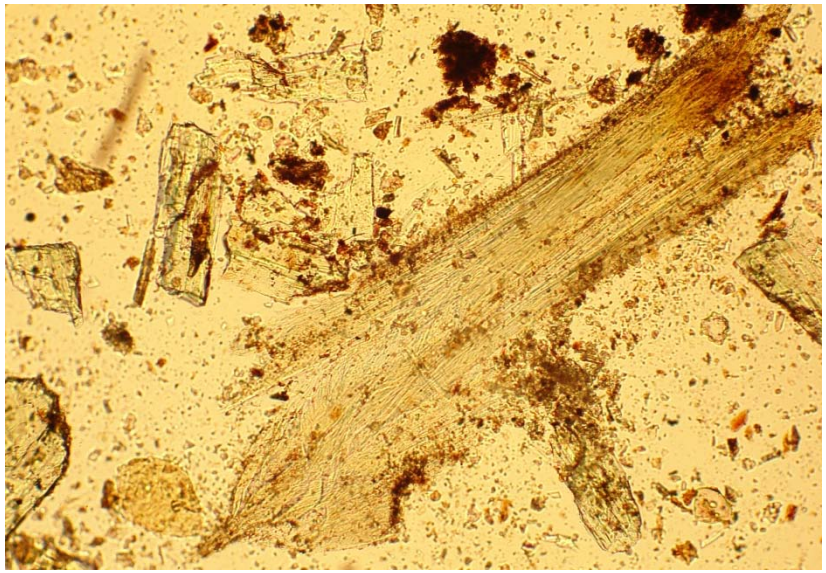
From a Libby Asbestos Superfund Site field sample

From the mine, NIST PE Round M12001 Sample #4

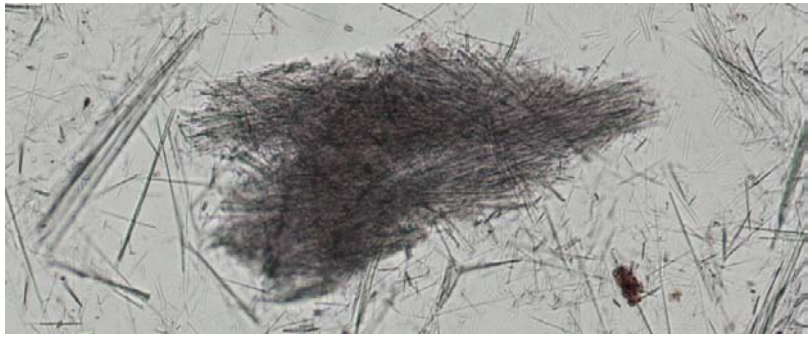
From the mine, NIST PE Round M12001 Sample #4

From a Libby Asbestos Superfund Site field sample

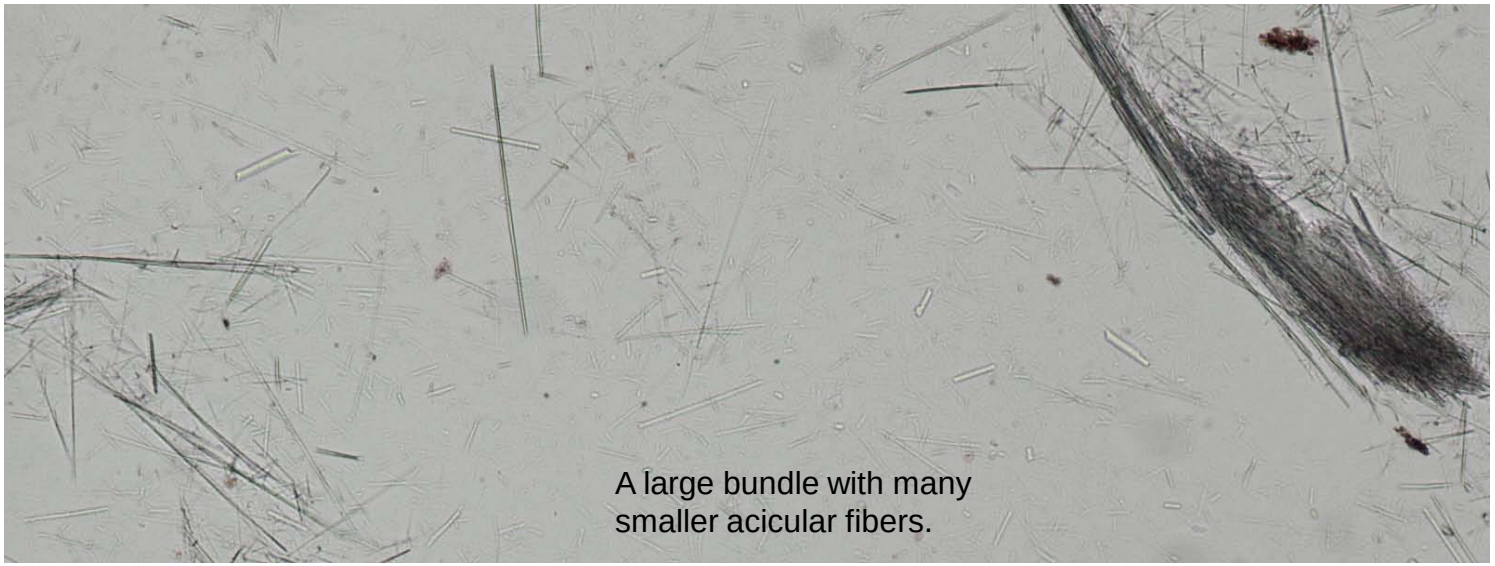
Some LA shows a “matted” or “felted” habit. The internal structure of these bundles is still fibrous. The green high-relief prismatic grains in the top right photo are hornblende. The bundles in the two top photos were found in Libby Asbestos Superfund Site field samples. The bundles in the lower two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

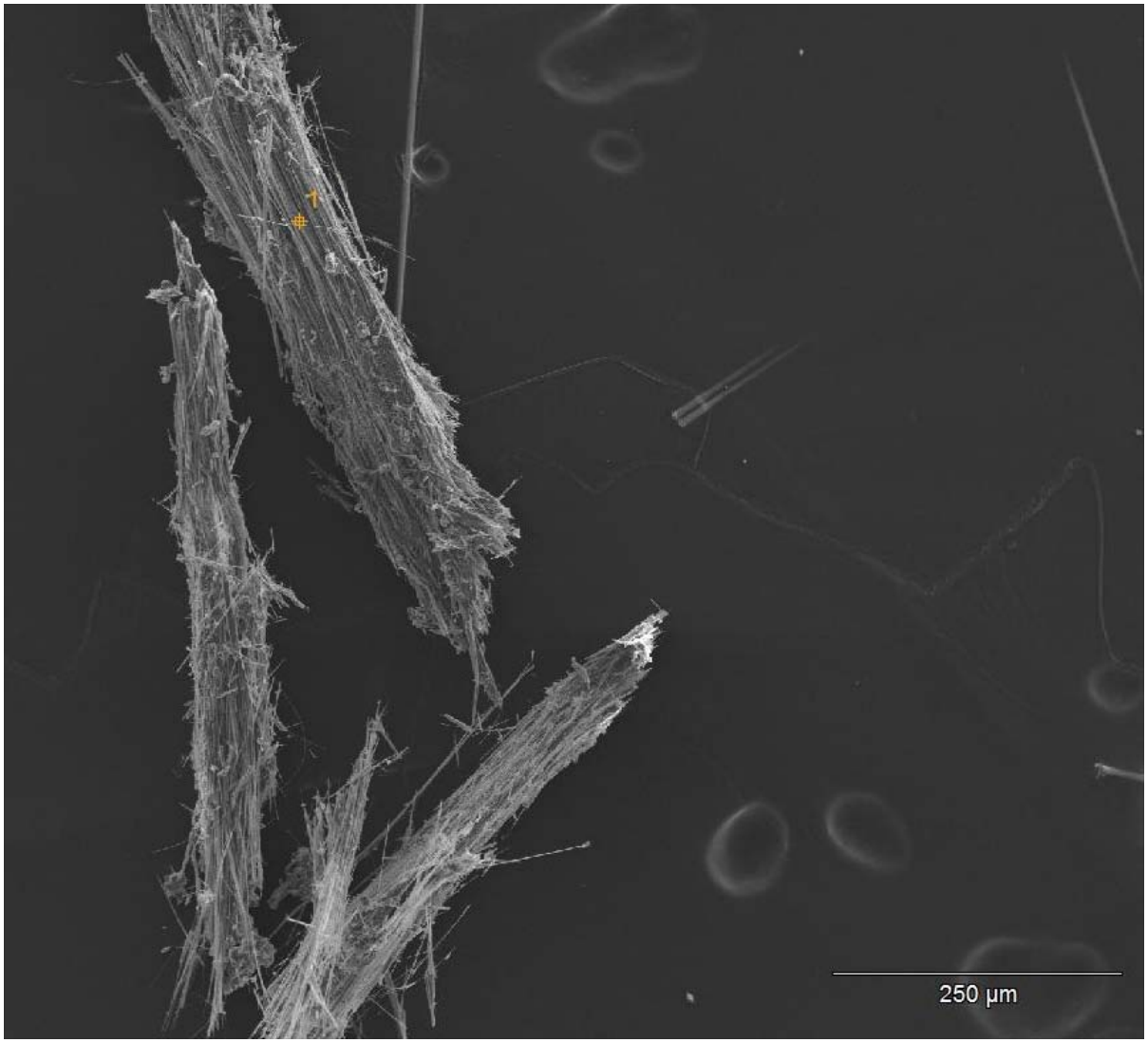
# ATTACHMENT 6

## SEM Photomicrographs of Representative Examples of LA Habits

# SEM Photomicrographs of Representative Examples of Libby Amphibole Habits

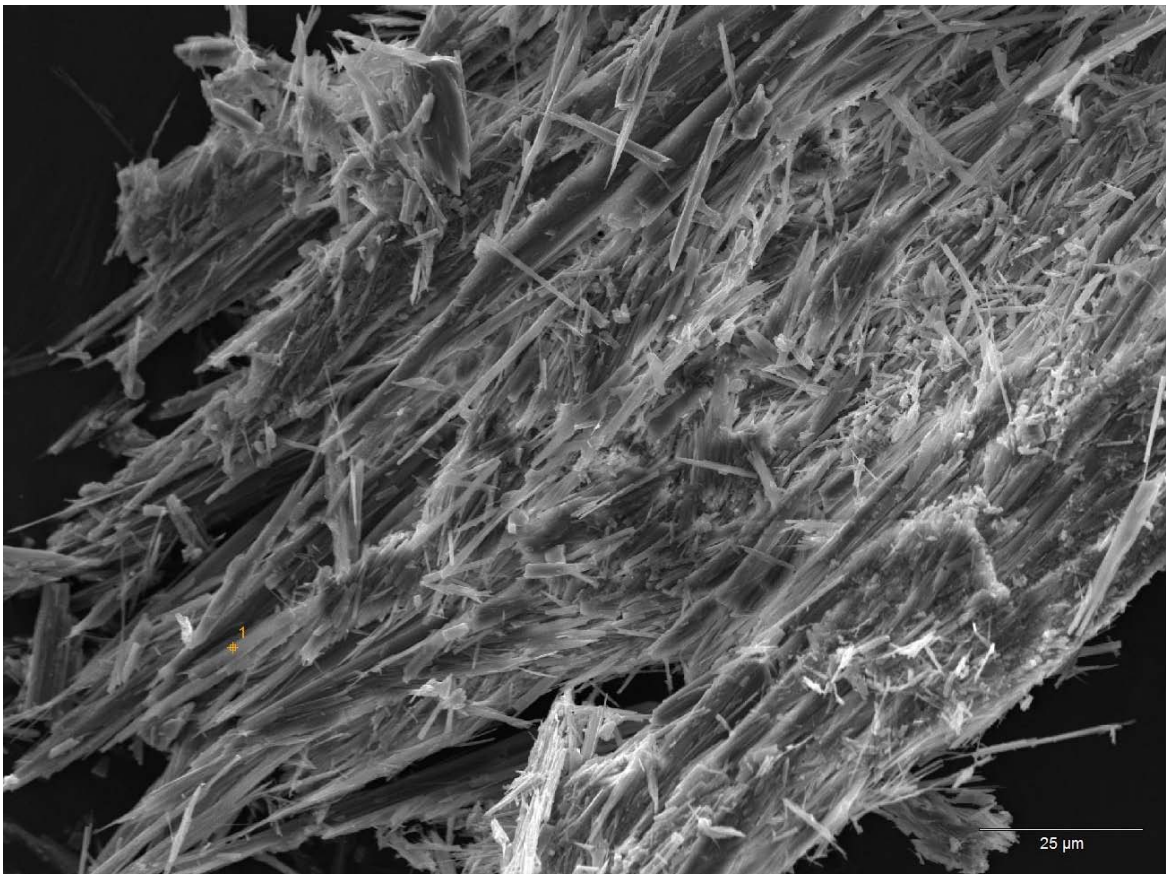
Individual bundles of Libby Amphibole (LA) were picked from soil samples at the ESAT Region 8 Laboratory and prepared for analysis by scanning electron microscopy (SEM). Slide mounts of these bundles were initially prepared in a refractive index (RI) liquid and the bundles were examined by PLM. Then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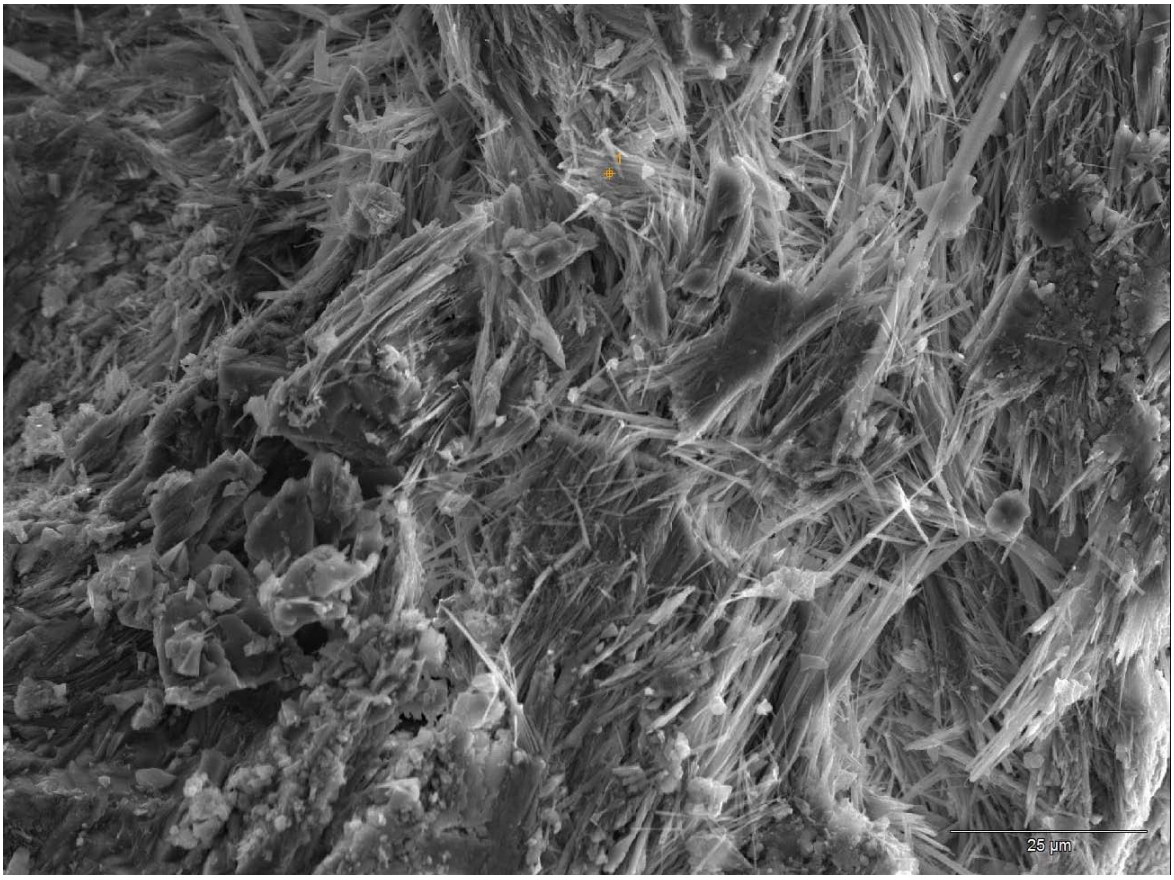
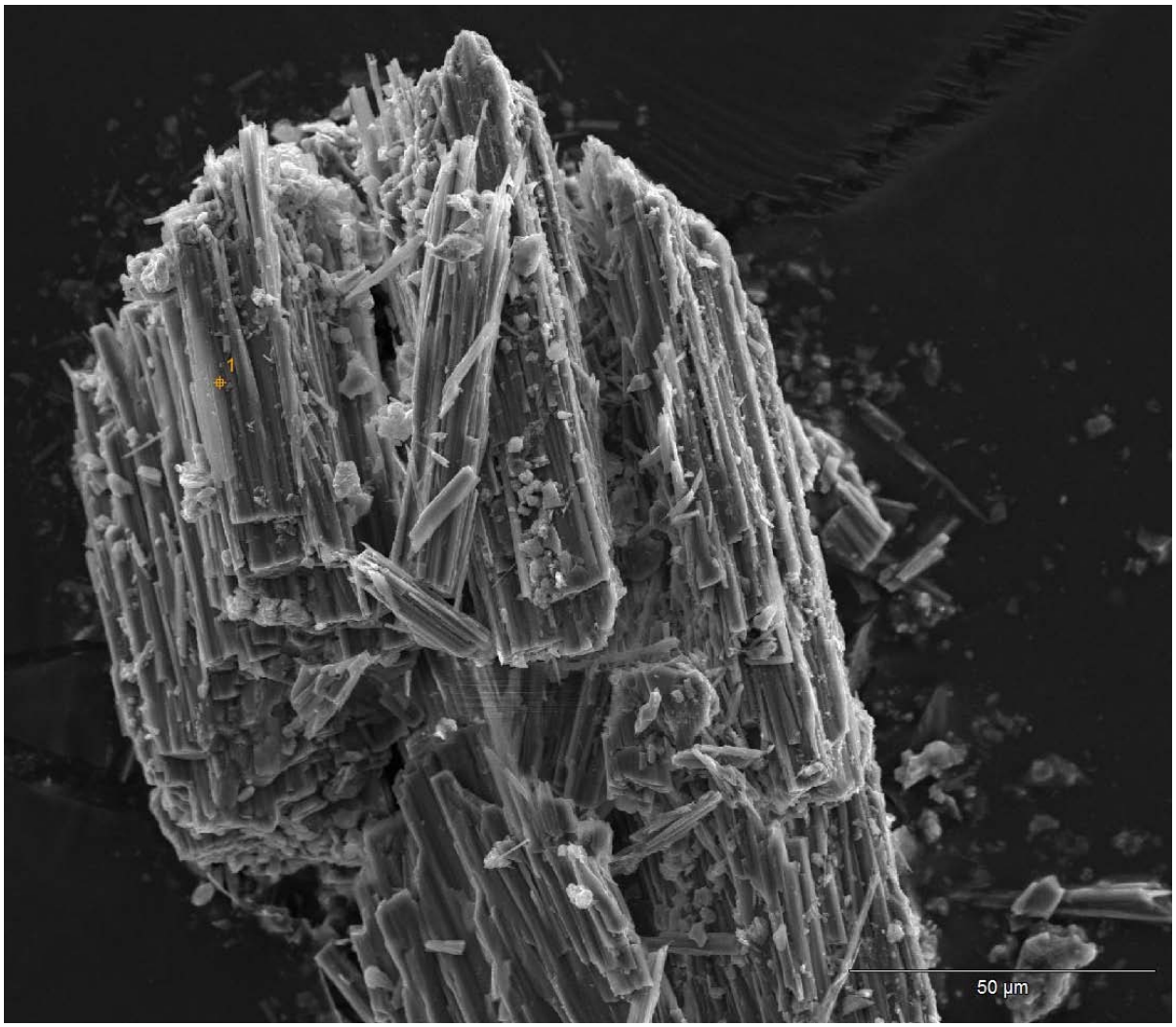


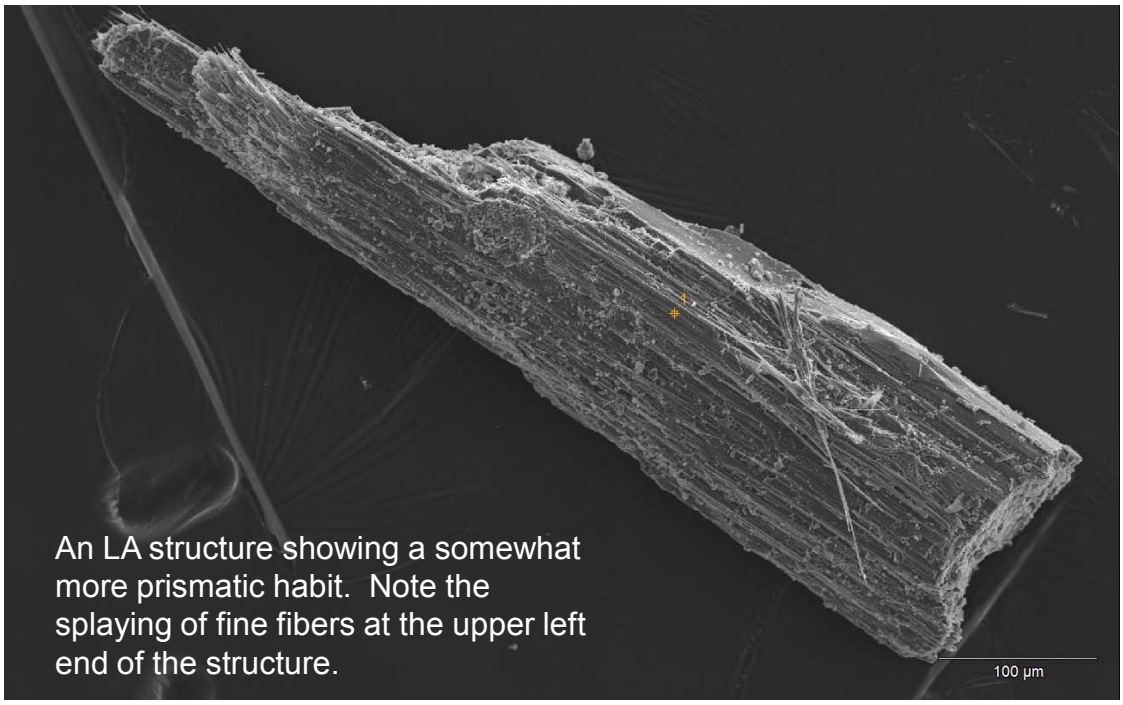

When this bundle of LA was viewed under PLM, its habit was described as “felted” or “matted” with the fibers crossing at high angles to one another. This is how the bundle appeared when it was subsequently viewed by SEM. The fibrous nature of the “felted” or “matted” habit is clear at this scale.

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

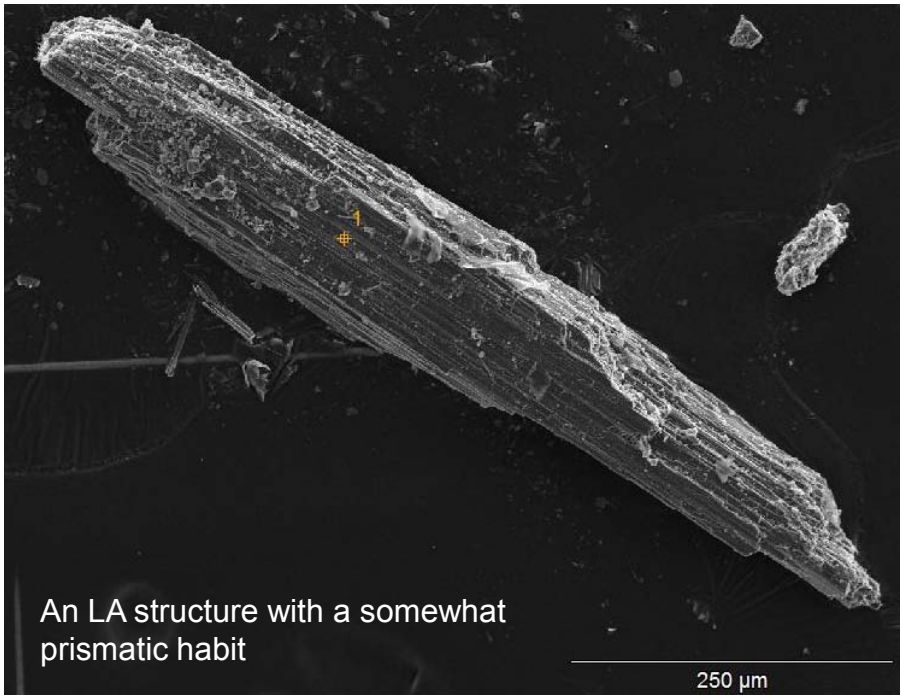



The average aspect ratio of the fibers in this bundle of LA is lower than those of the bundles in the previous examples. However, as seen by SEM, the bundle still splits readily into many small fibers.

