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REMEDIAL INVESTIGATION REPORT

OPERABLE UNIT 7 OF THE LIBBY ASBESTOS SUPERFUND SITE

December 2010