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




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




An LA structure with a somewhat prismatic habit

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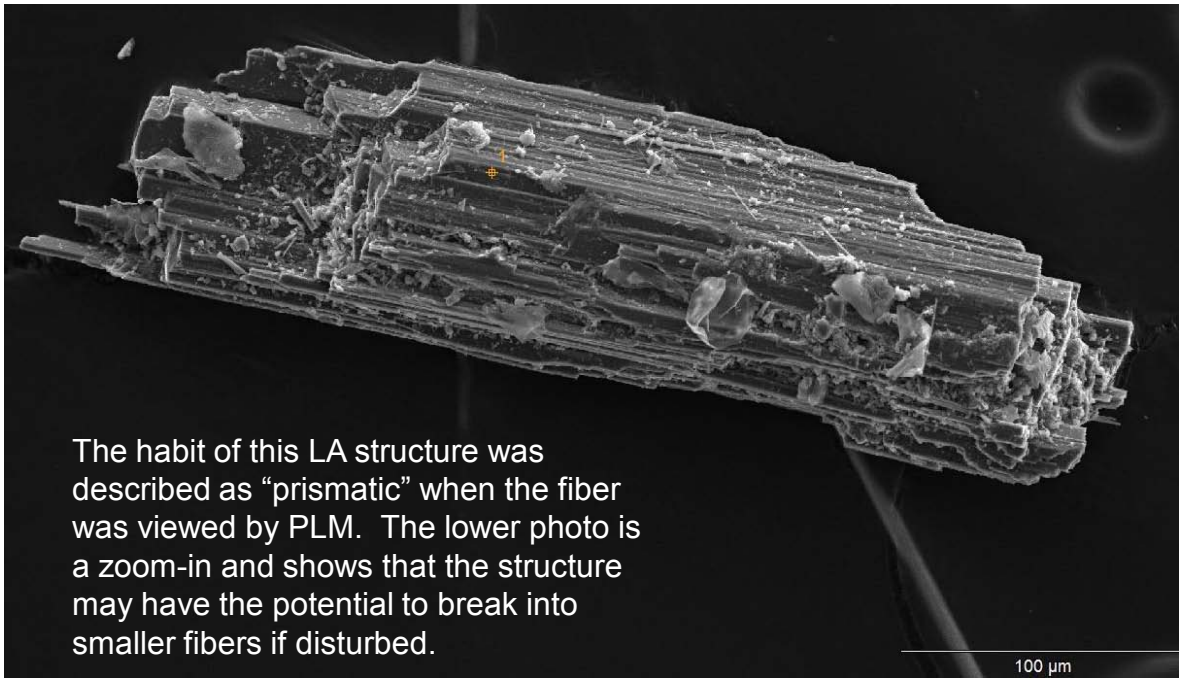
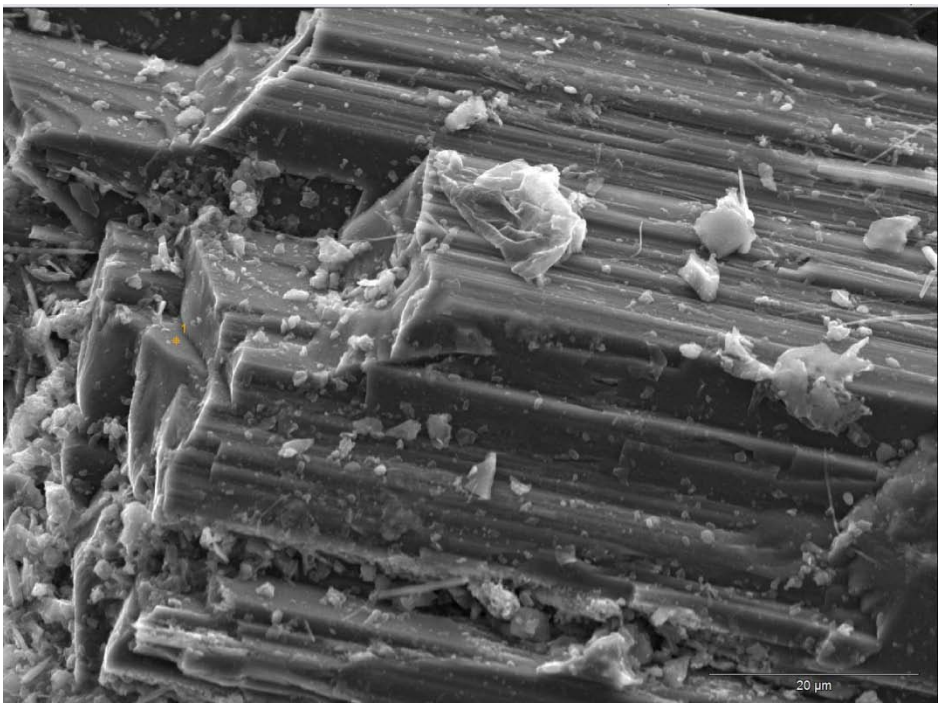


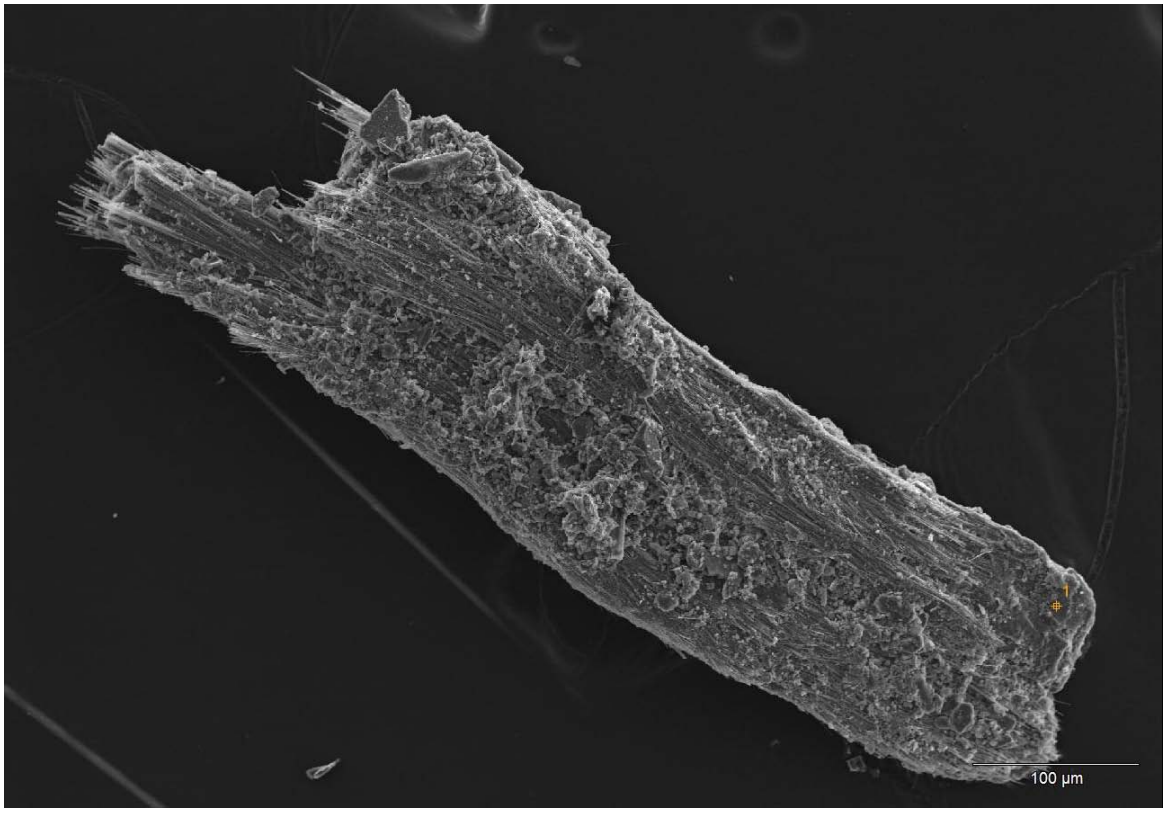

The habit of this LA structure was described as “prismatic” when the fiber was viewed by PLM. The lower photo is a zoom-in and shows that the structure may have the potential to break into smaller fibers if disturbed.




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.

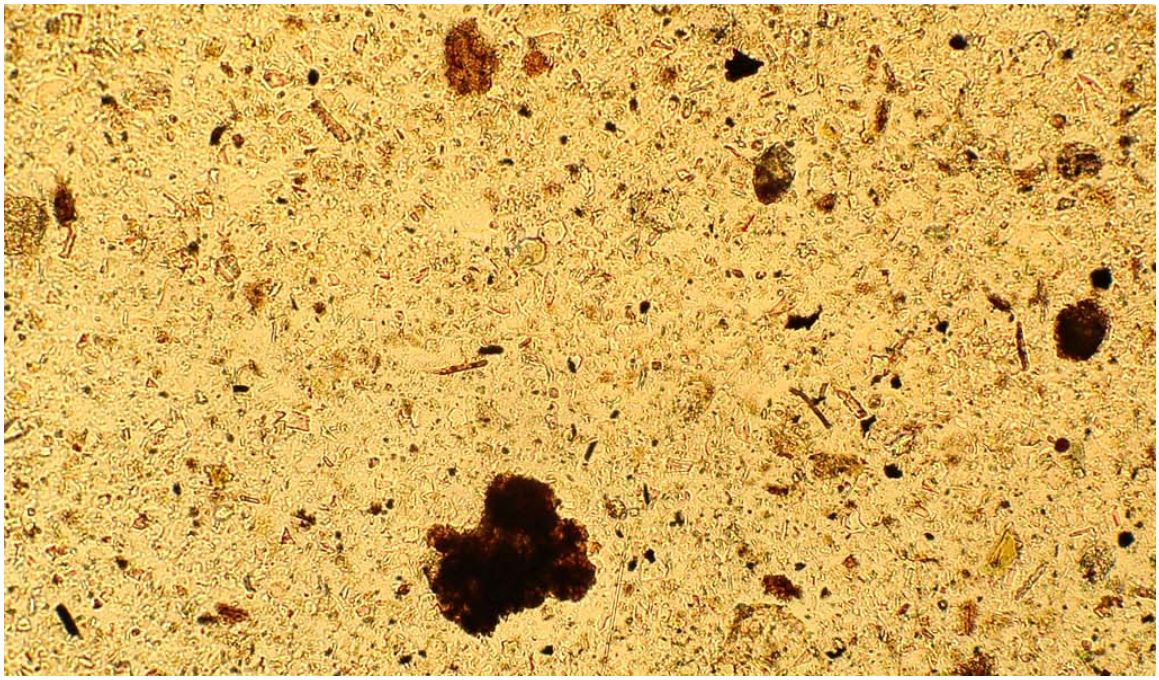
Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.

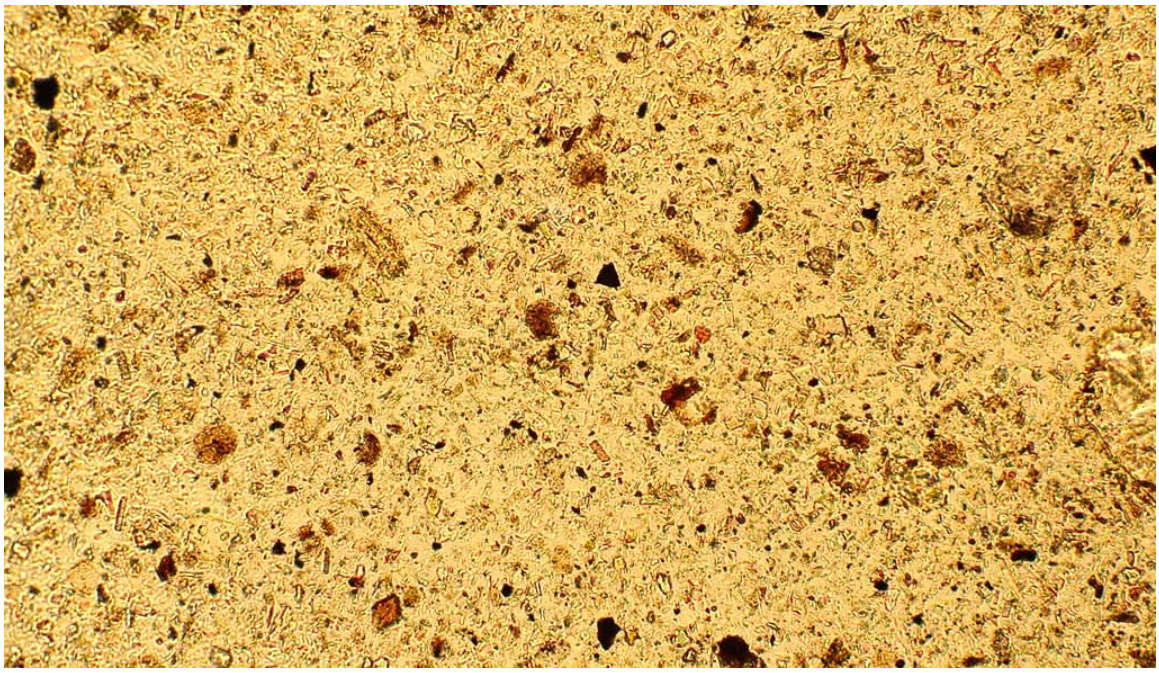
# ATTACHMENT 7

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

# 0.2% Libby Amphibole

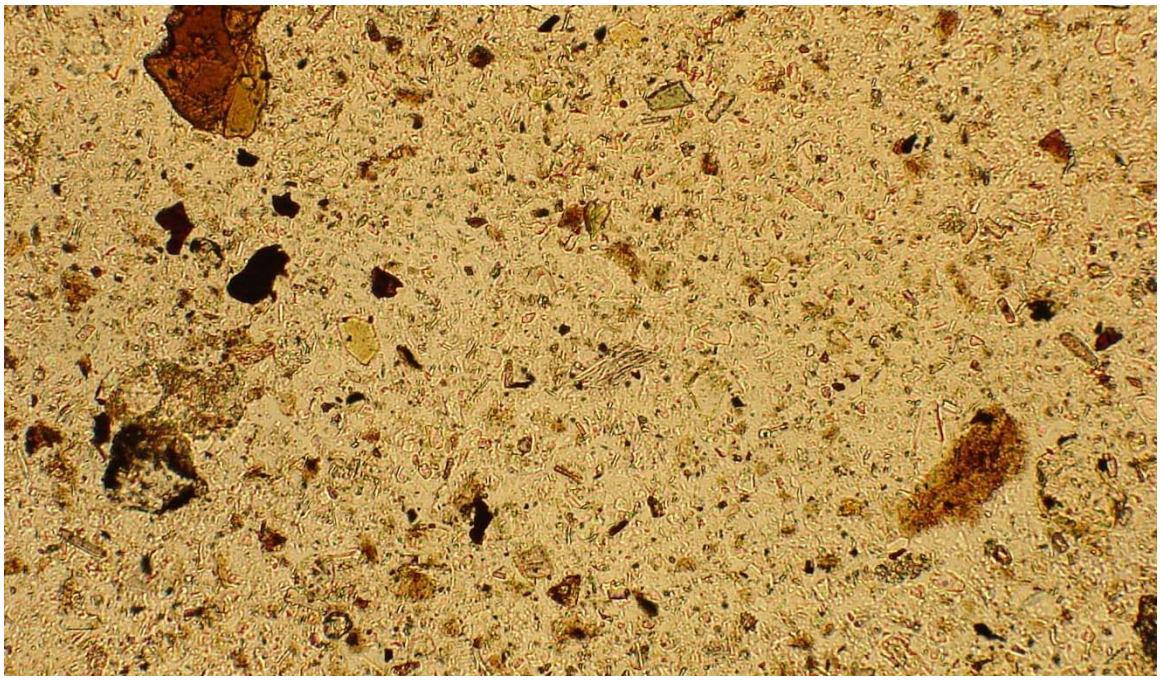
Photomicrographs of representative fields of view of the 0.2% Libby Amphibole by weight Controlled PE Reference Material. All photos taken at 100x, plane light in 1.55 refractive index oil. Width of each picture is approximately 1,500 microns.

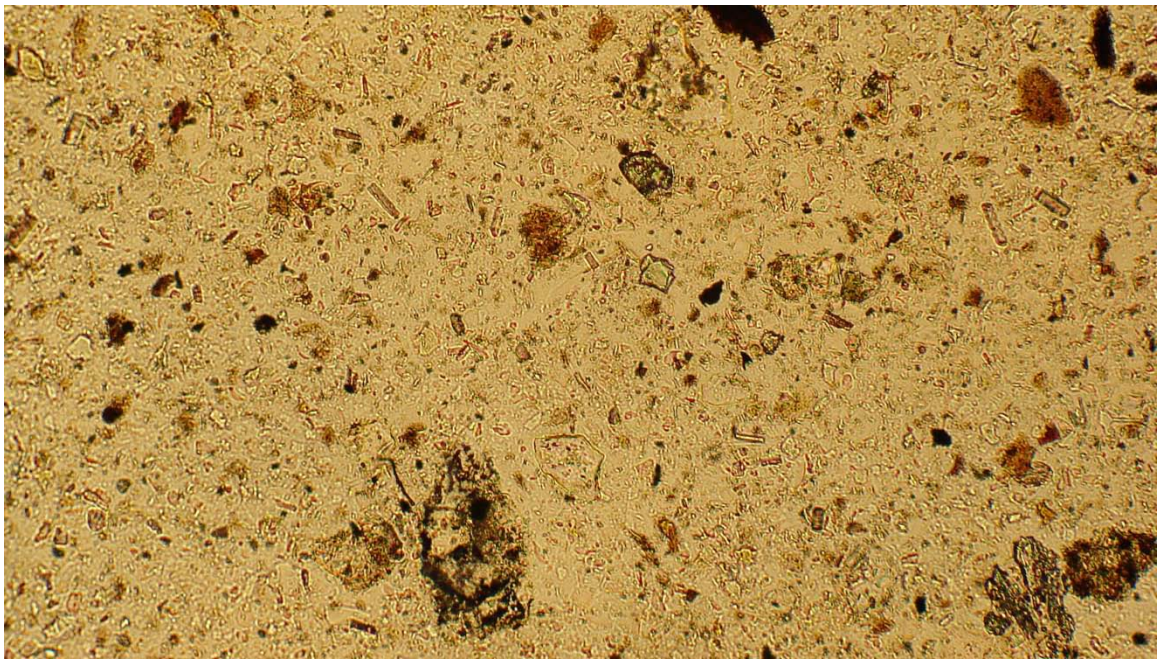




# 0.2% Libby Amphibole

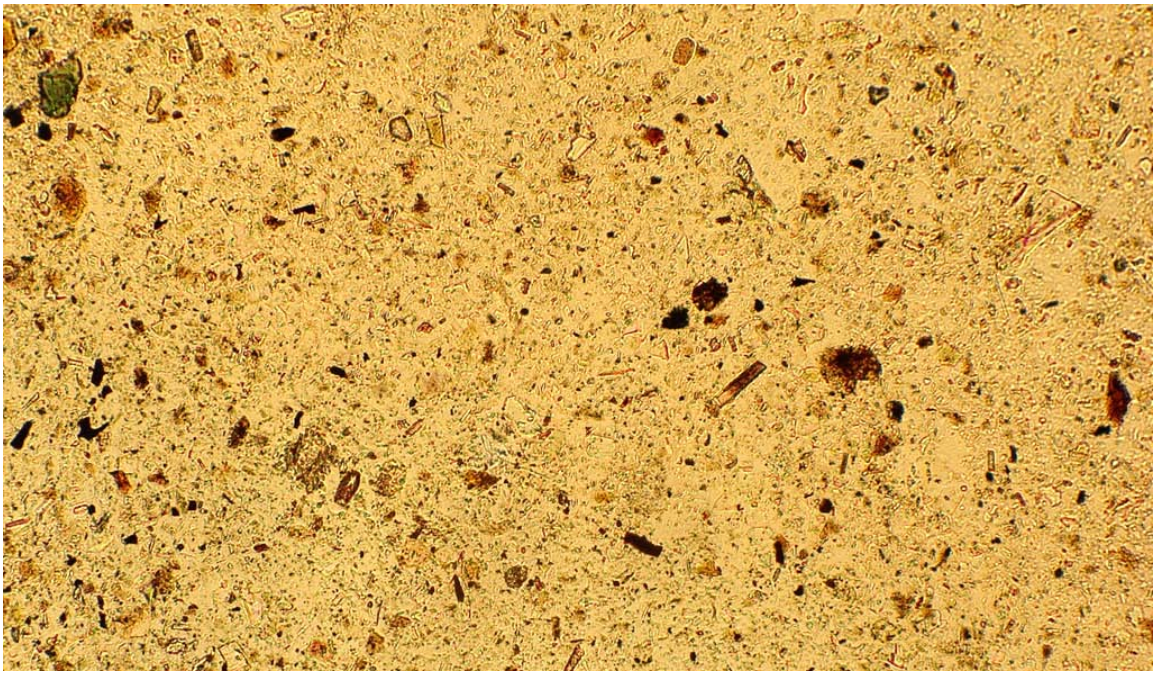
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

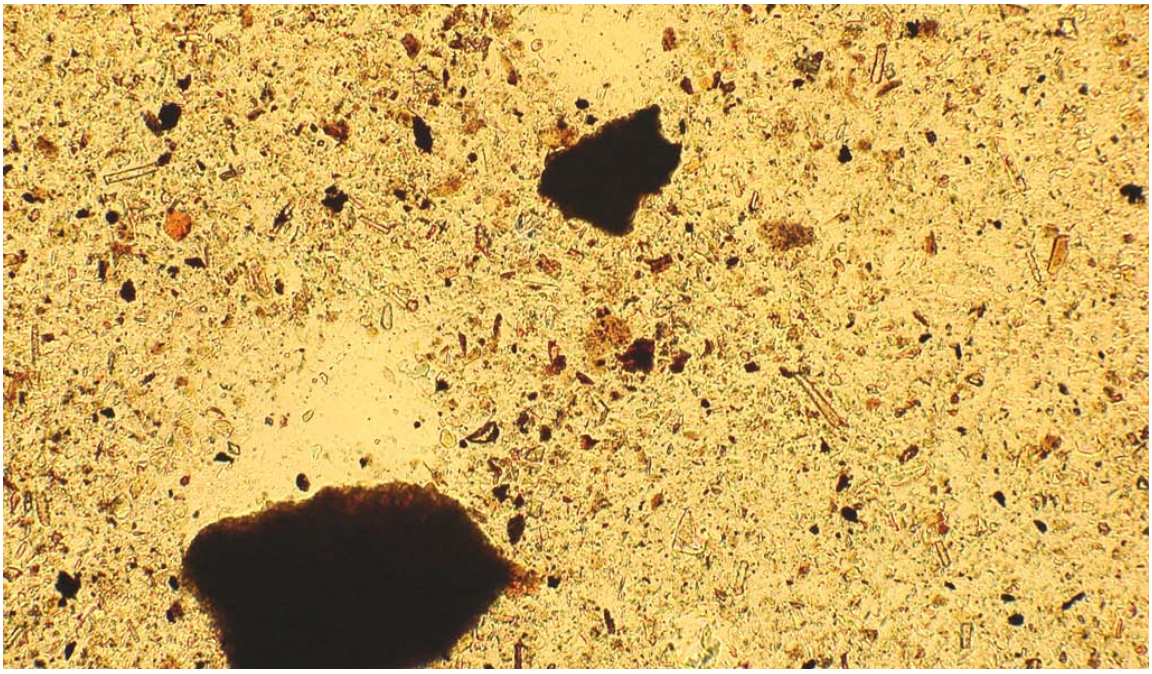




# 0.2% Libby Amphibole

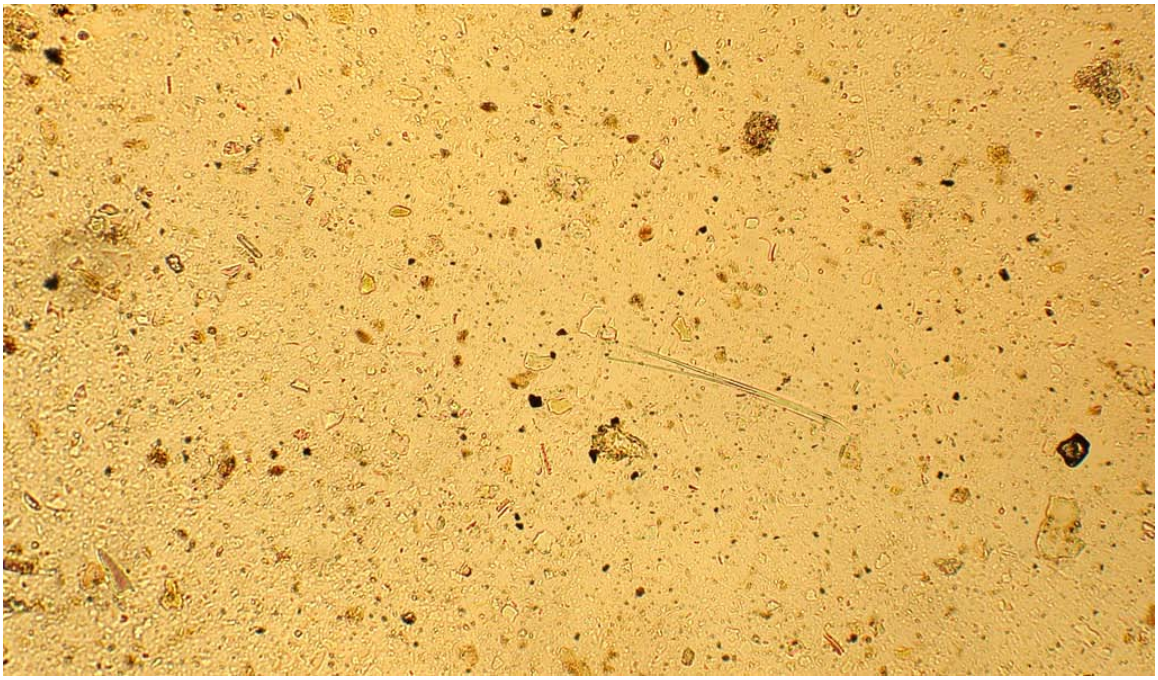
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

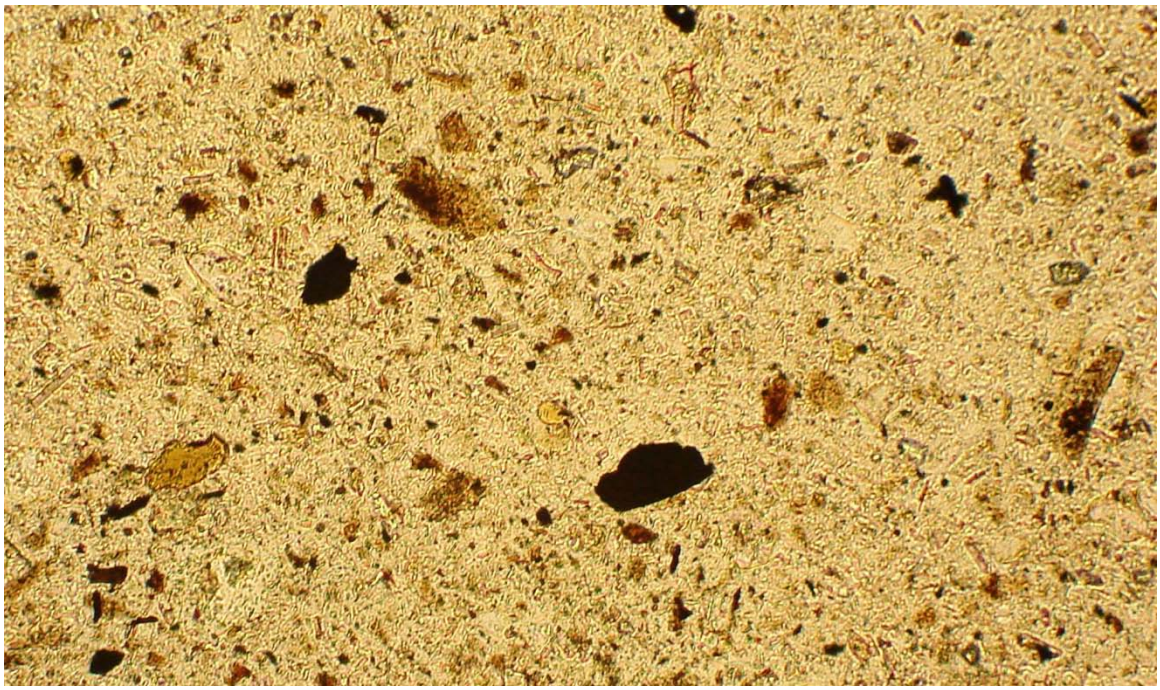




# 0.2% Libby Amphibole

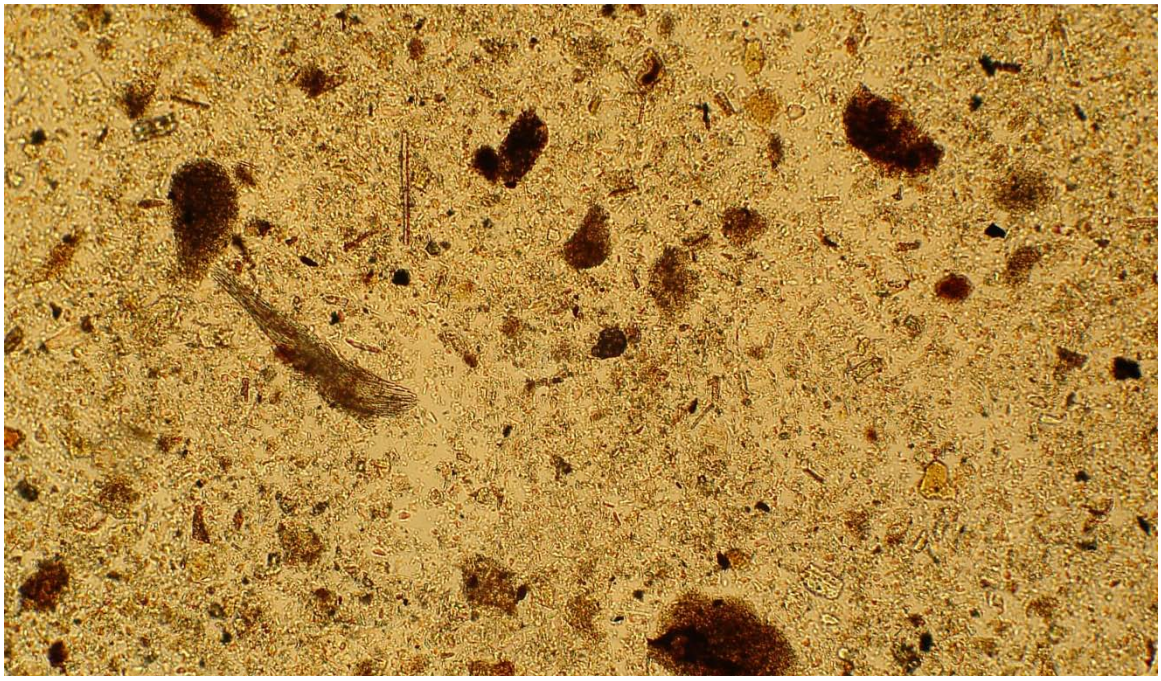
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

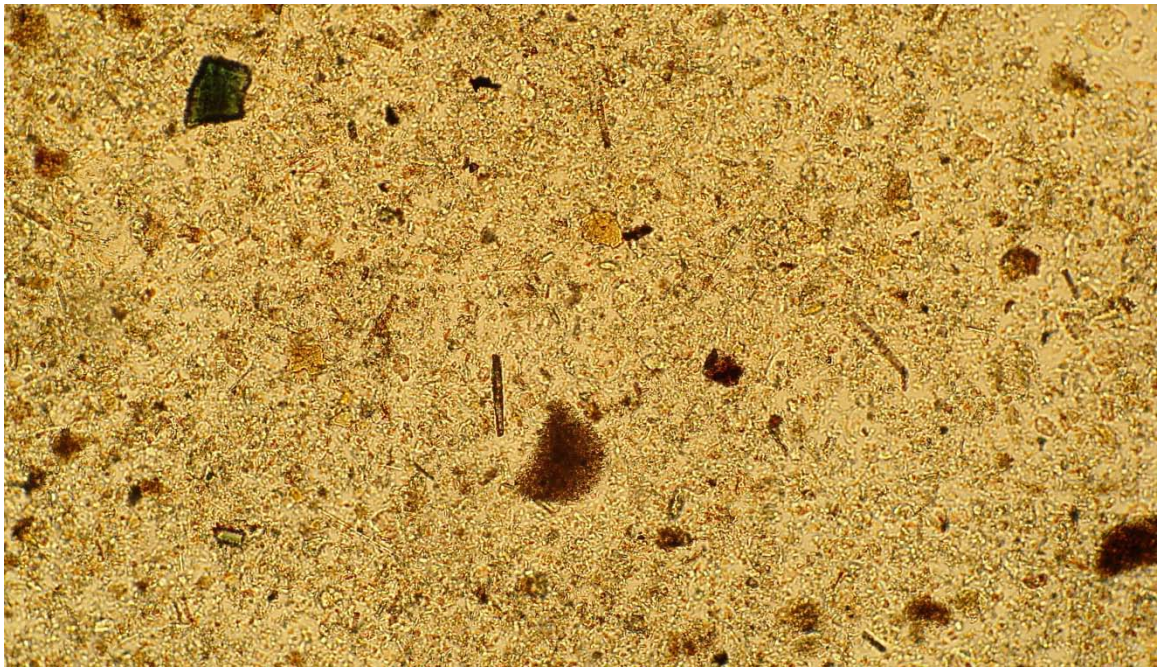




# 1.0% Libby Amphibole

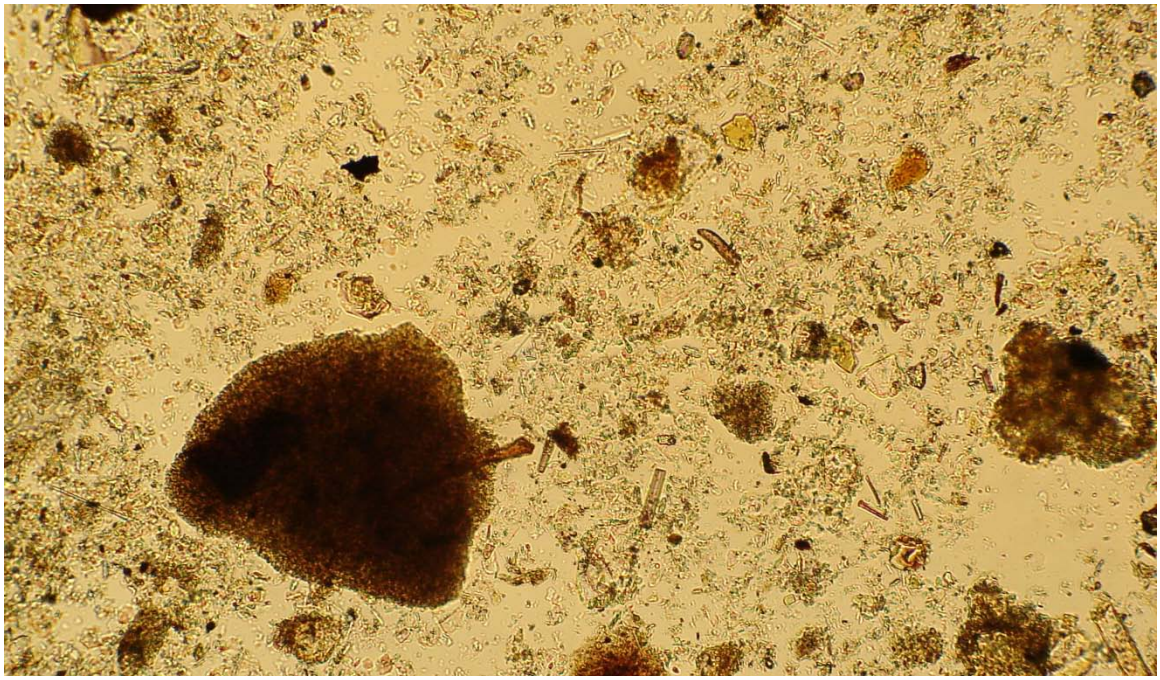
Photomicrographs of representative fields of view of the 1.0% Libby Amphibole by weight Controlled PE Reference Material. All photos taken at 100x, plane light in 1.55 refractive index oil. Width of each picture is approximately 1,500 microns.

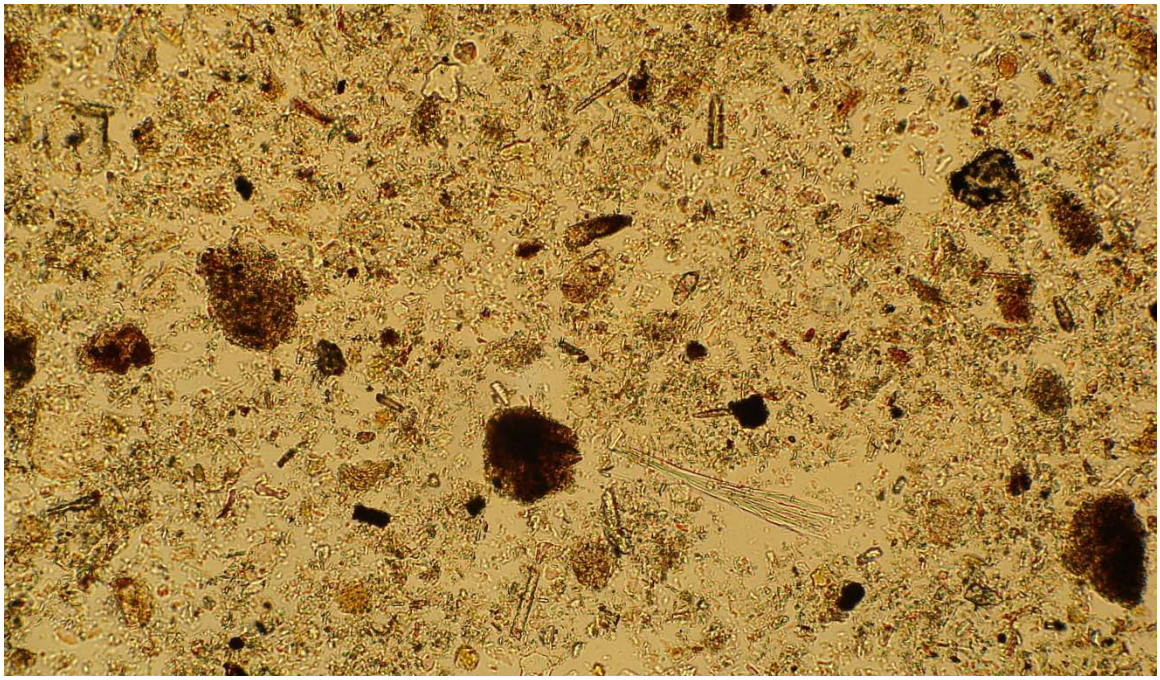




# 1.0% Libby Amphibole

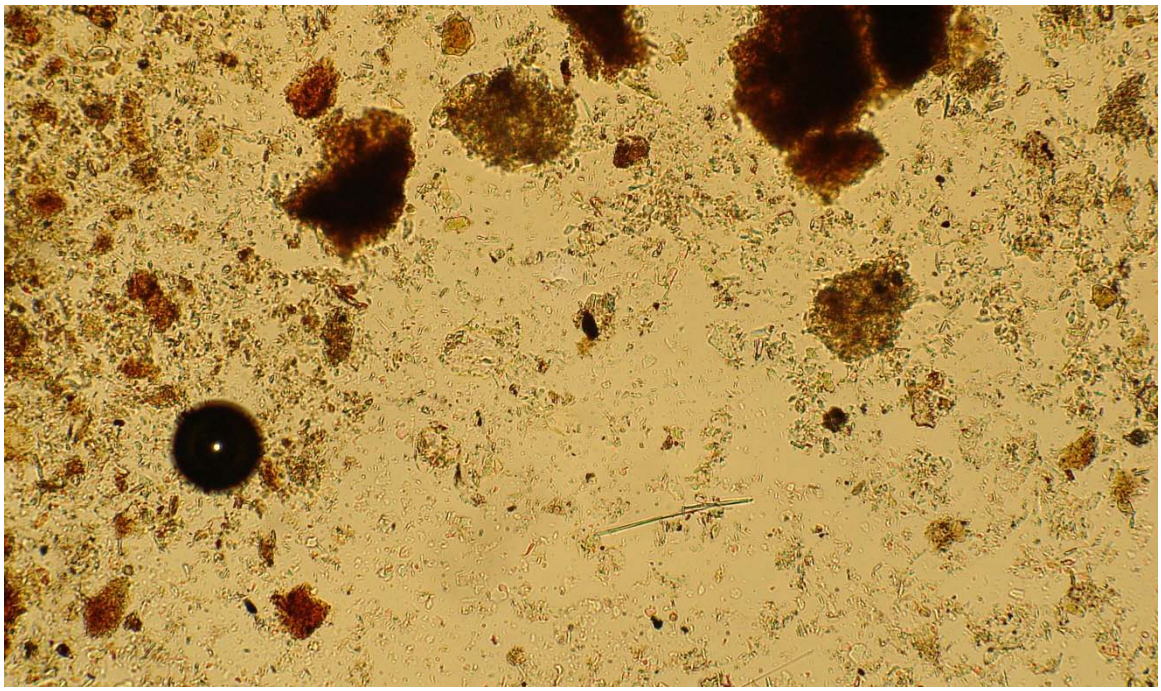
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

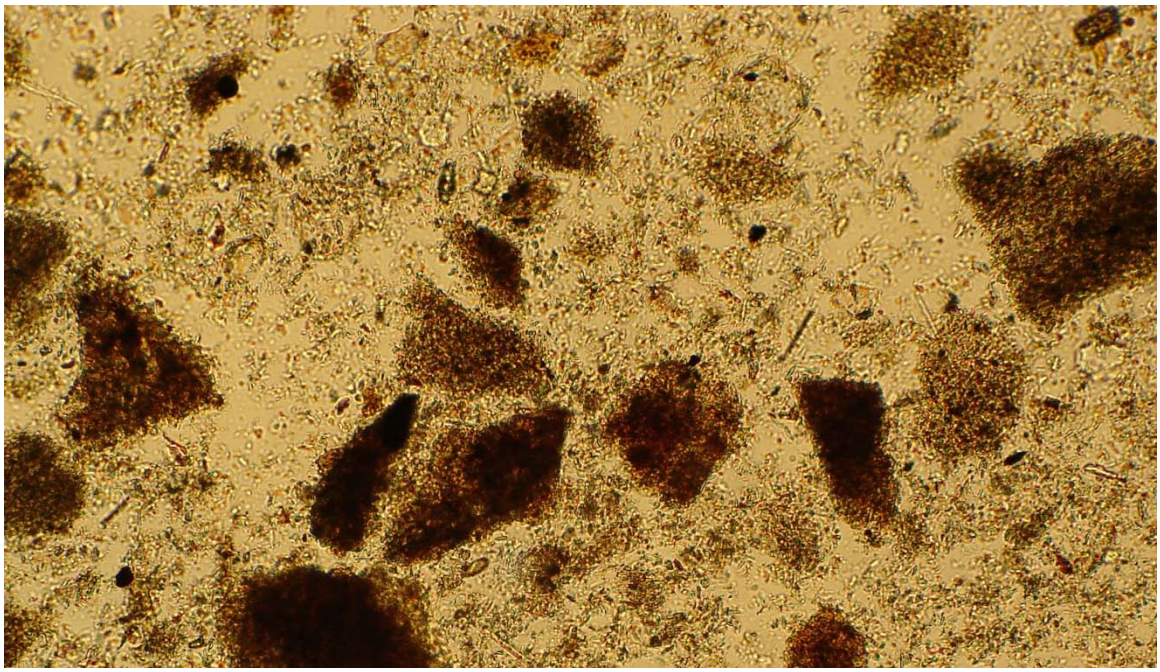




# 1.0% Libby Amphibole

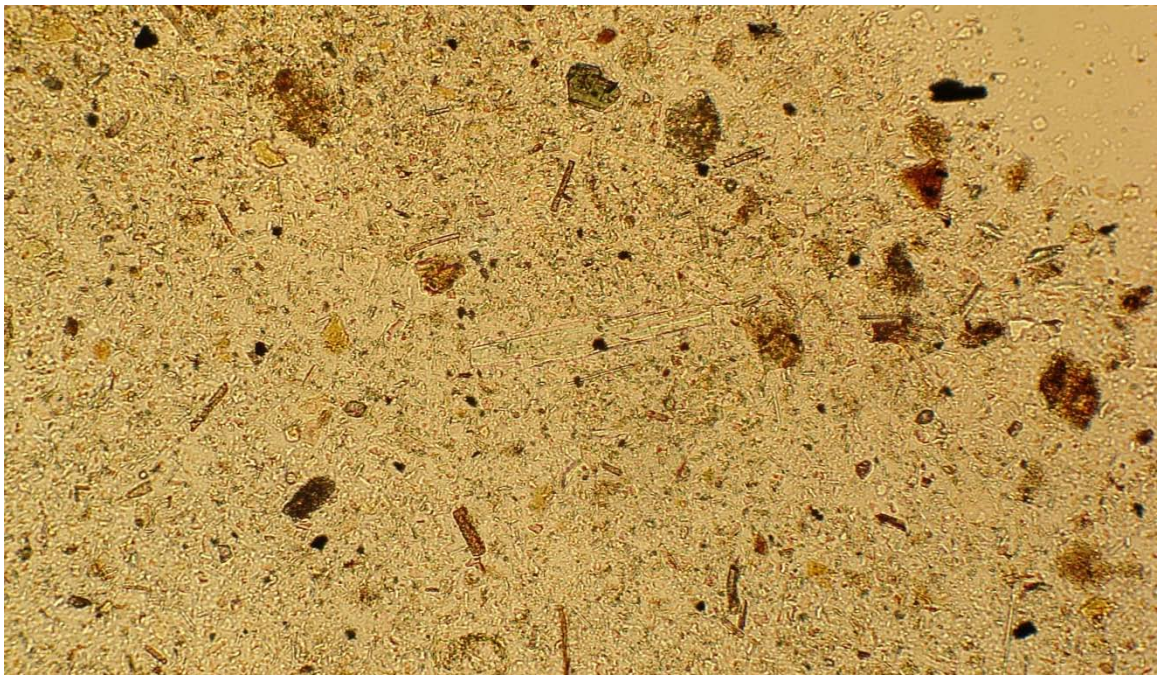
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

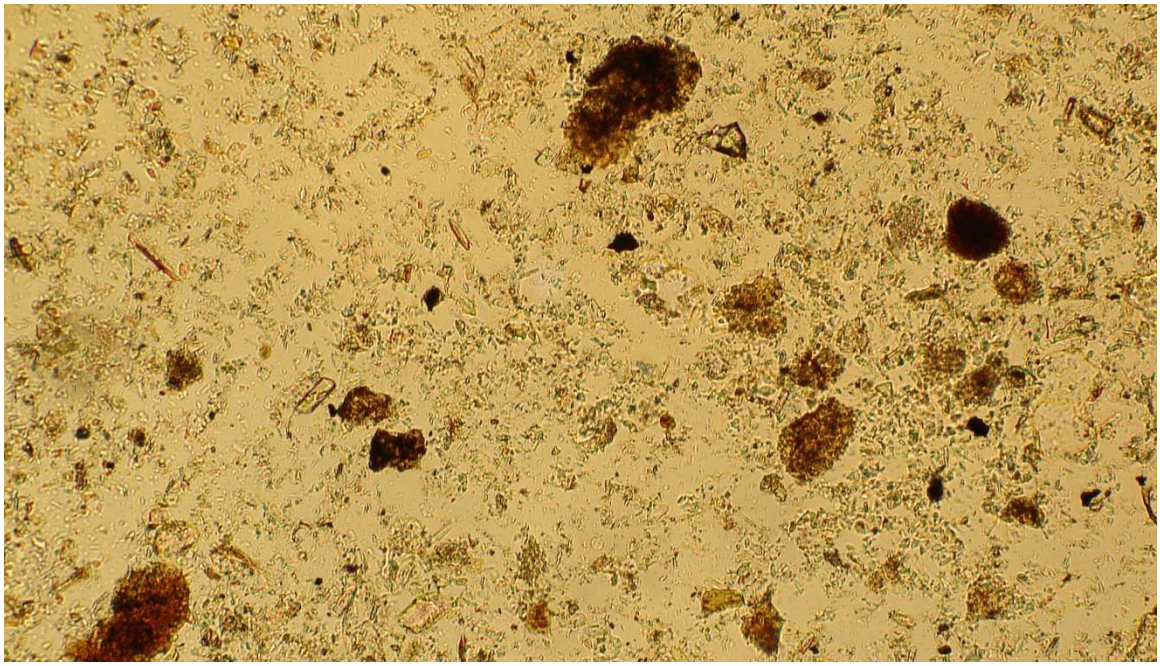




# 1.0% Libby Amphibole

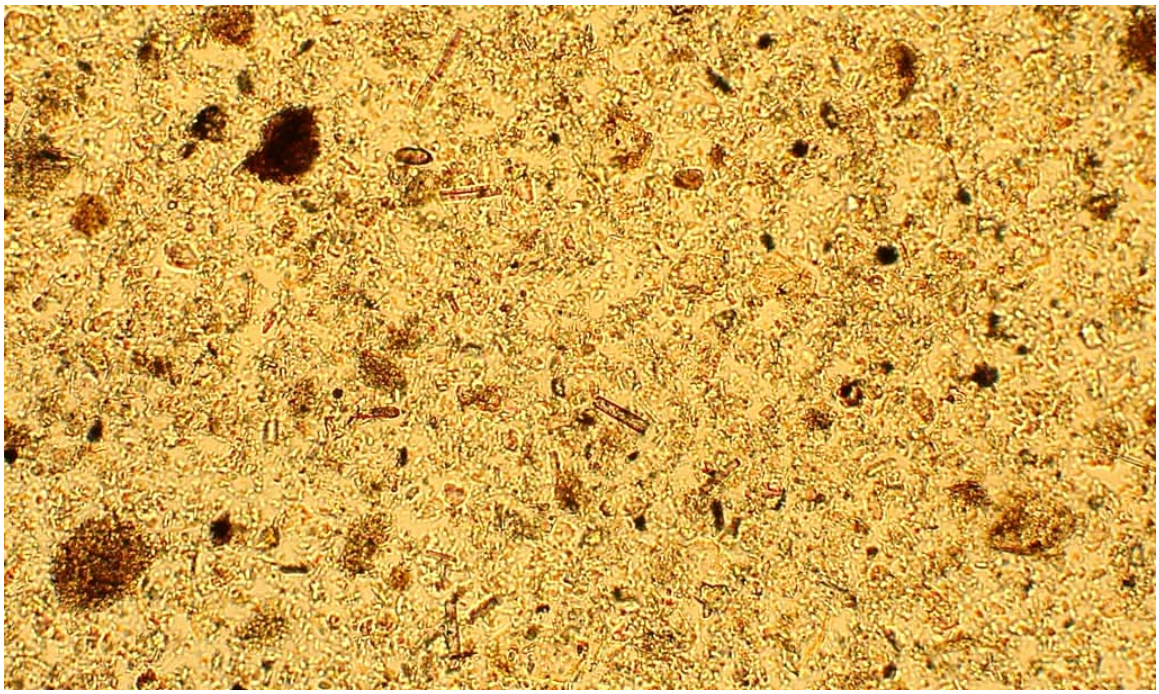
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

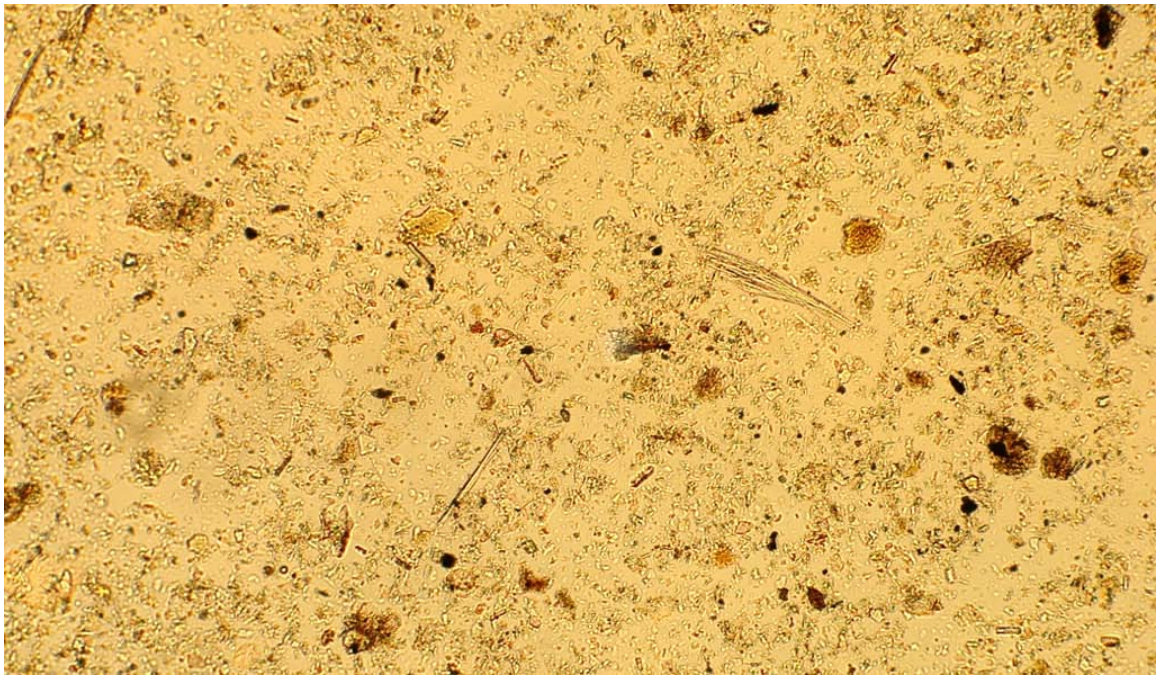




# 1.0% Libby Amphibole

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

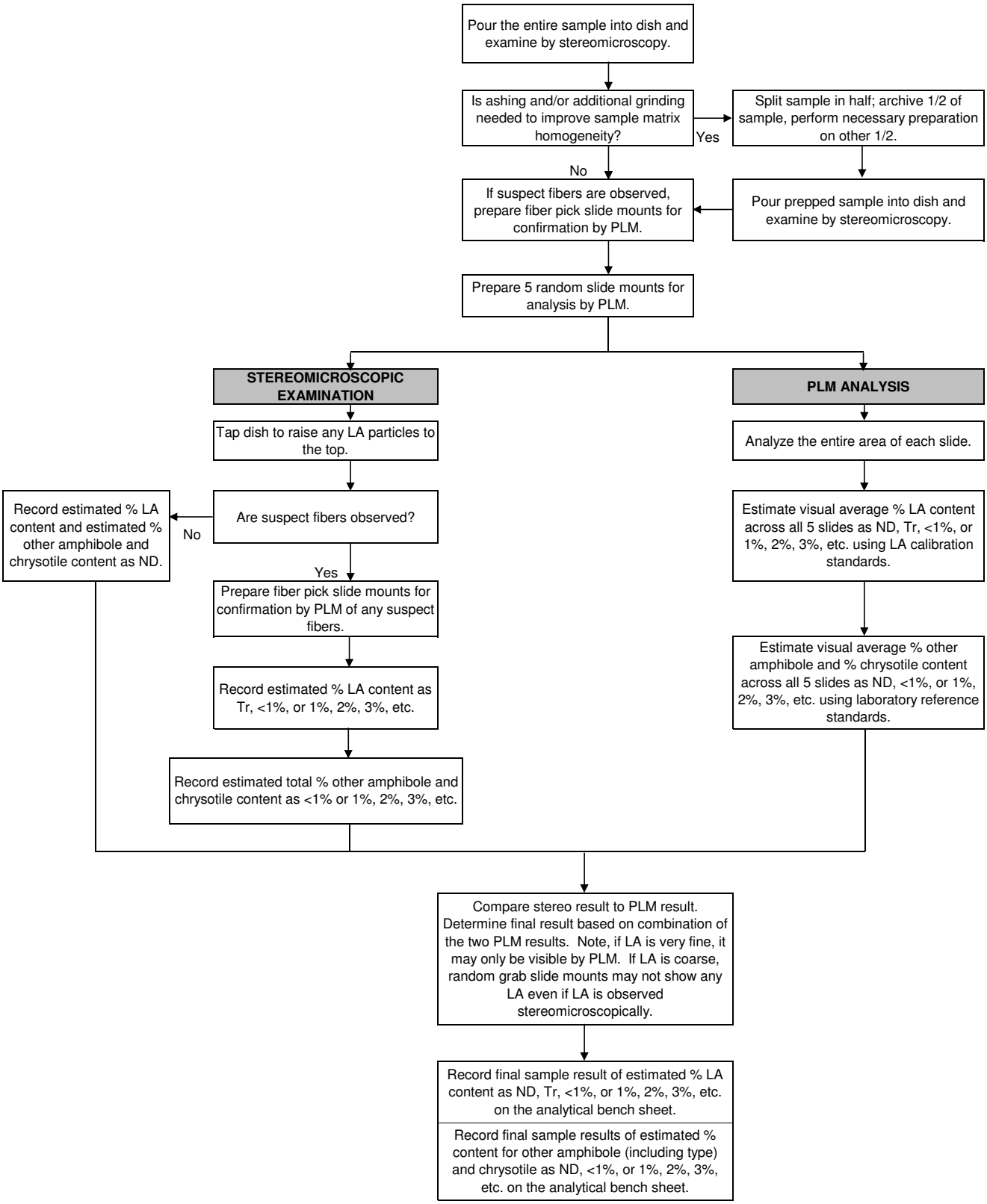




# ATTACHMENT 8

## Flow Chart for Determining Asbestos Content by Complementary Use of Stereomicroscopy and PLM Visual Estimation

# Flow Chart for Determining Asbestos Content by Complementary Use of Stereomicrosopic Examination and PLM Visual Estimation



# ATTACHMENT 9

## Becke Line Chart by F. D. Bloss

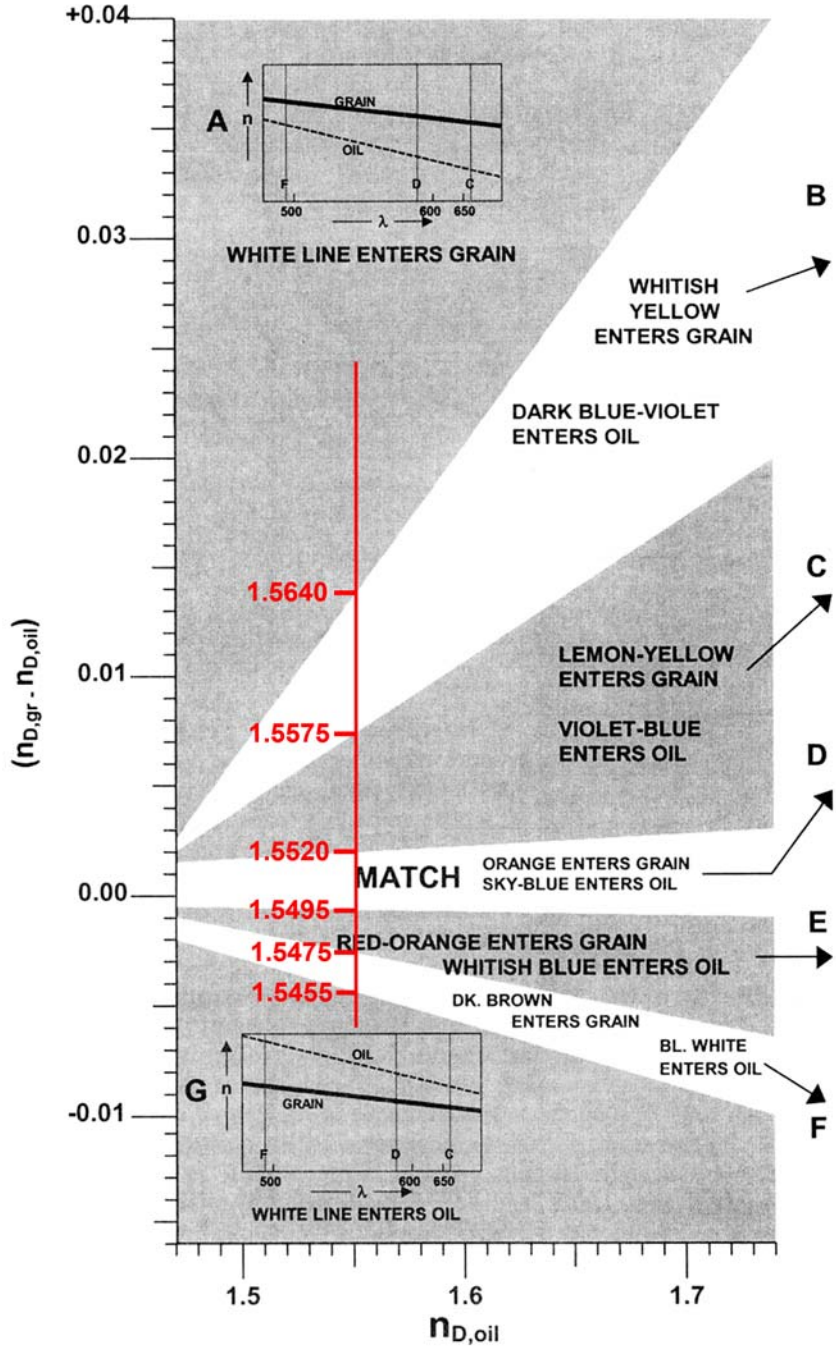



**Figure 5-9.** Use of the colors observed for the Becke lines or oblique illumination shadows to determine the amount by which $n_D$ for the solid exceeds $n_D$ for the liquid (if Cargille Series A or B liquids are being used). The Becke line movements are those observed when the objective is raised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

# Appendix B
# Troy Field Office Modification Records

# Record of Modification

to the

Troy Sampling and Quality Assurance Project Plan

Field Activities

TFO-00001

***Instructions to Requester:*** **Fax to contacts at bottom of form for review and approval.**

**File approved copy with Data Manager at the Troy Field Office (TFO).**

**Data Manager will maintain legible copies in a binder that can be accessed by TFO personnel.**

**If Modification is Temporary for a single Parcel, Data Manager will scan this and place in parcel's electronic file.**

**Project Work Plan/QAPP** (check one)**:**

- ● Outdoor Ambient Air Study Work Plan
- O Other (Title and approval date):_____

**Site-Specific Guidance/SOP:**

Title___NA_____ Number/Revision): _NA_____

Requester: **Catherine LeCours** Title: **Project Manager**

Company: **DEQ/Tetra Tech** Date: **March 3, 2010**

Description of Modification (attach additional sheets if necessary, state section and page numbers of each document that are affected by the proposed modification): **Sections 4.3.3 and 4.4.7 in the *Final Remedial Investigation Work Plan Outdoor Ambient Air Study – Operable Unit Number 7 of the Libby Asbestos Superfund Site.*** These Sections reference the use of .45 micrometer (µm) pore size, 25-millimeter diameter mixed cellulose ester filter cassettes for air sample collection during the Operable Unit 7 (Troy) ambient air study. The choice of .45 µm filter pore size was originally made to coincide with the EPA Asbestos Sampling SOP #2015, Section 5.2 - Filter Cassettes, Paragraph 5.2.1 - TEM Cassette Requirements, which recommends "The cassette shall be loaded with an MCE filter of pore size 0.45 µm, and supplied from a lot number which has been qualified as low background for asbestos determination". However, sampling at high air volumes can create excessive filter loading which may lead to air pump faults. This problem is greater using the finer-mesh 0.45 µm filter pore size and may be reduced by using an alternative 0.8 µm pore size filter.

Prior to beginning the Operable Unit 7 Ambient Air Study, Tetra Tech discovered that the 0.8 µm filter was used for TEM air monitoring in Libby during the Operable Unit 4 Ambient Air Study. Tetra Tech also selected 0.8 µm filters for the Operable Unit 7 ambient air sampling to minimize sample pump faults due to the high air volumes being collected (approximately 14,400 liters was typically collected during the Libby study). The Operable Unit 7 study has proposed to collect between 14,400 and 21,600 liters of air per sample which increases the likelihood of pump faults due to filter loading; therefore, upon discovering the protocol that was used during the Operable Unit 4 study, Tetra Tech decided to alter the procedure during the Operable Unit 7 study as well.

Provided below are the Operable Unit 4 Work Plan sections that reference the use of the 0.8 µm filter pore size. The text is from the *Summary of Outdoor Ambient Air Monitoring For Asbestos At The Libby Asbestos Site - Libby, Montana (October 2006 to June 2008).*

2.1.2 Year 1 Sampling Protocol

Filter Type

Samples were collected using 25-millimeter diameter, 0.8 µm pore size mixed cellulose ester (MCE) filter cassettes. In order to investigate whether the choice of pore size is an important determinant of observed concentrations, samples using 0.45 µm pore size filters were collected intermittently at selected stations. These stations were selected so that sampling stations from the each study area were represented.

4.5 Effect of Pore Size (0.8 µm vs. 0.45 µm)

Table 4-3 presents a comparison of 20 sample pairs matched on collection time, location, and height, but with differing filter pore sizes (0.45 µm vs. 0.8 µm). Results for each sample pair were compared using the Poisson ratio test (Nelson 1982). As seen, there were no pairs that were statistically different from each other for total LA at the 95% confidence level, although there was one pair that was different for chrysotile ($p < 0.05$). These results are consistent with the hypothesis that filter pore size has no substantial effect on the retention or analysis of LA structures. The basis for the one sample that was different for chrysotile is uncertain. However, because the difference was quite large, it seems unlikely that the reason could be differences in filter retention only.

Field Sampling Data Sheet where Modification is documented (attach associated correspondence): **N/A**

Potential Implications of Modification: This modification will not impact the air sampling protocol for Operable Unit 7, nor should it affect the analytical protocol.

Duration of Modification (Check one):

O Temporary

Date(s):_____ Station Number-_____

TA-_____

- Permanent (Proposed Text Modification Section) Effective Date: Throughout the duration of the Operable Unit 7 *Outdoor Ambient Air Study*

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): Sections 4.3.3 and 4.4.7 in the *Final Remedial Investigation Work Plan Outdoor Ambient Air Study – Operable Unit Number 7 of the Libby Asbestos Superfund Site*: **(Only changes are shown)**

4.3.3 Inventory and Procurement of Equipment and Supplies

- **Sample media – ~~0.45~~ 0.8 micrometer (µm) pore size, 25-millimeter diameter mixed cellulose ester filter cassettes**

4.4.7 Filter Type – Pore Size

**Samples will be collected using 25-millimeter diameter, ~~0.45~~ 0.8 µm pore size, as specified in EPA SOP 2015.**

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

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

Technical Review and Approval: _____ Date:_____
*(DEQ Project Manager or designate)*

EPA Review and Approval: _____ Date:_____
*(USEPA RPM or designate)*

## DATA QUALITY INDICATOR DEFINITIONS

***Reject*** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect 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.

# Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO-00002

***Instructions to Requester:*** **Fax to contacts at bottom of form for review and approval.**
**File approved copy with Data Manager at the Troy Field Office (TFO).**
**Data Manager will maintain legible copies in a binder that can be accessed by TFO personnel.**
**If Modification is Temporary for a single Parcel, Data Manager will scan this and place in parcel's electronic file.**

**Project Work Plan/QAPP** (check one)**:**

- ● Outdoor Ambient Air Study Work Plan
- O Other (Title and approval date):_____

**Site-Specific Guidance/SOP:**
Title___NA_____ Number/Revision): _NA_____

Requester: **Catherine LeCours** Title: **Project Manager**
Company: **DEQ** Date: **March 4, 2010**

Description of Modification (attach additional sheets if necessary, state section and page numbers of each document that are affected by the proposed modification): **Section 4.4.4 in the *Final Remedial Investigation Work Plan Outdoor Ambient Air Study – Operable Unit Number 7 of the Libby Asbestos Superfund Site*** references collecting approximately 3 liters per minute over the entire 5-day (120-hour) sampling event to achieve the target volume of 21,600 liters during the Troy ambient air study. A target volume of 21,600 liters will minimize the number of grid openings counted during laboratory analysis, reducing the time and cost of analysis while meeting the target analytical sensitivities.

However, sampling at high air volumes can create excessive filter loading which may lead to air pump faults, require additional analytical costs due to indirect sample analysis, or in some cases yield samples that can not be analyzed resulting in a loss of data. Therefore, the work plan reserved the option to "adjust this target volume based on changes in the target analytical sensitivities, sample results, or filter loading issues".

**OU7 Overloading Summary:**

Tetra Tech requested that analytical results for the first sampling period be obtained as soon as possible to determine if the specified pump flow rate of 3.0 liters per minute was excessive and filter overloading would result. Three samples from Period 1 yielded overloaded cassettes while the remaining five cassettes were not overloaded. Overloaded cassette filters were noted to have a black "soot-like" substance on the filter. The three overloaded cassettes were collected at stations T-4, T-4QC and T-5 (the DEQ Office, DEQ Office QC station, and the County Shops station). Tetra Tech consulted with the DEQ and resolved that the sample stations with the overloaded filter cassettes were likely located in dusty areas and near high traveled roads. Tetra Tech recommended relocating the sampling boxes at these locations to positions farther away from the main roads. Tetra Tech also reduced the sample flow rates from 3.0 to 2.0 liters per minute. These changes were made prior to starting sample Period 4.

The three overloaded cassettes were analyzed using a secondary dilution process. Additional grid openings would need to be counted in order to achieve the analytical sensitivity and would result in increased analytical time and costs. The number of grid openings to be counted would increase from 45 to either 73 or 146 to reach required sensitivity levels.

The initial analytical results for Sample Period 1 were received on December 8, 2009, just prior to the start of Sample Period 5. The other Period 1 sample results did not require secondary dilution; therefore Period 5 flow rates were returned to 3.0 liters per minute.

The DEQ recommended that rush analysis be completed for the Period 5 samples to help evaluate if relocating the sample boxes at T-4, T-4QC, and T-5 helped avoid filter overloading at these locations. No overloaded cassette filters were identified for any of the Period 5 samples. As a result, the new locations of the boxes and the 3.0 liters per minute flow rate was established for subsequent sampling periods.

In order to evaluate ongoing sampling activities and to check for potential overloading, the DEQ recommended that additional samples be sent in for analysis prior to the ESAT laboratory becoming operational. Samples from Period 11 were forwarded to the EMSL Libby mobile lab for analysis. Several cassette filters from Period 11 were subsequently found to be overloaded.

As a result of the periodic overloading of sample filters, DEQ directed Tetra Tech reduce the sample flow rates from 3.0 to 2.0 liters per minute for the remainder of the OU7 ambient air monitoring project. This modification was made as a permanent procedural change beginning in Sample Period 13.

Field Sampling Data Sheet where Modification is documented (attach associated correspondence): **N/A**

Potential Implications of Modification: Modifications to sampling protocol involve reprogramming sampling pumps to a flow rate of 2.0 liters per minute. Analytical protocol will be impacted as additional grids will be counted to meet analytical sensitivity requirements for the reduced air flow.

Duration of Modification (Check one):

O Temporary

Date(s):_____ Station Number-_____

TA-_____

- Permanent (Proposed Text Modification Section) Effective Date: March 4, 2010

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): Section 4.4.4 in the *Final Remedial Investigation Work Plan Outdoor Ambient Air Study – Operable Unit Number 7 of the Libby Asbestos Superfund Site:*

**4.4.4 Collection Interval and Flow Rates**

To ensure that target analytical sensitivities can be achieved, the target volume of air to be collected for each sample will be ~~21,600~~ 14,400 liters. Tetra Tech may adjust this target volume based on changes in the target analytical sensitivities,

sample results, or filter loading issues. A target volume of ~~21,600~~ 14,400 liters will ~~minimize~~ optimize the number of grid openings counted during laboratory analysis, reducing the time and cost of analysis while meeting the target analytical sensitivities

The number of grid openings to be counted for this volume of air will be ~~45~~ 67 and was calculated using the equation provided in Section 6.1 of SOP No EPA-LIBBY-09 (rev 1). This equation and the spreadsheet used to calculate grid openings are shown in Appendix F.

To help ensure that samples capture long-term averages, each sample will be collected over a 5-day (120-hour) interval. Thus, the target flow rate is approximately ~~3~~ 2.0 liters per minute over the entire sampling event to achieve the target volume of ~~21,600~~ 14,400 liters.

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

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

Technical Review and Approval: _____ Date:_____
*(DEQ Project Manager or designate)*

EPA Review and Approval: _____ Date:_____
*(USEPA RPM or designate)*

**DATA QUALITY INDICATOR DEFINITIONS**

***Reject*** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect 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.

# Record of Modification

to the

Troy Sampling and Quality Assurance Project Plan

Field Activities

TFO-00003

***Instructions to Requester:*** **Fax to contacts at bottom of form for review and approval.**
**File approved copy with Data Manager at the Troy Field Office (TFO).**
**Data Manager will maintain legible copies in a binder that can be accessed by TFO personnel.**
**If Modification is Temporary for a single Parcel, Data Manager will scan this and place in parcel's electronic file.**

**Project Work Plan/QAPP** (check one)**:**

- ● Outdoor Ambient Air Study Work Plan
- O Other (Title and approval date):_____

**Site-Specific Guidance/SOP:**
Title___NA_____ Number/Revision): _NA_____

Requester: **Catherine LeCours** Title: **Project Manager**
Company: **DEQ** Date: **October 11, 2010**

Description of Modification (attach additional sheets if necessary, state section and page numbers of each document that are affected by the proposed modification): ***Section 4.4.2 in the Final Remedial Investigation Work Plan Outdoor Ambient Air Study – Operable Unit Number 7 of the Libby Asbestos Superfund Site*** provides a general description of proposed ambient air sampling station locations. "As previously discussed, the predominant winds in Troy tend to flow in southeast and northwest directions, following the river corridor in which Troy is located. Two sampling stations (one each) will be placed in close proximity to the northwest and southeast boundaries of OU7. This will ensure that there are upwind and downwind sample collection stations for both directions the wind is blowing. Two stations (one each) will also be located on the northwest and southeast borders of downtown Troy in order to have upwind and downwind sample stations in the area with the highest population density. One sample station will be placed at the DEQ Troy Information Center in downtown Troy to measure LA concentrations in Troy. One station will be placed in the Kootenai Vista area in the northern portion of OU7 and the last station will be placed along or near Iron Creek Road in the southwestern portion of OU7." The ambient air monitoring stations will be relocated for the second year for more comprehensive coverage of the four "air zones" identified in OU7. This will provide additional data in support of human health risks related to ambient air exposure.

Field Sampling Data Sheet where Modification is documented (attach associated correspondence): **N/A**

Potential Implications of Modification: Re-locating the ambient air sampling stations within the four "air zones" will further support human health risk assessment for OU7. Moving the stations will not impact analytical protocol and is not anticipated to have any impact on analytical results.

Duration of Modification (Check one):

O Temporary

Date(s):_____ Station Number-_____

TA-_____

- Permanent (Proposed Text Modification Section) Effective Date: November 1, 2010

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): ***Section 4.4.2 in the Final Remedial Investigation Work Plan Outdoor Ambient Air Study – Operable Unit Number 7 of the Libby Asbestos Superfund Site and Table 4-2***:

As previously discussed, the predominant winds in Troy flow in southeast and northwest directions, following the river corridor in which Troy is located. ~~Two~~ Three sampling stations ~~(one each)~~ will be placed in new locations in close proximity to the ~~northwest~~ northern (1) and ~~southeast~~ southern (2) boundaries of OU7. This will ensure that there are upwind and downwind sample collection stations for both directions the wind is blowing. Two stations (one each) will be re-located ~~on the northwest~~ near the northern and ~~southeast~~ southern borders of downtown Troy in order to have upwind and downwind sample stations in the area with the highest population density. One sample station will be located in the densely populated area of downtown Troy and a final station will be re-located north of Troy in a developed area along the Kootenai River. ~~One sample station will be placed at the DEQ Troy Information Center in downtown Troy to measure LA concentrations in Troy. One station will be placed in the Kootenai Vista area in the northern portion of OU7 and the last station will be placed along or near Iron Creek Road in the southwestern portion of OU7.~~ Table 4-2 has the rationale for the new ambient air monitoring locations and Figure 4-2 shows the proposed new ambient air monitoring locations.

## TABLE 4-2

## OUTDOOR AMBIENT AIR SAMPLING LOCATIONS

| Station Number | Location* | Purpose |
|---|---|---|
| T1 | Upwind/downwind site near the NW border of OU7 | This site will be used to evaluate LA concentrations at the northernmost boundary of OU7 and confirm if any LA is entering or leaving OU7 |
| T2 | Community exposure site located within small community area NE of the Kootenai River | This site will be used to evaluate LA concentrations at the small community and the ~~middle~~ northern boundary of OU7 |
| T3 | City of Troy northern site | This site will be used to evaluate LA concentrations north of the Troy community |
| T4 | City of Troy population exposure site | This site will be used to evaluate LA concentrations in the Troy community (specifically in the population center). |
| ~~T4QC~~ | ~~City of Troy population exposure site~~ | ~~Co-located sample station of T4~~ |
| T5 | City of Troy southern site | This site will be used to evaluate LA concentrations south of the Troy community |
| T6 | SW upwind/downwind site | This site will be used to evaluate LA concentrations at the southwestern boundary of the OU and confirm if any LA is entering or leaving OU7 |
| T7 | SE upwind/downwind site | This site will be used to evaluate LA concentrations at the southeastern boundary of the OU and confirm if any LA is entering or leaving OU7 |

| Station Number | Location* | Purpose |
|---|---|---|
| TQC | Rotating co-located sampling station to each of the seven sampling locations | Co-located sampling station to evaluate analytical variability at each of the seven station locations |

Notes:

| | | | |
|---|---|---|---|
| LA | Libby Amphibole | SE | Southeast |
| NE | Northeast | SW | Southwest |
| NW | Northwest | OU | Operable Unit |

* Predominant winds in the area blow from the southeast and northwest. Stations on the southeast and northwest boundaries of OU7 will act as upwind and downwind receptors depending on wind direction. A summary of historical meteorological conditions is presented in Section 4.4.1.

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

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

Technical Review and Approval: _____ Date:_____
*(DEQ Project Manager or designate)*

EPA Review and Approval: _____ Date:_____
*(USEPA RPM or designate)*

## 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.

# Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO-00004

***Instructions to Requester:*** **Fax to contacts at bottom of form for review and approval.**
**File approved copy with Data Manager at the Troy Field Office (TFO).**
**Data Manager will maintain legible copies in a binder that can be accessed by TFO personnel.**
**If Modification is Temporary for a single Parcel, Data Manager will scan this and place in parcel's electronic file.**

**Project Work Plan/QAPP** (check one)**:**

- ● Outdoor Ambient Air Study Work Plan
- O Other (Title and approval date):_____

**Site-Specific Guidance/SOP:**
Title___NA_____ Number/Revision): _NA_____

Requester: **Catherine LeCours** Title: **Project Manager**
Company: **DEQ** Date: **October 11, 2010**

Description of Modification (attach additional sheets if necessary, state section and page numbers of each document that are affected by the proposed modification): **Section 5.2 in the *Final Remedial Investigation Work Plan Outdoor Ambient Air Study – Operable Unit Number 7 of the Libby Asbestos Superfund Site*** states "Field co-located samples will be collected from the same location throughout the project and will consist of a co-located sampling station (Station T4QC) to be built approximately seven feet from the proposed sampling station to be located at the DEQ Troy Information Center." For the second year of ambient air sampling this protocol will be changed so that the co-located sampling station will be named "TQC" and will move to a different sampling station during each sampling period.

Field Sampling Data Sheet where Modification is documented (attach associated correspondence): **N/A**

Potential Implications of Modification: Modifications to sampling protocol involve rotating the co-located sampling station among all of the seven ambient air sampling stations. Analytical protocol will not be impacted; however, moving the co-located sampling station will allow evaluation of analytical variability at all seven stations. A minimum of five co-located samples will be collected at each of the stations.

Duration of Modification (Check one):

O Temporary

Date(s):_____ Station Number-_____

TA-_____

- ● Permanent (Proposed Text Modification Section) Effective Date: November 1, 2010

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): **Section 4.4.2 in the *Final Remedial Investigation Work Plan Outdoor Ambient Air Study – Operable Unit Number 7 of the Libby Asbestos Superfund Site (Table 4-2) and Section 5.2 (Paragraph 5):***

# TABLE 4-2

# OUTDOOR AMBIENT AIR SAMPLING LOCATIONS

| Station Number | Location* | Purpose |
|---|---|---|
| T1 | Upwind/downwind site near the NW border of OU7 | This site will be used to evaluate LA concentrations at the northernmost boundary of OU7 and confirm if any LA is entering or leaving OU7 |
| T2 | Community exposure site located within small community area NE of the Kootenai River | This site will be used to evaluate LA concentrations at the small community and the ~~middle~~ northern boundary of OU7 |
| T3 | City of Troy northern site | This site will be used to evaluate LA concentrations north of the Troy community |
| T4 | City of Troy population exposure site | This site will be used to evaluate LA concentrations in the Troy community (specifically in the population center). |
| ~~T4QC~~ | ~~City of Troy population exposure site~~ | ~~Co-located sample station of T4~~ |
| T5 | City of Troy southern site | This site will be used to evaluate LA concentrations south of the Troy community |
| T6 | SW upwind/downwind site | This site will be used to evaluate LA concentrations at the southwestern boundary of the OU and confirm if any LA is entering or leaving OU7 |
| T7 | SE upwind/downwind site | This site will be used to evaluate LA concentrations at the southeastern boundary of the OU and confirm if any LA is entering or leaving OU7 |
| TQC | Rotating co-located sampling station to each of the seven sampling locations | Co-located sampling station to evaluate analytical variability at each of the seven station locations |

Notes:

| | | | |
|---|---|---|---|
| LA | Libby Amphibole | SE | Southeast |
| NE | Northeast | SW | Southwest |
| NW | Northwest | OU | Operable Unit |

* Predominant winds in the area blow from the southeast and northwest. Stations on the southeast and northwest bounds of OU7 will act as upwind and downwind receptors depending on wind direction. A summary of historical meteorological conditions is presented in Section 4.4.1.

**Section 5.2 (Paragraph 5):** ~~Field co-located samples will be collected from the same location throughout the project and will consist of a co-located sampling station (Station T4QC) to be built approximately seven feet from the proposed sampling station to be located at the DEQ Troy Information Center~~. Co-located field samples will be collected by Station TQC at a different station location each sample period throughout the project. Station TQC will be placed next to each of the seven stations throughout Year 2 monitoring. Station TQC will be moved after each sampling period (beginning with Sampling Station T1) and will be cycled through each of the remaining stations (T2, T3, T4…T7) so that a minimum of 5 co-located samples are collected from each of the seven sampling stations over the 36 sampling periods.

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

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

Technical Review and Approval: _____ Date:_____
*(DEQ Project Manager or designate)*

EPA Review and Approval: _____ Date:_____
*(USEPA RPM or designate)*

## 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.

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***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.

TETRA TECH EM INC.

May 4, 2012

John Podolinsky
Montana Department of Environmental Quality
Remediation Division
P.O. Box 200901
Helena, MT 59620-0901

**Subject: Transmittal of TFO-00005, Year 3 Outdoor Ambient Air Study SAP, Operable Unit Number 7 of the Libby Asbestos Superfund Site**
**DEQ Contract 407026; Task Order 96**

John:

For your review, Tetra Tech is transmitting by email a pdf file that makes up the TFO-00005 package for the Year 3 Ambient Air Work Plan/Sampling and Analysis Plan (SAP). TFO-00005 spells out the changes that were made to the Year 2 Work Plan and include: (1) a change in number and location of ambient air monitoring stations for Year 3, and (2) a Year 3 schedule. The three files being transmitted include: 1) TFO-00005, and 2) a map showing Year 3 ambient air station locations, and 3) a new SAP cover and signature pages.

If the documentation is satisfactory, please sign both the TFO and SAP signature pages and pass along to Victor for his signature, if necessary. Please let me know if you require any further changes or if you have any questions prior to signature.

Tetra Tech appreciates the opportunity to work with you and the DEQ Remediation Division on this important project. If you have any questions, please call me at 442-3461.

Sincerely,

Steve MacNeill
Project Scientist

cc: Tetra Tech file

Attachments: via email

7 West 6th Avenue, Suite 612, Helena, MT 59601
Tel 406.442.5588 Fax 406.442.7182
www.ttemi.com

# FINAL
# REMEDIAL INVESTIGATION WORK PLAN
# OUTDOOR AMBIENT AIR STUDY
# (TFO-00005: YEAR 3 UPDATE)

## Operable Unit Number 7
## of the Libby Asbestos Superfund Site

May 4, 2012

Prepared for:

**MONTANA DEPARTMENT OF ENVIRONMENTAL QUALITY**
**Remediation Division**
P.O. Box 200901
Helena, Montana 59620

Contract Number 407026
Contract Task Order Number 96

Prepared by:

**TETRA TECH EM INC.**
Power Block Building, Suite 612
7 West 6th Avenue
Helena, Montana 59601
(406) 442-5588

**LIBBY ASBESTOS SITE OPERABLE UNIT 7 OUTDOOR AMBIENT AIR MONITORING WORK PLAN**

**FOR THE**

**TROY ASBESTOS PROPERTY EVALUATION PROJECT**

**Prepared for:**

**MONTANA DEPARTMENT OF ENVIRONMENTAL QUALITY**

**REVIEWS AND APPROVALS**

| | | |
|---|---|---|
| Tetra Tech EM Inc. Project Manager: | J. Edward Surbrugg<br>J. Edward Surbrugg, Ph.D. | Date: 5/9/12 |
| DEQ Project Officer: | John Podolinsky | Date: 5-9-12 |
| EPA Remedial Project Manager: | Victor Ketellapper | Date: 5-9-12 |
| EPA Remedial Project Manager: | Elizabeth Pagen | Date: 5/10/12 |

# Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO-00005

***Instructions to Requester:*** **Fax to contacts at bottom of form for review and approval.**
**File approved copy with Data Manager at the Troy Field Office (TFO).**
**Data Manager will maintain legible copies in a binder that can be accessed by TFO personnel.**
**If Modification is Temporary for a single Parcel, Data Manager will scan this and place in parcel's electronic file.**

**Project Work Plan/QAPP** (check one):

- ● Outdoor Ambient Air Study Work Plan
- ○ Other (Title and approval date):_____

**Site-Specific Guidance/SOP:**
Title NA Number/Revision): NA

Requester: **John Podolinsky** Title: **Project Manager**
Company: **DEQ** Date: **May 3, 2012**

Description of Modification:

This modification covers two topics: 1) Year 3 ambient air station locations, and 2) Year 3 ambient air sampling schedule. The two sections from the original SAP that are impacted by these changes are provided below.

***Section 4.4.2 in the Final Remedial Investigation Work Plan Outdoor Ambient Air Study – Operable Unit Number 7 of the Libby Asbestos Superfund Site*** provides a general description of proposed ambient air sampling station locations. "As previously discussed, the predominant winds in Troy tend to flow in southeast and northwest directions, following the river corridor in which Troy is located. Two sampling stations (one each) will be placed in close proximity to the northwest and southeast boundaries of OU7. This will ensure that there are upwind and downwind sample collection stations for both directions the wind is blowing. Two stations (one each) will also be located on the northwest and southeast borders of downtown Troy in order to have upwind and downwind sample stations in the area with the highest population density. One sample station will be placed at the DEQ Troy Information Center in downtown Troy to measure LA concentrations in Troy. One station will be placed in the Kootenai Vista area in the northern portion of OU7 and the last station will be placed along or near Iron Creek Road in the southwestern portion of OU7." The ambient air monitoring stations will be relocated for the second year for more comprehensive coverage of the four "air zones" identified in OU7. This will provide additional data in support of human health risks related to ambient air exposure.

***3.0 DATA QUALITY OBJECTIVES***

***STEP 4 – DEFINE THE BOUNDARIES OF THE STUDY***

**Temporal Bounds:** The program will begin in fall 2009, and is scheduled to continue for one year in order to ensure that temporal variability on the scale of days and months is adequately captured in the data set. Temporal bounds include the changing of weather patterns, particularly wind speed and direction, over time. A summary of historical meteorological conditions and impacts on placement of outdoor ambient air sampling equipment is presented in section(s) 4.4.1 and

4.4.2. If additional data is needed to improve the temporal representativeness of the data set and/or to collect data that will allow an assessment of long-term trends that may arise from any removal or remedial actions, then the program could be extended for several years. These decisions will be made by the risk managers once the data collected from the initial year are evaluated, and after consultation with EPA's scientific support team at the site.

Field Sampling Data Sheet where Modification is documented (attach associated correspondence): **N/A**

Potential Implications of Modifications: (1) Re-locating the ambient air sampling stations within the four "air zones" for Year 3 will further support human health risk assessment for OU7. Moving the stations will not impact analytical protocol and is not anticipated to have any impact on analytical results. (2) Performing a third year of ambient air sampling will further support human health risk assessment for OU7. The third year of sampling will not involve any changes in sampling methods or analytical protocol and will not have any impact on analytical results.

Duration of Modification (Check one):

○ Temporary

Date(s):_____ Station Number-_____

TA-_____

● Permanent (Proposed Text Modification Section) Effective Date: May 4, 2012

Proposed Text Modifications in Associated Document: ***Section 4.4.2 in the Final Remedial Investigation Work Plan Outdoor Ambient Air Study – Operable Unit Number 7 of the Libby Asbestos Superfund Site and Table 4-2:***
As previously discussed, the predominant winds in Troy flow in southeast and northwest directions, following the river corridor in which Troy is located. For year three sampling efforts, four sampling stations will be re-established at locations used during year one. One station will be located within each of the four unique "air zones" as shown on the attached figure. Two sampling stations will be re-established at locations in close proximity to the northern (T21) and southern (T24) boundaries of OU7. This will ensure that there are upwind and downwind sample collection stations for both directions the wind is blowing. One sample station (T22) will be located in the densely populated area of downtown Troy and a final station (T23) will be re-located south of Troy along the Iron Creek Road. Table 4-2 has the rationale for the ambient air monitoring locations and the attached figure shows the proposed year 3 ambient air monitoring locations.

## TABLE 4-2

## YEAR THREE OUTDOOR AMBIENT AIR SAMPLING LOCATIONS

| Station Number | Location* | Purpose |
|---|---|---|
| T21 | Upwind/downwind site near the northern border of OU7. Community exposure site located within small community area NE of the Kootenai River | This site will be used to evaluate LA concentrations at the small community near the northern boundary of OU7 and confirm if any LA is entering or leaving OU7 in Air Zone 1. |
| T22 | City of Troy population exposure site | This site will be used to evaluate LA concentrations in the Troy community (specifically in the population center) of Air Zone 2. |
| T23 | Upwind/downwind site on the lower reach of Iron Creek Road community exposure area. | This site will be used to evaluate LA concentrations in the southwestern area of the OU and confirm if any LA is entering or leaving OU7 in Air Zone 3. |
| T24 | Upwind/downwind site near the SE border of OU7. Community exposure site located within small community area SW of the Kootenai River | This site will be used to evaluate LA concentrations at the small community near the southeastern boundary of OU7 and confirm if any LA is entering or leaving OU7 in Air Zone 4. |
| TQC | Rotating co-located sampling station to each of the four sampling locations | Co-located sampling station to evaluate analytical variability at each of the four station locations |

Notes:

| | | | |
|---|---|---|---|
| LA | Libby Amphibole | SE | Southeast |
| NE | Northeast | SW | Southwest |
| NW | Northwest | OU | Operable Unit |

* Predominant winds in the area blow from the southeast and northwest. Stations on the southeast and northwest boundaries of OU7 will act as upwind and downwind receptors depending on wind direction. A summary of historical meteorological conditions is presented in Section 4.4.1.

Proposed Text Modifications in Associated Document:

***3.0 DATA QUALITY OBJECTIVES***

***STEP 4 – DEFINE THE BOUNDARIES OF THE STUDY, Temporal Bounds:*** The year three ambient air sampling program will begin in Spring 2012, and is scheduled to continue for one year in order to ensure that temporal variability on the scale of days and months is adequately captured in the data set.

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

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

Technical Review and Approval: _____ Date: _____
*(DEQ Project Manager or designate)*

EPA Review and Approval: _____ Date: 5/10/12
*(USEPA RPM or designate)*

## 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.

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# Record of Modification

to the

Troy Sampling and Quality Assurance Project Plan

Field Activities

TFO- 0 0 0 0 1 (numbered by Data Manager)

***Instructions to Requester:*** **Fax to contacts at bottom of form for review and approval.**
**File approved copy with Data Manager at the Troy Field Office (TFO).**
**Data Manager will maintain legible copies in a binder that can be accessed by TFO personnel.**
**If Modification is Temporary for a Single Parcel, Data Manager will scan this and place in parcel's electronic file.**

**Project Work Plan/QAPP** (check one)**:**

- Troy Removal Design Investigation WP/SAP
- O Other (Title and approval date):_____

**Site-Specific Guidance/SOP (Number and Revision No.)** (check one)**:**

- O Tetra Tech Aggressive Attic Inspection SOP
- O CDM-LIBBY-05, Current Revision (30-point soil sample collection)
- O CDM-LIBBY-06, Current Revision (Visible Vermiculite Estimation)

**Other** (Title, Number/Revision): _____

Requester: Catherine LeCours Title: DEQ Project Manager

Company: Montana DEQ Date: July 21, 2010

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): Revise Page 23, Section 4.4.5, Sample Labeling and Identification to read as follows: "The sample labeling scheme is as follows: TD-XXXXX Where: TD identifies that a sample is collected in accordance with this RDI SAP and XXXXX represents a 5-digit numeric code.

Field logbook and page number / FSDS where Modification is documented (or attach associated correspondence): n/a

Potential Implications of Modification: A five digit sample identification number will be consistent with the remainder of the Libby Asbestos database.

Duration of Modification (check one):

O Temporary

Date(s):_____ AD-_____

BD(s)-_____ TT(s)-_____

- Permanent (Proposed Text Modification Section) Effective Date: July 21, 2010

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): see above

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

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

Technical Review and Approval: _____ Date:_____
*(DEQ Project Manager or designate)*

EPA Review and Approval: _____n/a_____Date:_____
(*USEPA RPM or designate*)

## DATA QUALITY INDICATOR DEFINITIONS

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***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.

# Record of Modification

to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO - 00002

***Instructions to Requester:*** **Fax to contacts at bottom of form for review and approval.**
**File approved copy with Data Manager at the Troy Field Office (TFO).**
**Data Manager will maintain legible copies in a binder that can be accessed by TFO personnel.**
**If Modification is Temporary for a Single Parcel, Data Manager will scan this and place in parcel's electronic file.**

**Project Work Plan/QAPP** (check one):

- ● Troy Removal Design Investigation WP/SAP
- ○ Other (Title and approval date): _____

**Site-Specific Guidance/SOP (Number and Revision No.)** (check one):

- ○ Tetra Tech Aggressive Attic Inspection SOP
- ○ CDM-LIBBY-05, Current Revision (30-point soil sample collection)
- ○ CDM-LIBBY-06, Current Revision (Visible Vermiculite Estimation)

**Other** (Title, Number/Revision): _____

Requester: Catherine LeCours  Title: Project Manager
Company: DEQ/Tetra Tech  Date: August 5, 2010

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): This modification requires changes to the Removal Design Investigation (RDI) inspection procedures for the Bruce Cole property, AD-202036. This property is an approximately 37 acre parcel adjacent the Kootenai River located southeast of Troy. Tetra Tech completed an RDI inspection at the property on August 5, 2010 due to known contamination in a small barn, which is the only building located on the property. The remaining areas on the property consist almost entirely of a hay field with a small dirt road leading to the barn. Tetra Tech conducted an Environmental Resource Specialist (ERS) cleaning effort in the barn on September 3, 2009 at which time 7 bags of Zonolite were removed from the interior; however, due to the amount of contamination on the floor of the building, Tetra Tech was unable to detail clean the barn under standard ERS cleaning procedures. Because of the size of the property and the fact that the only known vermiculite is within the interior of the barn, Tetra Tech requests a modification from the standard RDI procedures by limiting the RDI to a reasonable area surrounding the barn. DEQ agrees that this modification would be prudent. In addition, during the TAPE investigation, the owner requested that Tetra Tech not sample within the hay field on the property fearing that his crop would be trampled, so a full TAPE inspection protocol could not be completed. Instead, Tetra Tech completed a limited inspection in the area around the barn. If analytical results indicate detectable Libby Amphibole (LA), a re-examination of the property may be required and areas of interest may need to be expanded.

Field logbook and page number / FSDS where Modification is documented (or attach associated correspondence): TR-0041, pages 61 through 63.

Potential Implications of Modification: The TAPE field inspection encompassed an area of 130 feet both east and west of the barn and approximately 40 feet south of the barn into the hay field. There is a slight potential that vermiculite may be unidentified in the limited use area (LUA) hayfield or the LUA overgrown areas adjacent to the Kootenai River. This potential may also exist on the specific use area (SUA) dirt road outside the inspection boundary. The property owner mows the hay field twice per year and plows the field once every 7 years.

Duration of Modification (check one):

- ● Temporary

  Date(s): **August 4, 2010** **AD-202036** _____

  BD(s)-_**BD-202883** TT(s)-_**TD-22027, TD-22028, TD-22029 AND TD-22030**

- ○ Permanent (Proposed Text Modification Section) Effective Date: **NA**

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): **NA**

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

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

Technical Review and Approval: Catherine LeClaire Date: August 11, 2010
*(DEQ Project Manager or designate)*

EPA Review and Approval: _____ Date: _____
*(USEPA RPM or designate)*

Revised March 25, 2010

## 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.

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***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.

# Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO-00015

***Instructions to Requester:*** **Fax to contacts at bottom of form for review and approval.**
**File approved copy with Data Manager at the Troy Field Office (TFO).**
**Data Manager will maintain legible copies in a binder that can be accessed by TFO personnel.**
**If Modification is Temporary for a Single Parcel, Data Manager will scan this and place in parcel's electronic file.**

**Project Work Plan/QAPP** (check one)**:**

- ● Troy Asbestos Property Evaluation Work Plan
- O Other (Title and approval date):_____

**Site-Specific Guidance/SOP (Number and Revision No.)** (check one)**:**

- O CDM-LIBBY-10, Current Revision (30-point dust sample collection)
- O CDM-LIBBY-05, Current Revision (30-point soil sample collection)
- O CDM-LIBBY-06, Current Revision (Visible Vermiculite Estimation)

**Other** (Title, Number/Revision): _____

Requester: Catherine LeCours Title: Project Manager
Company: DEQ Date: May 4, 2010

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): **This modification calls for the elimination of rinsate (equipment) blanks as field QC samples. Equipment blanks will no longer be collected as they are currently not required by EPA for soil sampling within OU7 because: (1) detection levels for LA using current polarized light microscopy (PLM) analytical methods are not low enough to capture concentrations that would be expected in equipment blanks; and (2) the frequency of detection for LA in historically-collected project equipment blanks is extremely low.**

Field logbook and page number where Modification is documented (or attach associated correspondence):
**NA**

Potential Implications of Modification: **Although equipment decontamination procedures will remain unchanged, there is a slight potential that incomplete decontamination issues will not be caught due to the elimination of equipment blanks. As the PLM analytical detection limits are not low enough to identify the levels that would remain as a result of incomplete decontamination, the contaminants that would potentially be passed along to the next soil sample would not be identified.**

Duration of Modification (check one):

O Temporary
Date(s):_____ AD-_____
BD(s)-_____ TT(s)-_____

● Permanent (Proposed Text Modification Section) Effective Date: May 4, 2010

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): **The following text from Section 5.2 (Quality Control Samples) shall be removed. Equipment Rinsate Blanks** ~~**– Soil sampling equipment rinsate blanks will be collected at a rate of one per calendar week (Monday through Sunday) of sampling per field team. Equipment rinsate blanks will be collected by pouring distilled water over the sampling equipment into a decontaminated stainless steel sampling bowl, pouring the rinse water from the bowl into a sample bottle, placing the sample bottle in a re-closable plastic bag, and submitting it for analysis by method EPA 100.2, modification 20. Data from equipment blank samples will be used to evaluate whether the decontamination procedures result in sampling equipment that is asbestos-free. Equipment rinsate blank samples with elevated results may indicate inadequate equipment decontamination procedures. These results will be communicated to the field immediately upon receipt such that corrective action can be implemented.**~~

**The following text shall replace the deleted text from Section 5.2 (Quality Control Samples) Equipment Rinsate Blanks: Equipment blanks are currently not required by EPA for soil sampling at OU7 because: (1) detection levels for LA using current polarized light microscopy (PLM) analytical methods are not low enough to capture concentrations that would be expected in equipment blanks; and (2) the frequency of detection for LA in historically-collected project equipment blanks is extremely low.**

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

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

Technical Review and Approval: Catherine LeCours Date: May 2, 3010
*(DEQ Project Manager or designate)*

EPA Review and Approval: NA Date: NA
(*USEPA RPM or designate*)

## DATA QUALITY INDICATOR DEFINITIONS

***Reject*** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect 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.

# Record of Modification

to the

Troy Sampling and Quality Assurance Project Plan

Field Activities

TFO-00016

***Instructions to Requester:*** **Fax to contacts at bottom of form for review and approval.**
**File approved copy with Data Manager at the Troy Field Office (TFO).**
**Data Manager will maintain legible copies in a binder that can be accessed by TFO personnel.**
**If Modification is Temporary for a Single Parcel, Data Manager will scan this and place in parcel's electronic file.**

**Project Work Plan/QAPP** (check one)**:**

- ● Troy Asbestos Property Evaluation Work Plan
- O Other (Title and approval date):_____

**Site-Specific Guidance/SOP (Number and Revision No.)** (check one)**:**

- O CDM-LIBBY-10, Current Revision (30-point dust sample collection)
- O CDM-LIBBY-05, Current Revision (30-point soil sample collection)
- O CDM-LIBBY-06, Current Revision (Visible Vermiculite Estimation)

**Other** (Title, Number/Revision): _____

Requester: Catherine LeCours Title: Project Manager

Company: DEQ Date: May 4, 2010

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): **This modification adjusts the schedule of visual observations and soil sampling frequency for TAPE inspections. Changes implemented by this modification come from the recently approved OU4 General Property Investigation WP developed by Camp Dresser McKee (CDM) and approved as final in April 2010. This TFO is designed to provide consistency and efficiency between OU4 and OU7 sampling efforts in screening properties for potential future removal actions.**

Field logbook and page number where Modification is documented (or attach associated correspondence):
**NA**

Potential Implications of Modification: **This modification provides consistency to OU4 and OU7 characterization efforts and efficiency for potential future removal actions. No negative impacts to data quality or completeness of characterizations are anticipated.**

Duration of Modification (check one):

O Temporary

Date(s):_____ AD-_____

BD(s)-_____ TT(s)-_____

● Permanent (Proposed Text Modification Section) Effective Date: May 4, 2010

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): **The following table and text from the General Property Investigation WP (CDM 2010) replaces all references to associated visual observation and soil sampling protocols as set forth in Section 4 of the 2007 TAPE WP. New protocols are as follows:**

**Visual Inspection**

**Visual inspection of exterior soils will be completed in accordance with CDM-LIBBY-06. The number of point inspections to be completed per use area is defined in Table 4-2.**

**TABLE 4-2**
**TAPE VISUAL INSPECTION AND SOIL SAMPLING PROTOCOL**

| Area Type[1] | Visual Inspection Protocol[2] | Soil Sampling Protocol[3] |
|---|---|---|
| SUA (Flowerbed, Garden, Play Area, etc.) | 1 PI/100 $ft^2$ | 1 sample per use area type |
| Driveway (SUA) | 1 PI/200 $ft^2$ | 1 sample per use area |
| CUA (Yard, etc) | 1 PI/1,450 $ft^2$ | 1 sample per acre (43,560 $ft^2$) |
| LUA (Field, Pasture, etc.) | 1 PI/7,260 $ft^2$ | 1 sample per 5 acres (217,800 $ft^2$) |
| ISA (Shed, Carport, Garage, etc.) | 1 PI/100 $ft^2$ | 1 sample per use area |
| Crawlspace (ISA) | 1 PI /100 $ft^2$ | 1 sample per use area |
| NUA (Wooded Area, etc.) | No Inspection | No Sampling |

**Notes:**
**[1]Mulitple SUAs of the same type within the same general area may be combined to form one sample area. Examples include gardens along the drip line of the house, or multiple raised flower beds within a CUA.**
**[2]A minimum of 5 points will be inspected per use area regardless of size.**
**[3]All soil samples are 30-point composites. Areas where vermiculite is observed will also be sampled.**

**SUA – Specific Use Area**
**CUA – Common Use Area**
**LUA – Limited Use Are**
**NUA – Non Use Area**
**ISA – Interior Surface Area**
**PI – Point Inspection**
**$ft^2$ – square feet**

**Soil Sample Collection**

**The frequency of TAPE soil samples will be collected in accordance with Table 4-2, that defines the maximum area per soil sample. The soil samples will be collected following the procedures described in the TAPE Work Plan (Tetra Tech 2007). Thirty soil aliquots will be placed into a stainless steel bowl, homogenized, and placed in a re-closable plastic bag.**

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

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

Technical Review and Approval: Catherine LeCours Date: May 4, 2010
*(DEQ Project Manager or designate)*

EPA Review and Approval: NA Date: NA
(*USEPA RPM or designate*)

**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.

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***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.

# Appendix C
# Preliminary List of Applicable or Relevant and Appropriate Requirements

**APPENDIX E**

**Preliminary List of Chemical and Location Specific Federal and State Applicable or Relevant and Appropriate Requirements (ARARs)**

***Federal ARARs***

| Statue and Regulatory Citation | ARAR Determination | Description | Comment | Chemical | Location |
|---|---|---|---|---|---|
| National Historic Preservation Act (NHPA),<br>16 U.S.C. ' 470<br>40 CFR 6.301(b)<br>36 CFR 60, 63, 800 | Applicable | This statute and implementing regulations require federal agencies to take into account the effect of this response action upon any district, site, building, structure, or object that is included in or eligible for the National Register of Historic Places. | If cultural resources on or eligible for the national register are present, it will be necessary to determine if there will be an adverse effect and if so how the effect may be minimized or mitigated.<br><br>The unauthorized removal of archaeological resources from public or Indian lands is prohibited without a permit, and any archaeological investigations at a site must be conducted by a professional archaeologist.<br><br>If any remedial action activities are necessary beyond permitted, SHPO consultation and NHPA compliance will be addressed during remedial design. | | x |
| Archaeological and Historic Preservation Act<br>16 U.S.C. ' 469<br>40 CFR 6.301(c)<br>43 CFR 7 | Applicable | This statute and implementing regulations establish requirements for the evaluation and preservation of historical and archaeological data, which may be destroyed through alteration of terrain as a result of a federal construction project or a federally licensed activity or program. | | | x |
| Fish and Wildlife Coordination Act<br>16 U.S.C. '' 661, et seq.,<br>40 CFR 6.302(g)<br>50 CFR 83<br>33 CFR 320-330 | Applicable | This statute and implementing regulations require coordination with federal and state agencies for federally funded projects to ensure that any modification of any stream or other water body affected by any action authorized or funded by the federal agency provides for adequate protection of fish and wildlife resources. | If the remedial action involves activities that affect wildlife and/or non-game fish, federal agencies must first consult with the U.S. Fish and Wildlife Service and the relevant state agency with jurisdiction over wildlife resources. | | x |
| Endangered Species Act,<br>16 U.S.C. ' 1531<br>40 CFR 6.302(h)<br>50 CFR 17 and 402 | Relevant and Appropriate | This statute and implementing regulations provide that federal activities not jeopardize the continued existence of any threatened or endangered species. Endangered Species Act, Section 7 requires consultation with the U.S. Fish and Wildlife Service to identify the possible presence of protected species and mitigate potential impacts on such species. | If threatened or endangered species are identified within the remedial areas, activities must be designed to conserve the species and their habitat. To date no threatened or endangered species have been identified in the area of the site. | | x |
| Migratory Bird Treaty Act,<br>16 U.S.C. '' 703, et seq.<br>50 CFR 10.13 | Relevant and Appropriate | This requirement establishes a federal responsibility for the protection of the international migratory bird resource and requires continued consultation with the U.S. Fish and Wildlife Service during remedial design and remedial construction to ensure that the cleanup of the site does not unnecessarily impact migratory birds. | The selected remedial actions will be carried out in a manner to avoid adversely affecting migratory bird species, bald eagle and including individual birds or their nests. | | x |
| Clean Air Act (CAA)<br>42 U.S.C. ' 7401, et seq.<br>40 CFR 61, Subpart M<br>(delegated to the state and incorporated by reference at ARM 17.8.341) | Applicable | National Emission Standards for Hazardous Air Pollutants (NESHAPs) for Asbestos | The selected remedial actions will be carried out in a manner that will comply with all the National Emission Standard for Asbestos as required under NESHAP. | x | |
| Clean Air Act (CAA)<br>Air Cleaning<br>40 CFR 61.152<br>Note: Section 61.152(b)(3) is not delegated to the State | Relevant and Appropriate | This requirement establishes detailed specifications for air cleaning used as part of a system to control asbestos emissions control system. | These requirements would be applicable if air cleaning is part of the building demolitions. It would be relevant and appropriate to other air cleaning operations. | x | |
| Clean Air Act (CAA)<br>Air Cleaning<br>40 CFR 61.155 | Relevant and Appropriate | This requirement establishes detailed standards for operations that convert asbestos containing waste material into nonasbestos (asbestos-free) material. | These requirements would be applicable if the remedial action includes any treatment of asbestos containing material. | x | |

**APPENDIX D**

**Preliminary List of Chemical and Location Specific Federal and State Applicable or Relevant and Appropriate Requirements (ARARs)**

***State of Montana ARARs***

| Statue and Regulatory Citation | ARAR Determination | Description | Comment | Chemical | Location |
|---|---|---|---|---|---|
| Montana Asbestos Control Act<br>ARM 17.8.204<br>ARM 17.8.206 | Relevant and Appropriate | Ambient Air Monitoring & Ambient Air Methods and Data:<br><br>Require that all ambient air monitoring, sampling and data collection, recording, analysis and transmittal shall be in compliance with the Montana Quality Assurance Manual except when more stringent requirements are determined to be necessary. | These requirements will be followed unless an equivalent or more stringent approach is deemed appropriate. | x | |
| Montana Asbestos Control Act<br>ARM 17.8.220<br>ARM 17.8.223 | Applicable | Ambient air quality standard for settled particulate matter. Particulate matter concentrations in the ambient air shall not exceed the following 30-day average: 10 grams per square meter.<br><br>Ambient air quality standards for PM-10. PM-10 concentrations in the ambient air shall not exceed the following standards: 150 micrograms/cubic meter of air, 24- hour average; and 50 micrograms/cubic meter of air, expected annual average. | The removal action will involve significant soil disturbance. Particulate/dust levels will need to be controlled.<br><br>Each of the ambient air quality standards includes specific requirements and methodologies for monitoring and detection. These requirements will be followed unless an equivalent or more stringent approach is deemed appropriate. | x | |
| Montana Asbestos Control Act<br>ARM 17.8.304 | Applicable | Visible Air Contaminants. No source may discharge emissions into the atmosphere that exhibit opacity of 20 percent or greater, averaged over six consecutive minutes. This standard is limited to point sources, but excludes wood waste burners, incinerators, and motor vehicles. | No visible emissions are anticipated. | x | |
| Montana Asbestos Control Act<br>ARM 17.8.308 | Applicable | Airborne Particulate Matter. Emissions of airborne particulate matter from any stationary source shall not exhibit opacity of 20 percent or greater, averaged over six consecutive minutes. | This standard applies to the production, handling, transportation, or storage of any material; to the use of streets, roads, or parking lots; and to construction or demolition projects. | x | |
| Montana Asbestos Control Act<br>ARM 17.8.315 | Relevant and Appropriate | Odors. If a business or other activity will create odors, those odors must be controlled, and no business or activity may cause a public nuisance. | Action is not expected to produce nuisance level odors. | x | |
| Montana Water Quality Control Act<br>ARM 17.30.637 | Applicable | It states that no waste may be discharged and no activities conducted which, either alone or in combination with other waste activities, will cause violation of surface water quality standards; provided a short term exemption from a surface water quality standard may be authorized by the department for Aemergency remediation activities@ under the conditions specified in ' 75-5-308, MCA. | | x | |
| Montana Water Quality Control Act<br>ARM 17.30.705 | Applicable | Requires that for any surface water, existing and anticipated uses and the water quality necessary to protect these uses must be maintained and protected unless degradation is allowed under the nondegradation rules at ARM 17.30.708. | | x | |
| Montana Asbestos Control Act<br>ARM 17.74.351<br>ARM 17.74.365 | Applicable | Adopts and incorporates by reference 40 CFR subparts A and M (NESHAP) for asbestos, and the National Institute of Occupational Safety and Health (NIOSH) Manual of Analytical Methods for detecting asbestos by phase contrast microscopy (PCM) and a description of the 7402 Analytical Method for detecting asbestos by transmission electron microscopy (TEM).<br><br>It requires that training for asbestos workers, supervisors, inspectors, project management planners, and project designers meet requirements of 40 CFR 763, subpart E, Appendix C (Asbestos Model Accreditation Plan). | | x | |
| The Montana Asbestos Control Manual | Applicable | The Montana Asbestos Control Manual (the Manual) is adopted and incorporated by reference in ARM Title 17, Chapter 74, Subchapter 3. The Manual identifies practices and procedures for inspecting for asbestos, conducting asbestos projects, and clearing asbestos projects. The Montana Department of Environmental Quality administers NESHAP through its asbestos control program. The NESHAP contains standards that regulate building demolitions, renovations, asbestos disposal sites, and other sources of asbestos emissions. | | x | |
| Montana Code Annotated (MCA), Montana Floodplain and Floodway Management Act and Regulations ,<br>ARM 36.15.601 et seq. MCA 76-5-401 et seq. | Relevant and Appropriate | The Floodplain and Floodway Management Act and regulations specify types of uses and structures that are allowed or prohibited in the designated 100-year floodway and floodplain. Libby OU2 is adjacent to the Kootenai River, and these standards are relevant to all actions within the floodplain. | The Screening Plant is presumed to be outside the 100 year flood plain. No solid waste disposal will occur within the floodway or floodplain. | | x |

**APPENDIX D**

**Preliminary List of Chemical and Location Specific Federal and State Applicable or Relevant and Appropriate Requirements (ARARs)**

***State of Montana ARARs***

| Statue and Regulatory Citation | ARAR Determination | Description | Comment | Chemical | Location |
|---|---|---|---|---|---|
| Montana Code Annotated (MCA), Montana Antiquities Act, MCA 22-3-421, et seq. | Relevant and Appropriate | Montana Antiquities Act addresses the responsibilities of State agencies regarding historic and prehistoric sites including buildings, structures, paleontological sites, archaeological sites on state owned lands. Each State agency is responsible for establishing rules regarding historic resources under their jurisdiction which address National Register eligibility, appropriate permitting procedures and other historic preservation goals. The State Historic Preservation Office maintains information related to the responsibilities of State Agencies under the Antiquities Act. | | | x |
| Montana Code Annotated (MCA), Montana Human Skeletal Remains and Burial Site Protection Act (1991), MCA 22-3-801 et seq. | Applicable | The Human Skeletal Remains and Burial Site Protection Act is the result of years of work by Montana Tribes, State agencies and organizations interested in ensuring that all graves within the State of Montana are adequately protected. If human skeletal remains or burial sites are encountered during remedial activities within OU2 of the Libby Asbestos Site, then these requirements will be applicable. | | | x |
| Montana Code Annotated (MCA), Local Air Pollution Control Program MCA 75-3-301 | Applicable | The provisions of the Lincoln County Air Pollution Control Program, approved by Montana DEQ pursuant to § 75-2-301, MCA and administered by Lincoln County, are designed to regulate activities within a designated Air Pollution Control District to achieve and maintain such levels of air quality as will protect human health and safety and, to the greatest degree practicable, prevent injury to plant and animal life and property, and facilitate the enjoyment of the natural attractions of Lincoln County. | | | x |
| Montana Code Annotated (MCA) MCA 75-5-605 | Applicable | Prohibits the causing of pollution of any state waters. Section 75-5-103(21)(a)(i) defines pollution as contamination or other alteration of physical, chemical, or biological properties of state waters which exceeds that permitted by the water quality standards. States that it is unlawful to place or cause to be placed any wastes where they will cause pollution of any state waters. Any permitted placement of waste is not placement if the agency's permitting authority contains provisions for review of the placement of materials to ensure it will not cause pollution to state waters. | These requirements would be triggered only in the event that the removal action impacts surface of groundwater. Excavation may take place close to the Kootenai River. Precautions will need to be put into place to prevent accidental release of asbestos containing soils into the river. May also be applicable if disposal of RACM occurs on-site. | | x |
| Montana Code Annotated (MCA) MCA 87-5-502 and 504 | Applicable | Provide that a state agency or subdivision shall not construct, modify, operate, maintain or fail to maintain any construction project or hydraulic project which may or will obstruct, damage, diminish, destroy, change, modify, or vary the natural existing shape and form of any stream or its banks or tributaries in a manner that will adversely affect any fish or game habitat. The requirement that any such project must eliminate or diminish any adverse effect on fish or game habitat is applicable to the state in approving remedial actions to be conducted. The Natural Streambed and Land Preservation Act of 1975, MCA ' 75-7-101, et seq., (Applicable -- substantive provisions only) includes similar requirements and is applicable to private parties as well as government agencies. | Consultation with the Montana Department of Fish, Wildlife and Parks, and any conservation district or board of county commissioners (or consolidated city/county government) is encouraged during the designing and implementing of the remedial action for OU2 of the Libby Asbestos Site. | | x |

The ARARs for OU 7 will be included in the site-wide Feasibility Study and the Record of Decision for OUs 4, 5, 6, 7, and 8.

**Attachment 1**
**Libby Asbestos Site, Troy OU7 Residential/Commercial Cleanup Criteria, Specific Use Area Visible Vermiculite Action Level Technical Memorandum, April 23, 2009**

**(Note: EPA revised the attached 2009 Residential/Commercial Clean-up Criteria after**
**OU7 field work was completed for this RI)**

**ATTACHMENT**

Libby Asbestos Site, Troy Operable Unit 07 Residential/Commercial Cleanup Criteria, Specific Use Area Visible Vermiculite Action Level Technical Memorandum

Libby Asbestos Site
Troy Operable Unit 07
Residential/Commercial Cleanup Criteria
Specific Use Area Visible Vermiculite Action Level
Technical Memorandum

## I. Introduction

On December 15, 2003, the United States Environmental Protection Agency (EPA) issued a draft final Action Level and Clearance Criteria Technical Memorandum for the Libby Asbestos Site Residential/Commercial Cleanup (herein referred to as the "CCM"). The CCM provides detailed information regarding action levels that have been and continue to be used for determining which properties or situations require an emergency response cleanup. The action levels identified in the CCM are as follows:

Attics/Walls
- Visual confirmation of open, non-contained, or migrating vermiculite insulation.

Interiors
- Visual confirmation of vermiculite in the indoor living space.
- Concentration of Libby Amphibole (LA) in an indoor dust sample greater than 5,000 LA structures per square centimeter using AHERA counting methods.

Soils
- Visual confirmation of vermiculite or other vermiculite mine related materials in "specific use areas." A specific use area is defined as a garden, former garden, planter, or other defined area of a yard likely to receive significant use and generally not covered with grass.
- Concentration of LA in specific use areas or other yard soils by any analytical method greater than or equal to 1% Libby asbestos.

The CCM includes details to support the establishment of these action levels.

## II. Contaminant Exposure Routes for Libby OU4 versus Troy OU7

W.R. Grace maintained numerous vermiculite processing facilities within and near the town of Libby. These processing facilities provided a low-cost or even free source of LA-contaminated vermiculite for local residents. Given the availability of the vermiculite, many local residents in Libby acquired vermiculite materials for their gardens and yards and would transport the vermiculite by truck load to their properties. This availability resulted in the wide-spread distribution and use of LA-contaminated vermiculite observed in Libby today. Troy is located approximately 20 miles to the northwest of Libby. The probability of such widespread use of LA-contaminated vermiculite via the same distribution as observed in Libby (personal trucks) throughout Troy is believed to be low. However, vermiculite attic insulation has been noted throughout Troy Operable Unit (OU) 7. The attic insulation was distributed in bags thus easier to transport to Troy.

## III. Troy OU7 Investigations

In 2007, the Montana Department of Environmental Quality (DEQ) began inspections (both interior and exterior) of the residential and commercial areas of OU7. DEQ followed the basic protocol that had been established for OU4 with modifications based on “lessons learned” and updated data collection tools. The objective of the OU7 investigations is to identify those parcels that meet the emergency response cleanup criteria identified in the CCM.

One important fact to note is that not all vermiculite (commercially available or otherwise) contains LA. Thus, visual observation of vermiculite in soil does not necessarily confirm the presence of LA fibers. However, in OU4, through historical sampling and analysis a strong correlation between the visible confirmation of vermiculite in soil and the presence of LA fibers in soil samples has been observed. Given this experience, DEQ did not include the collection of soil samples from specific use areas that contained visible vermiculite initially in OU7. DEQ did semi-quantify the presence of visible vermiculite through a 30-point inspection and categorized the visible observations as none, low, intermediate, or high (CDM-Libby-06).

At the conclusion of the 2007 field season, anecdotal evidence reported to the field teams by property owners suggested that vermiculite observed in the specific use areas in OU7 did not come from the “local piles” or other sources in Libby. The property owners in OU7 reported the recent purchase of planting materials containing vermiculite from local hardware stores.

Based on this information, the OU7 sampling protocol changed in 2008 to include the collection of a soil sample from specific use areas and a description of the visible vermiculite. The field teams also returned to those parcels inspected in 2007 and collected a soil sample from specific use areas that had visible vermiculite. Thus, several lines of evidence could be considered when reviewing data from the exterior portion of a parcel potentially eligible for cleanup.

## IV. Troy Results

In 2008, 392 soil samples were collected from specific use areas with visible vermiculite in OU7. The results of those samples along with pertinent parcel information (e.g., use area description, visible vermiculite counts, vermiculite descriptions, etc.) are provided in the attached spread sheet. Some notable statistics are in the following table.

| Visible Vermiculite Description | # of Bin A/B1 | # of Bin B2 | Total # in Subset (description category) |
|---|---|---|---|
| Expanded Homeowner Purchase | 1 | 0 | 1 |
| Expanded Unknown Source | 114 | 0 | 114 |
| Unexpanded/Unexpanded Unknown Source | 98 | 2 | 100 |
| Unexpanded/Potting soil mix Homeowner purchase | 170 | 1 | 171 |
| Unknown | 1 | 0 | 1 |

The Visible Vermiculite Description is based on the inspection team's discussions with the property owners and their own observations. The "Bin" categories allow for ease of grouping of PLM-VE soil analytical results. Bin A is non-detect, Bin B1 is "trace" or less than 0.2% LA, and Bin B2 is between 0.2 and 1% LA by weight. Bin C are those soils with LA greater than 1% by weight. Please note there are no Bin C results from the specific use areas with visible vermiculite in OU7.

In summary, out of 392 soil samples collected from specific use areas with visible vermiculite, only three have LA fibers detected above 0.2%.

## V. Conclusions

The following conclusions can be drawn from the attached spread sheet and other evidence detailed in this memorandum:

- The majority of vermiculite present in soil in OU7 does not contain LA and did not come from the "local piles" in Libby;
- The vast majority of the 392 soil samples were semi-quantitatively categorized with very few "low" visible observations pursuant to the protocols in CDM-Libby-06. Therefore, the samples and use areas represented in the table do not include any "large piles of pure LA;" and
- There were five samples collected described as "expanded leaking from building" and only one of those had a Bin B2 (between 0.2 and 1% LA by weight) result.

## VI. Recommendations

DEQ offers the following recommendations based on the above discussion:

- Continue to collect soil samples from all use areas;
- Continue to semi-quantify the presence of visible vermiculite in all use areas; and
- Do not apply the action level of "visual confirmation of vermiculite or other vermiculite mine related materials" in "specific use areas" in OU7 as an independent cleanup criterion. Instead, consider several lines of evidence and criteria for cleanup decisions, including, but not limited to, the presence of visible vermiculite in exterior use areas.

**Attachment 2**
**Background Soil Study Report**

# BACKGROUND SOIL STUDY REPORT
# FOR
# OPERABLE UNIT 7 OF THE LIBBY ASBESTOS SUPERFUND SITE

***Revision 0 – March 2014***

Prepared for:

**MONTANA DEPARTMENT OF ENVIRONMENTAL QUALITY**
**Remediation Division**
P.O. Box 200901
Helena, Montana 59620

Contract Number 407026
Contract Task Order Number 113

Prepared by:

**TETRA TECH EM INC.**
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## *ACRONYMS AND ABBREVIATIONS*

| | |
|---|---|
| < | Less than |
| µm | Micrometer |
| % | Percent |
| ± | Plus or minus |
| | |
| ABS | Activity-based sampling |
| AMSL | Above mean sea level |
| AT | Averaging time |
| | |
| CDM Smith | CDM Smith Inc. |
| cc | Cubic centimeter |
| CoC | Chain-of-custody |
| CSM | Conceptual site model |
| DEQ | Montana Department of Environmental Quality |
| DQO | Data quality objective |
| ED | Exposure duration |
| EDD | Electronic data deliverable |
| EDS | Energy dispersive spectroscopy |
| EDXA | Energy dispersive x-ray analysis |
| EF | Exposure frequency |
| EPA | United States Environmental Protection Agency |
| ESAT | Environmental Services Assistant Team |
| ET | Exposure time |
| | |
| FBAS | Fluidized bed asbestos segregator |
| f/cc | Fibers per cubic centimeter |
| FSDS | Field Sample Data Sheet |
| FSP | Field sampling plan |
| ft | Feet |
| $ft^2$ | Square feet |
| | |
| g | Gram |
| | |
| HASP | Health and safety plan |
| HHRA | Human health risk assessment |
| HQ | Hazard quotient |
| IDW | Investigation derived waste |
| ISO | International Organization for Standardization |
| IRIS | Integrated Risk Information System |
| IUR | Inhalation unit risk factor |

| | |
|---|---|
| LA | Libby amphibole |
| L/min | Liters per minute |
| MARCH | Document Management Archive |
| MCE | Mixed-cellulose ester |
| MET | Meteorological |
| mm | Millimeter |
| MSL | Mean sea level |
| ND | Non-detect |
| OU | Operable Unit |
| PCM | Phase contrast microscopy |
| PCME | Phase contrast microscopy equivalent |
| PDF | Portable document format |
| PEL | Permissible exposure limit |
| PLM-VE | Polarized light microscopy visual estimation |
| PPE | Personal protective equipment |
| QA | Quality assurance |
| QA/QC | Quality assurance/quality control |
| QAPP | Quality assurance project plan |
| QC | Quality control |
| RBC | Risk based concentration |
| RfC | Reference concentration |
| RME | Reasonable maximum exposure |
| SAB | EPA scientific advisory board |
| SAED | Selective area electron diffraction |
| SAP | Sampling and analysis plan |
| s/cc | Structures per cubic centimeter |
| s/g | Structures per gram |
| SOP | Standard operating procedure |
| SPF | Troy Sample Preparation Facility |
| STEL | Short term exposure limit |
| TAPE | Troy Asbestos Property Evaluation |
| TAS | Target analytical sensitivity |
| TDL | Target detection limit |
| TEM | Transmission Electron Microscopy |

| | |
|---|---|
| Tetra Tech | Tetra Tech EM Inc. |
| TWA | Time weighted average |
| TWF | Time weighting factor |
| | |
| UCL | Upper confidence limit |
| USACE | U.S. Army Corps of Engineers |
| USDA | U.S. Department of Agriculture |
| USFS | U.S Forest Service |
| USGS | U.S. Geological Survey |
| | |
| XRD | X-ray defraction |

# 1.0 INTRODUCTION

The Montana Department of Environmental Quality (DEQ), Remediation Division, tasked Tetra Tech EM Inc. (Tetra Tech) to conduct a background soil study to evaluate background Libby Amphibole (LA) asbestos levels and associated mineralogy in soils in and around Operable Unit (OU) 7 of the Libby Asbestos Superfund Site. This work was performed under Contract No. 407026, Task Order 113.

The United States Environmental Protection Agency (EPA) is the lead agency for the Libby Asbestos Superfund Site except for OU7, where DEQ is the lead agency through a cooperative agreement with EPA. The OU7 soil background study was designed to complement OU4 background soil data (CDM Smith 2012). Background soil study data are needed to support development of the human health risk assessment (HHRA). This report presents the background soil study findings, conclusions, and recommendations.

The following subsections describe the purpose of the study and the organization of this report.

## 1.1 PURPOSE OF STUDY

It has been theorized that LA asbestos from Vermiculite Mountain may have been dispersed by glacial processes (rather than by human activity) during the Pleistocene period about 16,000 years ago (Langer et al. 2010). The purpose of this study is to (1) evaluate naturally-occurring or background concentrations and the distribution of LA asbestos and potentially other forms of asbestos in Troy Valley soils, and (2) characterize the potential for LA asbestos fibers to be released to the environment from soils and country rock not associated with mining operations at OU3. Establishing background levels of LA asbestos and its unique mineralogical characteristics in soils in and around OU7 is critical to establishing baseline LA asbestos conditions for OU7. The background soil study data will be used to evaluate whether and to what extent LA asbestos fibers are being released to the air in the Troy area from naturally-occurring source materials.

## 1.2 REPORT ORGANIZATION

This report contains the sections summarized below.

- Section 1.0 includes the introduction and purpose of study.
- Section 2.0 provides background information on the study.
- Section 3.0 describes the study design.
- Section 4.0 presents the study results.
- Section 5.0 presents conclusions and recommendations based on the study results.
- Section 6.0 lists the references cited in this report.

Figures and tables are provided after the references.

# 2.0 STUDY BACKGROUND

Vermiculite mining and associated processing operations were active at a mine on Vermiculite Mountain near Libby, Montana, from the 1920s until 1990. While in operation, the mine may have produced as much as 80 percent of the world's supply of vermiculite (EPA 2005). The processed and exfoliated vermiculite was primarily used as insulation in buildings and as a soil amendment. The Libby vermiculite deposit includes amphibole asbestos. For decades, the processing of vermiculite ore and generation and disposal of waste materials resulted in the widespread presence of amphibole asbestos throughout the Libby community. In 1999, EPA Region 8 dispatched an emergency response team to investigate media reports of abundant amphibole asbestos and high rates of asbestos-related disease in Libby. Subsequent environmental investigations found asbestos throughout many areas in and around Libby that included a form of amphibole asbestos known as LA asbestos.

## 2.1 SITE LOCATION

OU7 of the Libby Asbestos Superfund Site encompasses the town of Troy, Montana, which lies 18 miles northwest of Libby in the Kootenai River valley at elevations ranging from 1,850 feet above mean sea level (AMSL) at the northern end to 2,500 feet AMSL on the mountain slopes surrounding the valley. The topography of OU7 consists of relatively flat river valley terraces on both sides of the gently graded Kootenai River. Several tributaries flow into the Kootenai River along the 8-mile stretch of the river in OU7. OU7 is approximately 8 miles long and 1.8 miles wide at its widest point. The OU7 boundary was selected to ensure that investigations captured most of the older properties in and around Troy that are more likely to contain LA asbestos or LA asbestos source materials.

The OU7 boundary is depicted on Figure 2-1.

## 2.2 SITE HISTORY

Some of the Libby vermiculite mine workers lived in Troy and commuted daily to work at the mine in Libby. The mine workers were exposed to asbestos-containing materials at the mine and processing facilities, and they transported asbestos-containing dust to their homes on clothes and equipment. In addition, residents of Troy traveled to Libby for activities such as shopping, working (other than at the mine), and attending school sporting events. They likely came into contact with LA asbestos in Libby during these visits. The asbestos-containing vermiculite ore and waste materials in varying forms may have been used for amending soils (as fill or as a conditioner), building materials (plaster, concrete, or chinking amendment), wood burning, and insulating buildings in and around Troy. The ore and waste may also have been spilled or placed in transportation corridors.

## 2.3 GEOLOGY AND SOILS

The mountains surrounding the Kootenai Valley are generally composed of folded, faulted, and metamorphosed blocks of Precambrian sedimentary rocks and minor basaltic intrusions. Primary rock types are meta-sedimentary argillites, quartzites, and marbles (Ferreira et al. 1992).

The vermiculite deposit at Vermiculite Mountain is located approximately 7 miles northwest of Libby in the Rainy Creek drainage. The vermiculite deposit specific to the Libby vermiculite mine is classified as a deposit within a large ultramafic intrusion, such as pyroxenite plutons, which is zoned and cut by syenite or alkalic granite and by carbonatitic rock and pegmatite. The formation of vermiculite and asbestiform amphiboles in the Libby mine deposit has been assessed to be the result of the hydrothermal alteration of augite by high-temperature silica-rich solutions (Larsen and Pardee 1929; Boettcher 1967; Van Gosen et al. 2008; Meeker et al. 2003).

The Vermiculite Mountain deposit is contained within the Rainy Creek alkaline-ultramafic complex. The Rainy Creek complex is described as the upper portion of a hydrothermally altered alkalic igneous complex composed primarily of magnetite pyroxenite, biotite, pyroxenite, and biotitite (Boettcher 1967). The original ultramafic body is an intrusion into the Precambrian Belt Series of northwestern Montana, likely deposited during the early Cretaceous Period (Langer et al. 2010). A syenite body southwest of and adjacent to the altered pyroxenite is associated with numerous syenite dikes that cut the pyroxenites. Generally, the Vermiculite Mountain amphiboles are classified as one of three main types including, in order of decreasing abundance, winchite (approximately 84 percent[%]), richerite (approximately 11%), and tremolite (approximately 6%). Other minerals include magnesio-riebeckite, edenite, and magnesio-arfvedsonite (Meeker et al. 2003). Generally, Vermiculite Mountain amphiboles occur as either vein-fillings or replacement of the primary pyroxene of the Rainy Creek complex. Traditionally, amphibole asbestos is believed to occur as a vein-filling mineral formed during hydrothermal or low-temperature alteration. The amphiboles in samples from Vermiculite Mountain appear to be forming as direct replacements of pyroxene through fluid infiltration in microfractures (Meeker et al. 2003).

During Pleistocene time, the Kootenai Valley was located beneath glacial Lake Kootenai, which was present as a result of glacial advances from the most recent Pinedale glacial advance more than 16,000 years ago (Langer et al. 2010). Glacial advances scoured what is now Vermiculite Mountain and deposited sediments in the Rainy Creek delta of glacial Lake Kootenai (Figure 2-2). When the glaciers receded, Lake Kootenai gradually drained, resulting in erosion of the Rainy Creek delta and redeposition of glacial sediments down the entire Kootenai River drainage below the elevation of 2,450 feet AMSL (Locke and Smith 2004; Smith 2006). Glacial Lake Kootenai finally drained around 11,000 years ago (Ehlers and Gibbard 1996) and the Kootenai River occupied its present channel. The presence of amphibole asbestos in alluvial sediment layers has been demonstrated at several locations in the Kootenai Valley (Adams et al. 2010). The U.S. Geological Survey (USGS) reported the presence of multiple layers of glacial sediments containing amphibole material from Vermiculite Mountain in active gravel quarries near Libby (Adams et al. 2010). The USGS also reported that some soils from a sample area within the valley contained a range of 0.004% to 0.047% LA (by mass).

The sequence of events that occurred as Pleistocene glaciation waned and ice receded to the north includes deposition of lacustrine and glacial outwash sediments sourced from multiple locations, including the area of Vermiculite Mountain, as well as locations to the south, southeast, and north of the Kootenai Valley (Langer et al. 2010). Amphibole asbestos eroded by glaciation was deposited as glacial

outwash in the vicinity of Rainy Creek and as lacustrine sediments in nearby areas of glacial Lake Kootenai. Additionally, some of this sediment was dispersed more broadly in downstream locations during erosion and redeposition associated with changing lake levels as the ice receded and temporarily re-advanced. Much of the glacial outwash and lacustrine sediments observed in the Kootenai Valley were derived from locations other than Vermiculite Mountain. Sediment sources in the Libby area other than Vermiculite Mountain include Pipe Creek and Quartz Creek north of Libby, as well as sources south and southeast of Libby. In the Troy area, drainages to the south of Troy fed the glacial lakes that eventually coalesced to form glacial Lake Kootenai, and much of the lacustrine sediments and glacial outwash sediments in the Troy area were likely derived from these sources to the south (Figure 2-2).

It is possible that large amounts of material containing amphibole asbestos were eroded from Vermiculite Mountain during Pleistocene glaciation and this material was subsequently deposited over a broad area through deposition of glacial outwash and lacustrine sediments. Sediment samples from glacial deposits analyzed by Langer et al. (2010) show that Pleistocene glaciation likely resulted in amphibole asbestos from Vermiculite Mountain being deposited in lacustrine sediments in glacial Lake Kootenai and re-deposited during a re-advance of the Purcell Trench Glacier lobe. Two thin, discrete lake bottom deposits have been determined to contain LA-bearing sediments but these layers are covered in most places with more than 30 meters of other fine-grained sediments that do not contain LA from Vermiculite Mountain (Langer et al. 2010). The distribution of lacustrine sediment layers that potentially contain LA from Vermiculite Mountain was described in Langer et al. (2010). Ongoing erosion of the lacustrine sediment exposures, as well as disturbance of sediments by human activity, likely resulted in additional dispersal of glacial deposits that potentially contain amphibole asbestos.

Some soils in the Kootenai Valley have formed in lacustrine sediments, glacial till, loess, and loess that has been influenced by volcanic ash. The sediments and till are largely derived from the pre-Cambrian sedimentary rocks. Figure 2-3 shows the soils that have formed on lacustrine and glacial outwash deposits, some of which have been reworked by wind. Much of the soil within the Troy community has been modified by residential construction, industrial operations, and residential activities. These modifications include soil disturbance during construction, road building, railroad operations, gardening, incorporating vermiculite into the soil, and other activities.

The remnant lacustrine sediment terraces that surround Troy (Figure 2-3) generally have a fine-silty textured surface layer and are underlain by silt loam and clay loam texture sediments (U.S. Department of Agriculture [USDA] 1995). The fine-textured surface layers have developed directly from loess, or from mixtures of loess and glacial deposited materials, including lacustrine sediments. The loess in the Kootenai Valley has generally been influenced by volcanic ash depositions. The primary soil type in Libby (USDA soil unit 103) has developed in alluvial deposits and has a surface layer of gravelly silt loam. This soil is underlain by stratified alluvial deposits of sand, silt, and gravel (USDA 1995). The main soil type in Troy (USDA soil unit 108) developed on lacustrine and glacial outwash terraces and has a silt loam surface texture.

X-ray powder diffraction (XRD) analyses by the USGS of shallow, subsurface soil from more than 10 sites in the Libby area show that it is composed of major (greater than 20%) quartz; minor (5% to 20%) muscovite (or illite) and albitic feldspar; and trace (less than 5%) orthoclase, clinoclore, non-fibrous amphibole (likely magnesio-hornblende), calcite, amorphous material (probably organic), and possible pyrite and hematite. Other minerals are likely present at levels below 0.5% and are generally not detectable by routine XRD analysis. These mineral components represent the average components for the area and likely vary to some extent depending on local conditions. Surface soil contains the above components with the addition of more organic material.

## 2.4 TOPOGRAPHY AND SURFACE WATER HYDROLOGY

The topography in the vicinity of Kootenai Valley is generally characterized by mountainous terrain exhibiting steep-sided valley walls. Portions of the Kootenai Valley, particularly in the intermontane Purcell Trench near Bonners Ferry, are characterized by relatively flat terrain and broad flood plains. Pleistocene glaciation has influenced the local topography through erosion and glacial sediment deposition. Examples of local topographic features associated with Pleistocene glaciation include cirques high in the mountains created by alpine glaciers, broadened valleys created by continental glacial lobes, moraines, glacial outwash terraces, and glacial lake-bed surfaces. A prominent regional feature of the effects of Pleistocene glaciation is the Purcell Trench west of Troy, which is a broad intermontane valley sculpted by the advancement of a glacial lobe southward between the Purcell and Cabinet Mountains in the east and the Selkirk Mountains in the west.

The Kootenai River is the primary river in the Kootenai Valley. The river flows in a westward to northwestward direction, cutting a deep valley through Libby and Troy. Several tributaries feed the river from the north and south and have carved smaller valleys in the surrounding mountains. Several lakes in the area also lie in the Kootenai River drainage, including Bull Lake (south of Troy) and numerous alpine lakes at higher elevations. The flow direction of the river and its tributaries is generally controlled by the underlying geologic bedrock structure and is affected in localized areas by the presence of Pleistocene glacial till, moraine, outwash, and lacustrine deposits (Langer et al. 2010).

## 2.5 OCCURRENCE OF LIBBY AMPHIBOLE ASBESTOS

The main contaminant of concern at the Site is asbestos. The vermiculite deposit near Libby contains a distinct form of naturally-occurring amphibole asbestos that is comprised of a range of mineral types and morphologies. Historically, the form of asbestos present in the vermiculite deposit was classified as tremolite/actinolite (Larsen and Pardee 1929; Larsen 1942; McDonald et al. 1986; Amandus and Wheeler 1987; Leake 1997; Wylie and Verkouteren 2000; Bandli and Gunter 2006; Sanchez et al. 2008).

More recently, the USGS performed electron probe micro-analysis and XRD analysis of 30 samples obtained from asbestos veins at the mine (Meeker *et al.* 2003). Using new mineralogical naming rules recommended by Leake *et al.* (1997) and Hawthorne *et al.* (2012), the results indicate that the asbestos at the mine includes a number of related amphibole types. The most common forms are winchite and richterite, with lower levels of tremolite, magnesio-riebeckite, edenite, and magnesio-arfvedsonite.

Some minerals may also be classified as actinolite, depending on the valence state of iron. Because there are no toxicological data to distinguish differences in toxicity among these different forms (and mineralogical name changes do not alter the asbestos form present), the EPA does not believe it is important to attempt to distinguish among these various amphibole types. Therefore, the mixture of asbestos present at the Site is referred to as "Libby amphibole" or LA.

It had been thought that the source of LA asbestos throughout the Libby Superfund Site was from the W.R. Grace Mine, transported by human activities, or wind-blown dust into OU7. As such, the LA asbestos may have come from one, or some combination of several, primary sources outside of OU7, including vermiculite mining waste, vermiculite ore, vermiculite processing waste, bulk residuals from vermiculite processing, LA asbestos-containing rock, or LA asbestos-containing vermiculite insulation. Residential use of vermiculite from the Libby mine, primarily as building insulation and as a soil amendment, was common. In some cases, vermiculite insulation was found after it sifted from the attic into interior and exterior walls. In rare cases, vermiculite was found as an additive in building materials such as plaster, mortar, and concrete. The LA asbestos-containing soil found in OU7 is generally associated with vermiculite used as a soil amendment in flowerbeds and gardens, for leveling of low spots, and for backfilling of utilities.

As described in Section 2.3, previous studies (Langer et al. 2010 and Smith 2006) have shown two situations where sediments potentially containing LA asbestos may have been deposited into Glacial Lake Kootenai: (1) as lake-bottom sediments derived from meltwater flowing down Rainy Creek, and (2) as lake-bottom sediments eroded from the Rainy Creek outwash and re-deposited during a re-advance of the Purcell Trench Glacier lobe. When the glaciers receded, Glacial Lake Kootenai gradually drained, resulting in erosion of the Rainy Creek delta and redeposition of glacial sediments down the entire Kootenai River drainage below the elevation of approximately 2,450 feet AMSL (Locke and Smith 2004; Smith 2006). Native soils in and around OU7 that developed in these lacustrine and glacial sediment deposits may contain LA asbestos.

## 2.6 HHRA Data Gaps

Because the nature of LA asbestos and associated exposure pathways in OU7 are similar to those in Libby, and the vermiculite insulation found in Troy is similar in morphology and mineralogy to that found in Libby, a systematic screening of interiors and exteriors of Troy area residences, public areas, schools, and businesses was completed to gather information to determine how many Troy area properties contain LA asbestos. This systematic screening is referred to as the Troy Asbestos Property Evaluation (TAPE) (Tetra Tech 2007).

Potentially complete and significant inhalation exposure pathways for LA asbestos and the data needed to quantify human health risks for exposure pathways were identified during development of the HHRA Work Plan (Tetra Tech 2011) and from available data compiled from ongoing investigations in OU7 and OU4. A conceptual site model (CSM) depicting potential human inhalation exposure pathways to LA asbestos was prepared as part of the HHRA work plan development and is shown in Figure 2-4.

The background soil study was designed to address two data gaps required to complete the OU7 HHRA: (1) characterizing background LA asbestos levels in soil in and around OU7, and (2) evaluating human health inhalation exposure to disturbed background soils through activity-based sampling (ABS). The HHRA will evaluate potential human health inhalation exposure pathways for LA asbestos to determine: (1) if post-removal risks to human health are acceptable, and (2) if exposure to LA in background soils is a significant portion of the overall risk. The EPA policy is to not clean up soils to a concentration lower than background (EPA 2002); therefore, it is important to determine the naturally-occurring levels of LA asbestos in and around OU7.

## 3.0 STUDY DESIGN

This section summarizes procedures used for collecting and analyzing background soil samples and collecting background soil-related activity-based air samples in OU7. Soil sampling procedures are summarized in Section 3.1 and air sampling procedures are summarized in Section 3.2. Soil and air sample preparation and analytical methodologies are described in Section 3.3. Data quality is discussed in Section 3.4.

Tetra Tech designed a background soils sampling study based on the OU4 background study conducted in 2011 (CDM Smith 2012). The OU7 investigation was conducted in 2012 to evaluate LA levels in background soils near OU7 (Tetra Tech 2012).

The OU7 background soil sample locations were selected using geologic maps, the Soil Survey of the Kootenai National Forest Area (USDA Forest Service 1995), visual inspections, general knowledge of the area, and other criteria described below. The 1995 soil survey provided important soil information and maps used to select the sites. Soil mapping units were differentiated based on landform, slope, parent material, vegetation, aspect, elevation, and rock outcrop. Background soil samples were collected from mapping units for soils that developed from parent materials deposited during the historical glacial Lake Kootenai era. The soil mapping units are shown in the table below.

**TROY SOIL MAPPING UNITS FOR BACKGROUND SOIL SAMPLING**

| Soil Mapping Unit | Elevation Mapped | Distinguishing Characteristics |
|---|---|---|
| 102: Andic Dystric Eutrochrepts, lacustrine terraces | 2,000 to 3,700 | Soils formed in lacustrine deposits. Silt loam and silty clay loam textures |
| 106: Andic Dystrochrepts, glacial outwash terraces | 2,000 to 4,000 | Soils formed in glacial outwash deposits. Surface loess layer 4 to 14 inches thick |
| 108: Andic Dystric Eutrochrepts, lacustrine terraces – Andic Dystrochrepts, glacial outwash terraces, complex | 2,000 to 4,000 | Formed in lacustrine and glacial outwash deposits intricately mixed. Stratified layers of sand and gravel and silt loam and silty clay loam sediments |
| 110: Eutrochrepts, glacial outwash terraces | 2,000 to 3,500 | Soils formed in glacial outwash deposits that have been reworked by wind |

## 3.1 SOIL SAMPLE COLLECTION

The following sections summarize the background soil sample collection process.

### 3.1.1 Selection of Soil Sampling Locations

The selection of soil sampling locations representative of background conditions was complicated by several factors:

- Because of the complex geology and geological history of the area, soils in the Troy Valley may differ in their mineral compositions from location to location—what is background in one location may not be representative of background in all locations.
- Human activities, such as construction of buildings and roads, often result in substantial disturbance of soils, and may involve moving soils from one location to another. To the extent that soils may differ from location to location, soils in the area of human disturbances may not necessarily be representative of background at that location.
- Historic releases of vermiculite from the Libby mine and from other vermiculite processing and transporting facilities may have resulted in the deposition of vermiculite and LA in soils, altering their asbestos content compared to what would have been present had the mine never operated.

Because of these factors, the following criteria were used in selecting locations for collection of background soil samples:

1. To facilitate access, all sampling locations were on City, County, State, or Federal land or lands currently being leased by a government entity.
2. All sampling locations were in or near the Troy Valley and below 2,450 feet AMSL, the approximate elevation of Glacial Lake Kootenai sediments deposited in the Kootenai River drainage (Locke and Smith 2004; Smith 2006).
3. No sampling locations were in areas exhibiting evidence of historic or recent anthropogenic activities (in the past 50 to 100 years) that would have resulted in substantial disturbance or mixing of soil.
4. No sampling locations were within about 100 meters of any known or suspected local vermiculite emission sources (e.g., railroads, highways).
5. No sampling locations were in areas with fill material.
6. No sampling locations were in areas where visible vermiculite was observed.

Eleven background soil sampling locations dispersed throughout the Troy Valley were identified. These are depicted on Figure 2-3.

### 3.1.2 Soil Sample Collection

Two independent 30-point composite samples were collected from each background soil sampling location. Each composite was collected so as to be representative of the entire soil sampling location area. This was accomplished by laying out a 30-point grid within the sampling area. Two independent grab samples were collected from within about 1 to 2 feet of each grid node. The first aliquot was placed into a compositing container, and the second aliquot was placed into a second compositing container. This procedure continued until an aliquot had been collected from all 30 grid nodes. Grid node locations were occasionally adjusted to avoid obstructions such as trees, rocks, etc. Approximately 500 grams of soil were collected from each of the two composited samples for laboratory analysis. Table 3-1 is a summary of background soil sampling and analytical activities.

Background soil sample aliquots were collected using a 1-inch stainless steel coring device to create an approximately 6-inch deep hole. Prior to sample collection, loose organic debris (e.g., leaves, pine needles, duff) were manually removed. Soil samples were collected in accordance with project-specific SOP CDM-LIBBY-05, Revision 3, Soil Sample Collection at Residential and Commercial Properties (a copy of this SOP is included in Appendix C of the Sampling and Analysis Plan (SAP) [Tetra Tech 2012]), with the following project-specific modifications:

- Sample aliquots were collected using a 1-inch diameter stainless steel coring device.
- All sampling locations were pre-determined and did not require a use area designation.
- The top inch of each core was removed to minimize the potential contribution of historic deposition of airborne LA asbestos released from past mining, milling, and transporting activities.
- The remaining portion of each core sample (1 to 6 inches) was homogenized in a stainless steel bowl in the field. The field team noted if soil horizons were visible within the core and, if so, recorded the thickness of the horizons and the soil's appearance (e.g., color, grain size).
- Soil samples were delivered to the Troy Sample Preparation Facility (SPF) and stored for ABS activities under chain-of-custody (CoC) and transported to the appropriate laboratory for preparation and analysis.

At the time of collection, the amount of visible vermiculite (none, low, intermediate, high) was recorded. A Garmin Global Positioning System (GPS) unit was used to record the latitude and longitude at the center of each background soil sampling location. The GPS location coordinates were recorded in the field logbook and on field sampling data sheets (FSDSs), and are associated with the unique identification number for each background soil sampling location. Property-specific information was collected for each sampling location and recorded on a Background Soil/ABS field form. Copies of the completed field forms are provided in Appendix A.

## 3.2 ACTIVITY-BASED AIR SAMPLE COLLECTION

Potential exposure to background LA asbestos was assessed using an ABS scenario and soils collected from the background soil sampling locations. The ABS scenario was conducted at the Troy SPF using one of the three containers of soil obtained from each background location. The ABS scenario was completed in an activity area constructed with temporary walls and floor of polyethylene sheeting. The interior surfaces of the activity room were wet-wiped with paper towels between each ABS scenario. Two pre-ABS air samples were collected inside the ABS activity room prior to commencing the ABS in order to document baseline airborne LA asbestos levels inside the activity room.

After the soil from each 5-gallon container had been emptied onto the polyethylene floor sheeting, a single grab soil sample (approximately 200 grams) was collected. Because the soil was thoroughly homogenized prior to being emptied onto the floor, this grab sample was expected to be representative of the average level of LA asbestos in the soil at the sampling location.

The selected ABS activity was a digging scenario that simulates a child digging and playing in the dirt. Sampling personnel sat on the ground and emptied the soil from the 5-gallon container onto the polyethylene floor sheeting. They then used a hand trowel to place the soil back into the container. Once all the soil has been placed back into the container, the process was repeated for a total of 120 minutes.

One activity-based air sample was collected using the soil from each background soil sampling location. During ABS sample collection, the sampler wore both a high-volume and a low–volume pump to collect two “replicate” samples (that is, each filter represents the same sample collection duration, but different total sample air volumes). The appropriate flow rate for each sampling pump was optimized to achieve the highest air sample volume possible without causing the filter to become overloaded. The high volume pump flow rate was 10 liters (L) per minute (L/min) and the low volume pump flow rate was 4.0 L/min. This resulted in sample volumes of 1,200 L and 480 L, respectively. The high volume air samples were collected using a Gilair high volume air sampling pump while the lower volume air samples were collected using an SKC AirChek 5000 personal air sampling pump.

Each activity-based air sample was collected for a duration of 120 minutes using cassettes with 25-millimeter diameter mixed cellulose ester (MCE) filters with an 0.8 micrometer (µm) pore size. The cassettes were affixed to the chest of the actor performing the child play activity to simulate the breathing zone position of a child. The top cover from the cowl extension on the cassettes was removed and the cassette was oriented face down. The actors monitored the cassettes throughout the sampling activities to ensure they remained directed toward the activity and free of obstruction.

When it was necessary to relieve an actor during sample collection, a properly suited relief actor switched places. The active actor stopped, removed the sample pump, passed it to the relief actor, and assisted with donning and adjusting the sample pump and cassette placement. The exchanges took less than 60 seconds, so the sampling pumps and event time clocks were not halted during the exchange.

## 3.3 SAMPLE PREPARATION AND ANALYSIS

Soil sample preparation and analysis is summarized in Section 3.3.1. Fluidized bed asbestos segregator (FBAS) filter preparation and analysis is summarized in Section 3.3.2. Activity-based air sample preparation and analysis is summarized in Section 3.3.3.

### 3.3.1 Soil Sample Preparation and Analysis

Each background soil sample (except for the soil used for ABS) was dried and homogenized at the Troy SPF and then split into three fractions:

1. The first fraction was processed in basic accordance with the current version of project-specific soil sample preparation SOP ISSI-LIBBY-01, Revision 11 (a copy of this SOP is included in Appendix C of the SAP [Tetra Tech 2012]); the fines were analyzed for asbestos by polarized light microscopy visual estimation (PLM-VE) in basic accord with the project-specific SOP SRC-LIBBY-03, Revision 3 *Analysis of Asbestos Fibers in Fine Soil by Polarized Light Microscopy* (SRC 2012), and the coarse fraction was archived.

2. The second fraction was processed by FBAS and analyzed by Transmission Electron Microscopy (TEM) (see Section 3.3.2 for details).

3. The third fraction was archived.

### 3.3.2 FBAS Filter Preparation and Analysis

The second fraction of each background soil sample was used to prepare an FBAS filter for TEM analysis, in basic accord with site-specific SOP ESAT-LIBBY-01, Revision 0, *The Fluidized Bed Asbestos Segregator Method for Determination of Releasable Asbestos Fibers in Soil.* For five of the 22 soil samples, the FBAS fraction was used to prepare three replicate FBAS filters. Each was sent to a different lab for analysis.

During sample preparation, the Troy SPF determined whether the samples were rich in rock flour. This information was provided to the analytical laboratory in response to a 2011 Environmental Services Assistant Team (ESAT) report titled *Controlling Matrix Interference Effects of Rock Flour in the Fluidized Bed Method for Analysis of Asbestos in Soil* (TechLaw, Inc. 2011), in which ESAT recommends that soil samples prepared by FBAS for TEM analysis that are rich in rock flour undergo indirect preparation with ashing in order to control matrix interference effects of the rock flour during TEM analysis.

Each FBAS filter was submitted for asbestos analysis using TEM in basic accord with the International Organization for Standardization (ISO) 10312 method (ISO 1995) counting protocols, and the current version project-specific laboratory modifications LB-000016, LB-000029, LB-000067, and LB-000085, as well as laboratory modification LB-000066C (although a more recent version [LB-000066D] is available for this laboratory modification, the earlier version was intentionally used for this study to ensure a higher frequency of structure photographic images).

Rock flour-rich soil samples underwent indirect preparation with ashing at the analytical laboratory in basic accord with SOP EPA-LIBBY-08, *Indirect Preparation of Air and Dust Samples for TEM Analysis*, and applicable modifications in the ESAT report (TechLaw, Inc. 2011).

All amphibole structures (including not only LA asbestos but all other asbestos types) that exhibited applicable Selective Area Electron Diffraction (SAED) patterns and Energy Dispersive X-Ray Analysis (EDXA) spectra and met specified recording rules (see below), were counted.

#### *Initial Analysis (High Magnification) of FBAS Filter*

Initially, each FBAS filter was analyzed using a magnification of 20,000. The size (length and width, aspect ratio), structure type, and the mineral type for all structures longer than 0.5 µm with an aspect ratio of 3:1 or higher were recorded. For each filter, a minimum of two grid openings on each of two grids was counted. Counting continued until one of the following occurred:

1. A target analytical sensitivity of 6.3E+03 gram$^{-1}$ (g$^{-1}$) was achieved.
2. 50 LA asbestos structures were recorded.
3. An area of 1.2 milimeter$^2$ of filter had been examined.

When one of these criteria was achieved, the analyst completed the grid opening being examined, and ceased.

#### *Supplemental Analysis (Low Magnification) of FBAS Filter*

After completing the initial examination at a magnification of 20,000, if fewer than 50 LA asbestos structures were recorded and the target sensitivity had not been achieved, the magnification was decreased to 5,000 times and only phase contrast microscopy equivalent (PCME) structures (i.e., length > 5 µm, width ≥ 0.25 µm, aspect ratio ≥ 3:1) were recorded until one of the following was achieved:

1. The target analytical sensitivity of 6.3E+03 g$^{-1}$ was achieved.

2. 50 LA asbestos structures were recorded (including the structures counted at high magnification).

3. An area of 3.0 mm$^2$ of filter had been examined (including the filter area counted at high magnification).

When one of these criteria was achieved, the analyst completed the grid opening being examined, and ceased.

### 3.3.3 Activity-Based Air Sample Preparation and Analysis

Two (one high volume and one low volume) activity-based air samples were collected from each background soil sample. Both were analyzed for PCME LA asbestos by TEM (low magnification) in basic

accord the ISO 10312 method (ISO 1995) counting protocols, and the current version project-specific laboratory modifications LB-000016, LB-000029, LB-000066D, LB-000067, and LB-000085.

## 3.4 DATA QUALITY

The field data verification process is summarized in Section 3.4.1 and the analytical data verification process is summarized in Section 3.4.2.

### 3.4.1 Field Data Verification

Field data were reviewed for completeness, accuracy, and consistency. Data recorded on the FSDSs and in logbooks were checked against the OU7 Scribe Database to ensure consistency of data throughout. Finally, a review of the OU7 Scribe Database occurred after data entry to verify that the data were complete and no errors were present.

### 3.4.2 Analytical Data Verification

Ten percent of the PLM-VE analytical data were verified in basic accord with project-specific PLM data review and verification SOP, EPA-Libby-10, Revision 0, and 10 percent of the TEM analytical data were verified in basic accord with project-specific SOP, TEM data review and verification SOP EPA-LIBBY-09, Revision 2. In addition, field quality control (QC) data were reviewed for adherence to the frequency and QC limits specified in the SAP (Tetra Tech 2012).

### 3.4.3 Statement on Data Quality

The objective of the background soil study was to characterize background concentrations and distribution of LA asbestos in Troy Valley soils. The resulting data will be used in the HHRA to support exposure scenario and risk assessment calculations. Background soil study data quality objectives (DQOs) are presented in the SAP (Tetra Tech 2012).

To accomplish this objective, Tetra Tech collected and analyzed 22 soil samples from 11 background soil sampling locations and 16 background soil-related activity-based air samples (including field QC samples). The soil samples were analyzed for LA asbestos by PLM-VE. The ABS samples were analyzed for LA asbestos by TEM. A portion of each soil sample was also prepared by FBAS and analyzed for LA asbestos by TEM.

To ensure that the data are of acceptable quality to achieve the project objective, both field and analytical data were reviewed and verified in accordance with the procedures specified in the SAP (Tetra Tech 2012) and summarized above. On the basis of the data review and verification findings, the data meet the study objective and appear to be of acceptable quality for supporting HHRA exposure scenario and risk assessment calculations.

## 4.0 BACKGROUND SOIL STUDY FINDINGS

Table 4-1 presents the concentrations and LA fiber counts for OU7 background soils prepared and analyzed by FBAS and for concentrations in air using an ABS scenario. FBAS soil concentrations, reported as structures per gram (s/g), are provided for high magnification (High Mag) analysis and for standard PCME LA analysis. PCME analysis records structures with a length > 5 µm, width ≥ 0.25 µm, and an aspect ratio (length:width) ≥ 3:1. Results for ABS air samples are expressed as PCME LA structures per cubic centimeters of air (s/cc).

LA was detected in 10 of 11 OU7 background soils prepared by FBAS and analyzed by PCME (Table 4-1). Only soils from Shannon Flats West had no detected LA. LA was detected in the air sample collected during the ABS scenario in soil from the Shannon Flats West location. The PCME determined maximum LA concentration in OU7 background soil is 2.95E+05 s/g; the average LA concentration is 5.24E+05 s/g. Five soil samples were split and submitted to three different laboratories for analysis. Split sample results indicate different fiber counts and different mineralogical compositions are possible from different laboratories.

The lower part of Table 4-1 presents the concentrations of LA in air samples from the ABS activity of playing with the background soil. LA was detected in 7 of the 11 air samples collected during the ABS activity. The maximum concentration was 2.73E-03 s/cc and the average concentration was 3.2E-04 s/cc. LA fibers with the mineralogical composition of WRTA-NaK were the most common form identified.

## 5.0 CONCLUSIONS

The following conclusions are drawn from the results of the background soils study:

- LA has been detected in background soils in and around OU7 that are not thought to be affected by anthropogenic releases from vermiculite mining and processing activities.
- The types of LA structures in background soils within the Kootenai Valley include a mixture of NaK (WRTA) and XX (AC, TR, CH), which indicate that a portion of the LA structures observed likely do not originate from the Libby vermiculite ore body (Meeker and others 2003, and Gunter and Sanchez 2009).
- The concentration of LA in background soils (≤ 0.02%) is well below the detection limit of PLM-VE, but can be reliably detected by FBAS following preparation using the rock flour method and analysis by TEM. FBAS soil preparation method appears to be a more sensitive metric of LA detection in soil than either ABS or field visible vermiculite observations.
- The results support the conclusion that there is a non-zero level of LA in soils in and around OU7 that is not attributable to vermiculite mining and processing activities associated with the Libby Mine. Further, these results support the data reported by Adams and others 2010, Langer et al, 2010, and Gunter and Sanchez, 2009, indicating that low level detections of amphibole fibers in soils within the Kootenai Valley originated from normal geologic, soil building processes unrelated to mining and milling of vermiculite ore from Vermiculite Mountain.

Soil analytical data collected as part of this study will be applied to a site-wide risk assessment being conducted for the Libby Asbestos Site. The risk assessment will provide information, in part, on the health-effects associated with background levels of LA in naturally occurring soils at OU7.

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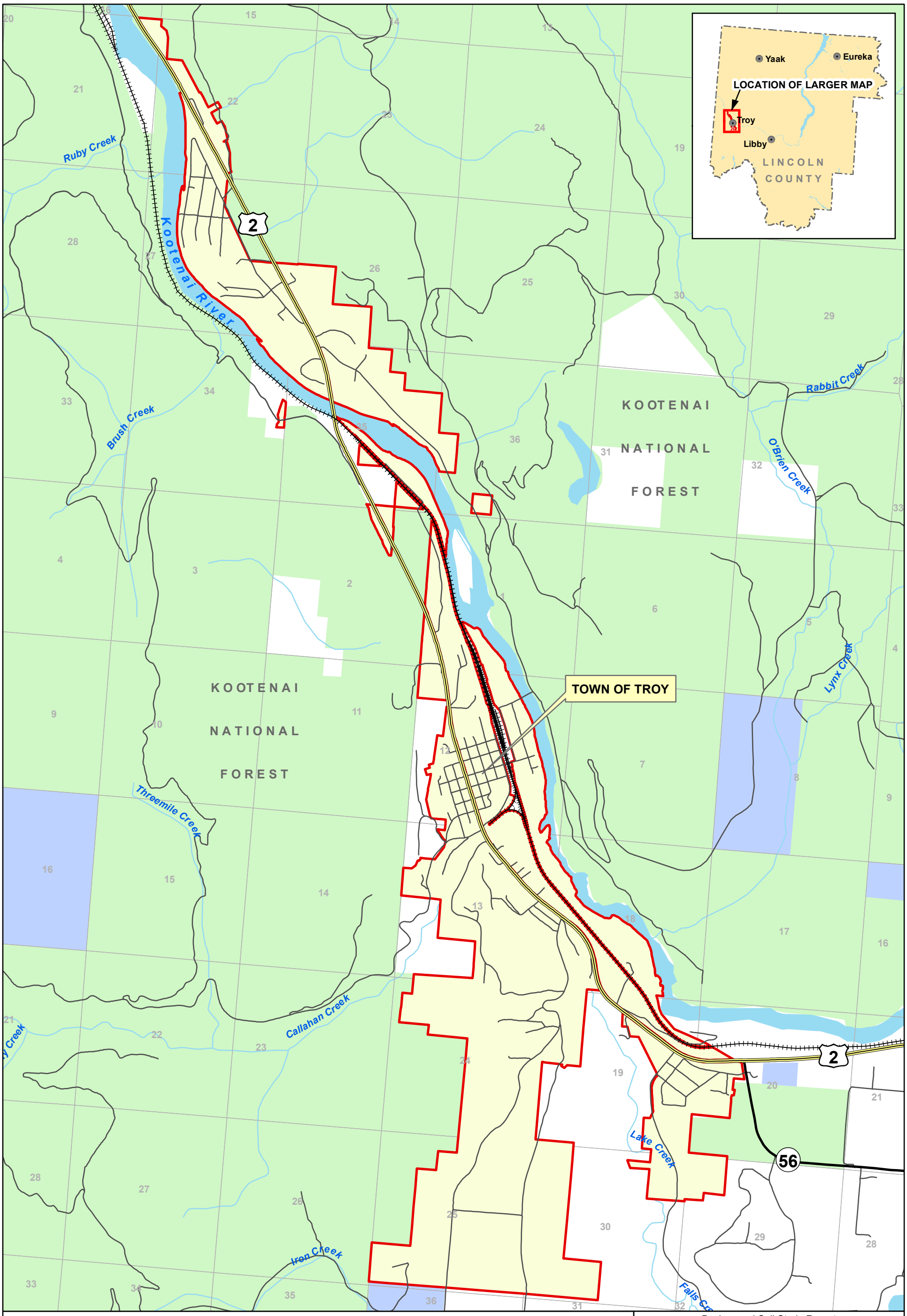
## *FIGURES*




**LEGEND**

- OPERABLE UNIT 7
- STATE HIGHWAY
- US HIGHWAY
- OTHER ROADS
- RAILROAD
- NATIONAL FOREST LAND
- PRIVATE LAND
- STATE LAND
- WATER

N

0 1 Miles

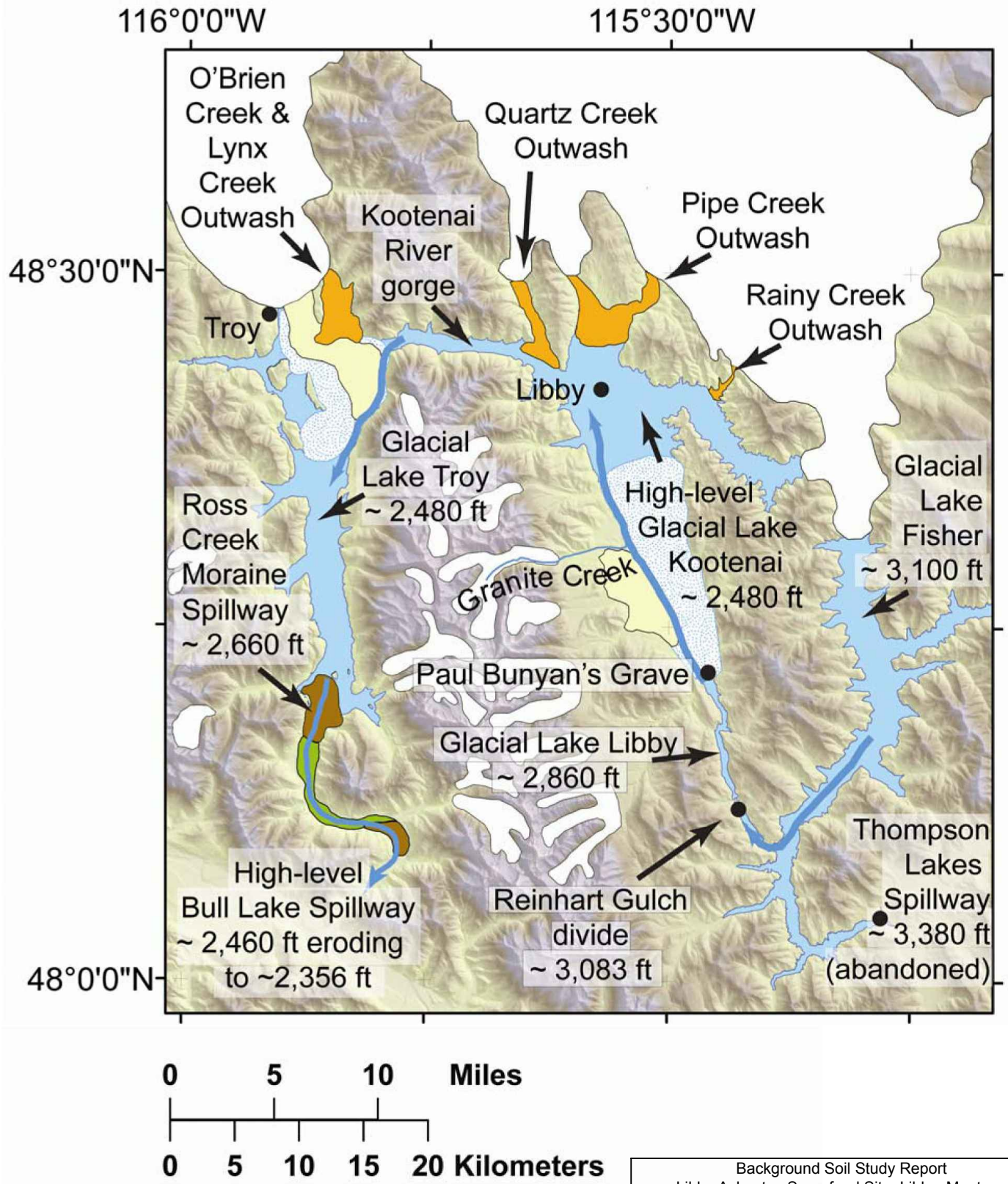
Background Soil Study Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 2-1**

LOCATION OF OPERABLE UNIT 7

TETRA TECH EM INC.

Figure_2-1_OU7_Location_031314.mxd - DWH - TTEMI HELENA



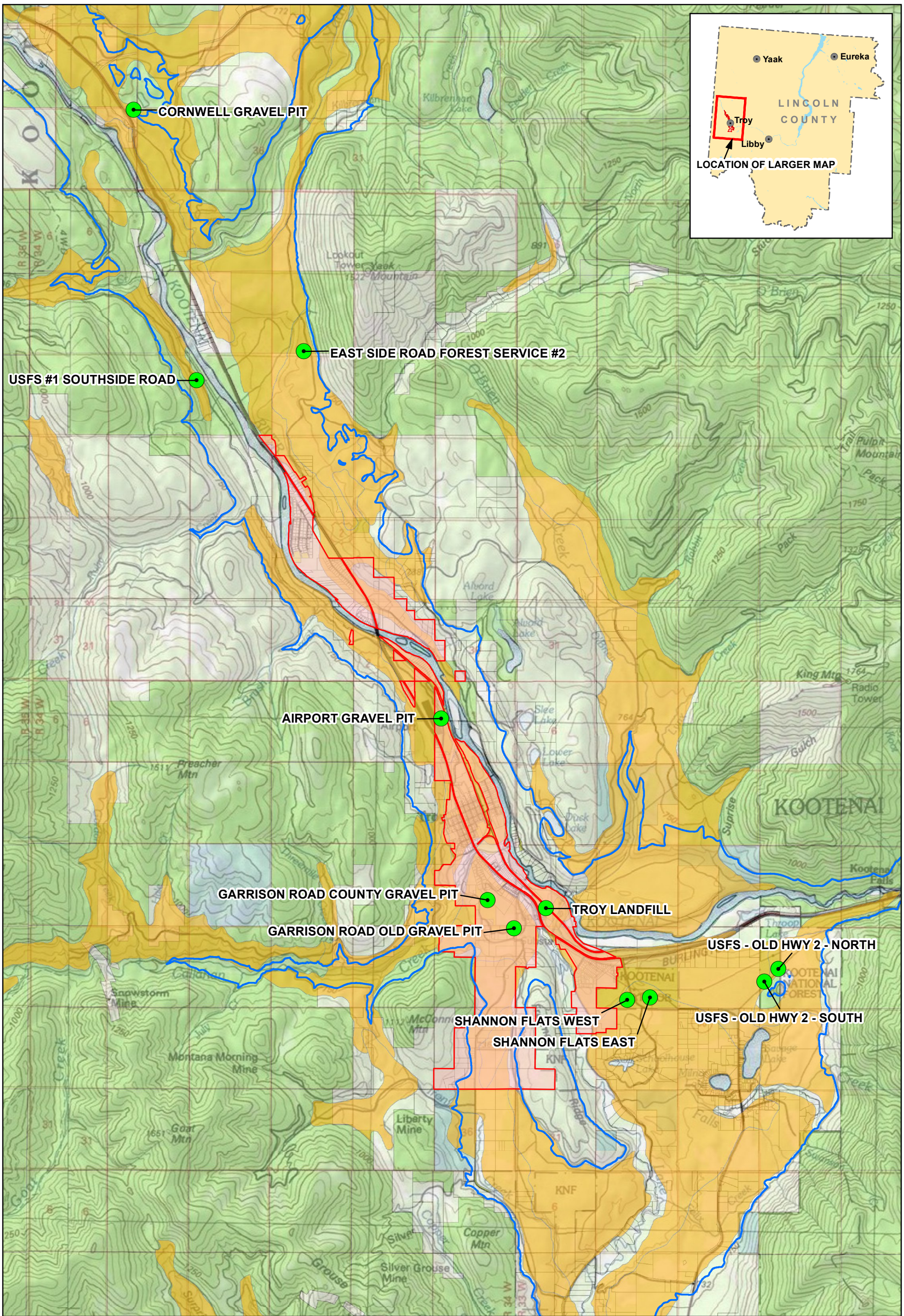

Source: Figure 5 in Langer et al. (2010); used with author permission

Background Soil Study Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 2-2**
GLACIAL LAKE KOOTENAI
DURING PLEISTOCENE TIME

TETRA TECH EM INC.

Figure_2-2_Glacial Lake Kootenai.dwg - DWH - 03/13/2014




Topographic Map Copyright:© 2011 National Geographic Society, i-cubed

**Legend**

- Background Sampling Area
- Maximum Elevation of Glacial Lake (2,450 feet above sea level)
- Soil mapping Units 102, 106, 108, and 110. Lacustrine Sediments and Glacial Outwash Sediments Source: SURRGO Soils:NCRS

Property Ownership

- Federal
- State Land
- Local Government
- Private
- Operable Unit 7 Area

N

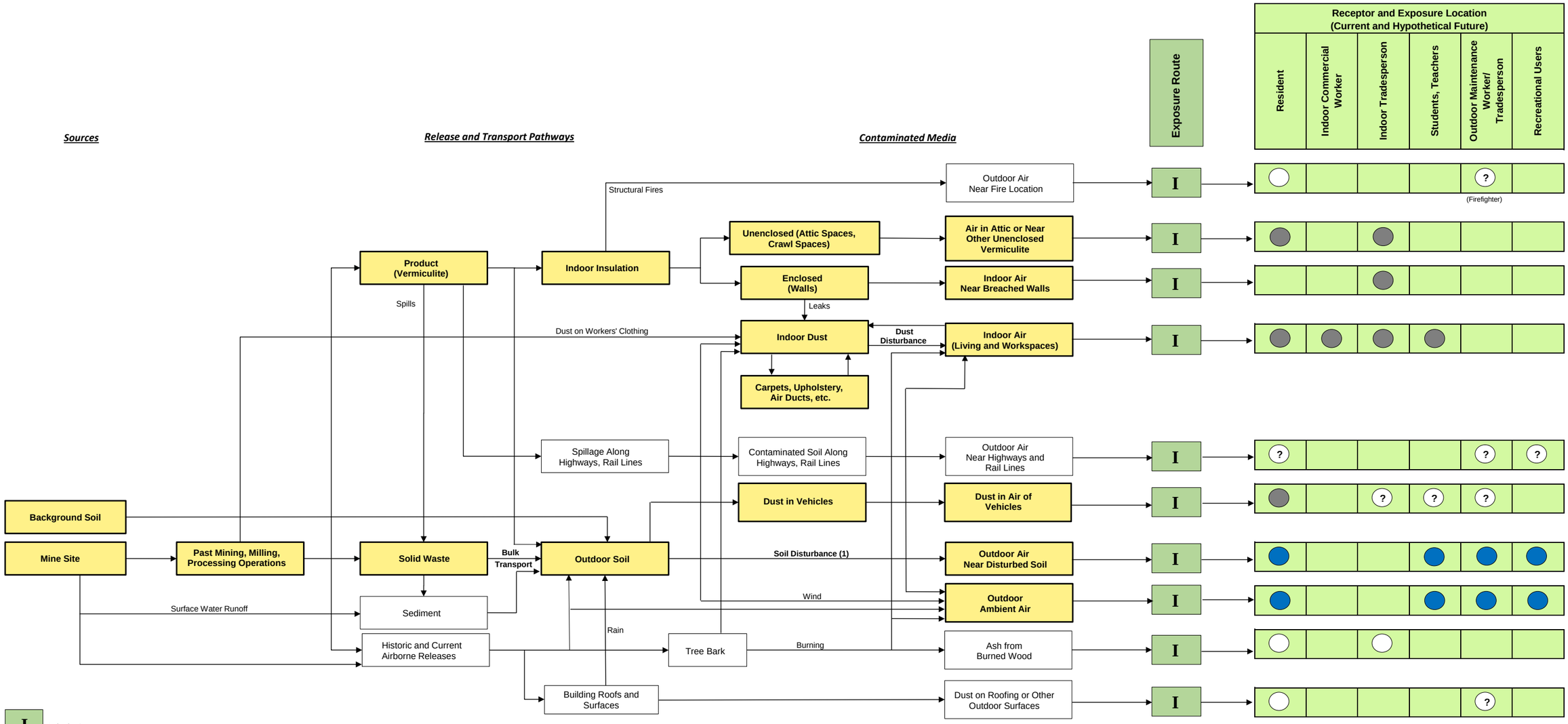
0 1 Miles

Background Soil Study Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 2-3**
LOCATION OF TROY AREA
LACUSTRINE/GLACIAL OUTWASH SEDIMENTS
AND BACKGROUND SAMPLING AREAS

TETRA TECH EM INC.

Figure_2-3_Background_Sampling_Areas_031314.mxd - DWH - TTEMI HELENA




I Inhalation exposure route

Pathway is complete and exposure may be significant; will be quantitatively evaluated in the Human Health Risk Assessment (HHRA).

Pathway is complete but is believed to be minor in comparison to other pathways; will be qualitatively evaluated in the HHRA.

? Pathway may be complete but magnitude of exposure is uncertain; will be qualitatively evaluated in the HHRA. Further investigation may be necessary.

Pathway is incomplete or believed to be negligible; Further evaluation is not warranted.

Medium investigated by the Troy Asbestos Property Evaluation (TAPE), Outdoor Ambient Air (AA), and the Activity-Based Sampling (ABS) Programs.

Receptor and exposure locations evaluated by the Background Soils ABS program for inclusion in the HHRA.

(1) Soil disturbance activities identified as representative of potential exposures are bicycling and playing on background soil.

Background Soil Summary Report
Libby Asbestos Superfund Site, Libby, Montana

**FIGURE 2-4**
Conceptual Site Model for
Potential Human Inhalation Exposure Pathways to Libby Asbestos
ABS Receptor and Exposure Locations
Operable Unit 7

TETRA TECH EM INC.

## *TABLES*

**Table 3-1**
**Locations, Activities, and Number of Samples for Background Study at OU7**

| Soil Samples | Location | Collection Technique | Soil Volume | Use/Analysis | Number of Samples | Notes |
|---|---|---|---|---|---|---|
| Background Soil For FBAS/TEM and ABS | Background Soil - 11 Locations | Two independent 30-point composite samples from 0-1" bgs (300 $ft^2$ area) | 500 gram zip-top bag (each sample) | Archive only | 22 | Two independent 30-point grab samples will be collected from a 300 $ft^2$ grid area at each background location. Each sample will thoroughly composited and the two samples from each background location will be archived. |
| | Background Soil - 11 Locations | Two independent 30-point composite samples from 1-6" bgs (300 $ft^2$ area) | 500 gram zip-top bag (each sample) | Background Soil for FBAS/TEM Analysis | 22 | Two independent 30-point composite samples will be collected from a 300 $ft^2$ grid area at each background location. Each sample will be thoroughly composited, then placed in a zip-top bag for delivery to the SPF. ESAT will split into three subsamples for 1) LA and Mineralogy, 2) PLM-VE, and 3) archive sample. |
| | Background Soil - 11 Locations | Two independent 30-point composite samples from 1-6" bgs (300 $ft^2$ area) | 5-gallon bucket | Child playing/digging ABS scenario | 22 | A single 5-gallon bucket of soil (consisting of 60 aliquots) will be collected from each background location using the same techniques as described for the FBAS/TEM samples. The soil will be used to perform ABS for the child digging scenario described below. A high and low volume pump will be worn by the field staff and one of the two resulting air samples will be selected for TEM analysis. |

| ABS Scenario | Location | Receptor | Age Group | Sample Height | Activity | Activity Time | Locations per Scenario | Events per Location (a) | Total Samples per Scenario |
|---|---|---|---|---|---|---|---|---|---|
| Background ABS | Background Soil (5-gallon bucket); Moved to Off-Site Location for ABS | Resident | Child | Breathing Zone for Child | Digging | 120 Minutes of Digging/Homoginizing Background Soil | 11 | 1 | 10 |

Notes:
ABS: Activity-based sampling
ESAT: Environmental Services Assistance Team
FBAS: Fluidized bed asbestos segregator
PLM-VE: Polarized light microscopy visual estimation
TEM: Transmission Electron Microscopy
LA: Libby Amphibole
TEM: Transmission Electron Microscopy

# TABLE 4-1 2012 OU7 Background Soil FBAS and ABS Air Results
## (Soil collected from 1 to 6 inch depth)

**OU7 BACKGROUND SOIL FBAS RESULTS**

| Background Sampling Location | Sample ID | Sample Date | Number of LA Structures (High Mag Only) | Concentration LA (s/g) (High Mag Only) | Number of PCME LA Structures | Concentration PCME LA (s/g) | LA Fiber Count by Mineral Class | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | WRTA-NaK Fibers | WRTA-NaX Fibers | WRTA-XX Fibers | WRTA-XK Fibers | AC-XX Fibers | TR-XX Fibers | CH-XX Fibers |
| USFS - Old Hwy 2 - North | TK-00002 | 11/26/2012 | 1 | 3.87E+04 | 0 | 0 | 0 | 0 | 1 | 0 | 0 | 0 | 0 |
| USFS - Old Hwy 2 - North | TK-00004 | 11/26/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| USFS - Old Hwy 2 - South | TK-00006 | 11/26/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| USFS - Old Hwy 2 - South | TK-00008 | 11/26/2012 | 2 | 3.68E+05 | 0 | 0 | 1 | 1 | 0 | 0 | 0 | 0 | 0 |
| Shannon Flats East | TK-00011 | 11/27/2012 | 1 | 9.68E+04 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| Shannon Flats East | TK-00011 | 11/27/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Shannon Flats East | TK-00011 | 11/27/2012 | 0 | 0 | 1 | 3.78E+04 | 0 | 0 | 0 | 0 | 1 | 0 | 0 |
| Shannon Flats East | TK-00013 | 11/27/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Shannon Flats West | TK-00015 | 11/27/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Shannon Flats West | TK-00017 | 11/27/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Troy LandFill | TK-00019 | 11/27/2012 | 2 | 1.94E+05 | 2 | 7.76E+04 | 1 | 1 | 0 | 0 | 0 | 0 | 0 |
| Troy LandFill | TK-00021 | 11/27/2012 | 2 | 7.74E+04 | 1 | 1.55E+04 | 0 | 1 | 1 | 0 | 0 | 0 | 0 |
| Garrison Road Old Gravel Pit | TK-00023 | 11/27/2012 | 1 | 3.87E+04 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| Garrison Road Old Gravel Pit | TK-00025 | 11/27/2012 | 0 | 0 | 1 | 1.55E+04 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| Garrison Road County Gravel Pit | TK-00027 | 11/27/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Garrison Road County Gravel Pit | TK-00027 | 11/27/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Garrison Road County Gravel Pit | TK-00027 | 11/27/2012 | 0 | 0 | 1 | 1.89E+05 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| Garrison Road County Gravel Pit | TK-00029 | 11/27/2012 | 0 | 0 | 1 | 1.85E+05 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| Airport Gravel Pit | TK-00031 | 11/28/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Airport Gravel Pit | TK-00031 | 11/28/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Airport Gravel Pit | TK-00031 | 11/28/2012 | 3 | 7.04E+05 | 2 | 1.89E+05 | 4 | 0 | 0 | 0 | 0 | 0 | 0 |
| Airport Gravel Pit | TK-00033 | 11/28/2012 | 2 | 1.85E+05 | 2 | 7.38E+04 | 4 | 0 | 0 | 0 | 0 | 0 | 0 |
| USFS #1 Southside Road | TK-00035 | 11/28/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| USFS #1 Southside Road | TK-00037 | 11/28/2012 | 1 | 9.13E+04 | 1 | 3.65E+04 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| EAST SIDE ROAD FOREST SERVICE #2 | TK-00039 | 11/28/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| EAST SIDE ROAD FOREST SERVICE #2 | TK-00039 | 11/28/2012 | 5 | 4.61E+05 | 8 | 2.95E+05 | 3 | 0 | 0 | 0 | 5 | 0 | 0 |
| EAST SIDE ROAD FOREST SERVICE #2 | TK-00039 | 11/28/2012 | 6 | 2.79E+06 | 1 | 1.89E+05 | 4 | 1 | 0 | 1 | 0 | 0 | 0 |
| EAST SIDE ROAD FOREST SERVICE #2 | TK-00041 | 11/28/2012 | 3 | 2.77E+05 | 4 | 1.48E+05 | 3 | 0 | 0 | 0 | 2 | 1 | 1 |
| Cornwell Gravel Pit | TK-00043 | 11/28/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Cornwell Gravel Pit | TK-00043 | 11/28/2012 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Cornwell Gravel Pit | TK-00043 | 11/28/2012 | 0 | 0 | 1 | 1.90E+05 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| Cornwell Gravel Pit | TK-00045 | 11/28/2012 | 1 | 9.13E+04 | 1 | 3.65E+04 | 2 | 0 | 0 | 0 | 0 | 0 | 0 |

**OU7 BACKGROUND SOIL ABS RESULTS**

| Background Sampling Location | Sample ID | Sample Date | Number of LA Structures (High Mag Only) | Concentration LA (s/cc) (High Mag Only) | Number of PCME LA Structures | Concentration PCME LA (s/cc) | LA Fiber Count by Mineral Class | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | WRTA-NaK Fibers | WRTA-NaX Fibers | WRTA-XX Fibers | AT-XX Fibers | AC-XX Fibers | AN-XX Fibers | CH |
| Shannon Flats East | TK-00047 | 12/4/2012 | NA | NA | 9 | 2.73E-03 | 6 | 1 | 2 | 0 | 0 | 0 | 0 |
| Shannon Flats West | TK-00049 | 12/4/2012 | NA | NA | 9 | 1.98E-03 | 2 | 0 | 7 | 0 | 0 | 0 | 0 |
| USFS - Old Hwy 2 - North | TK-00051 | 12/4/2012 | NA | NA | 3 | 1.35E-03 | 2 | 0 | 0 | 0 | 0 | 0 | 1 |
| USFS - Old Hwy 2 - South | TK-00054 | 12/4/2012 | NA | NA | 1 | 2.19E-04 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| USFS #1 Southside Road | TK-00055 | 12/5/2012 | NA | NA | 1 | 2.18E-04 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| Troy LandFill | TK-00057 | 12/5/2012 | NA | NA | 1 | 4.05E-04 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| Garrison Round Old Gravel Pit | TK-00059 | 12/5/2012 | NA | NA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Garrison Round CountyGravel Pit | TK-00070 | 12/5/2012 | NA | NA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Airport Gravel Pit | TK-00072 | 12/5/2012 | NA | NA | 5 | 1.62E-03 | 1 | 2 | 1 | 0 | 0 | 1 | 0 |
| EAST SIDE ROAD FOREST SERVICE #2 | TK-00074 | 12/6/2012 | NA | NA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Cornwell Gravel Pit | TK-00076 | 12/6/2012 | NA | NA | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |

Notes:

- All soil samples are 30-aliquot composite samples collected from the 1 to 6 inch depth.
- Samples TK-00011, TK-00027, TK-00031, TK-00039, and TK-00043, 3 FBAS filters were generated and sent to 3 different laboratories for analysis. Only one FBAS filter was generated from the remaining samples.
- Sample TK-00051, 2 LA structures were identified plus one Chrysotile structure identified and counted.

AT-XX Actinolite/Tremolite - Neither sodium nor potassium are clearly present
AC-XX Actinolite - Neither sodium nor potassium are clearly present
AN-XX Anthophyllite - Neither sodium nor potassium are clearly present
CH Chrysotile
LA Libby Amphibole asbestos
WRTA-NaK Winchite/Richterite/Tremolite/Actinolite - Both sodium and potassium are clearly present
WRTA-NaX Winchite/Richterite/Tremolite/Actinolite - Only sodium is clearly present.
WRTA-XX Winchite/Richterite/Tremolite/Actinolite - Neither sodium nor potassium are clearly present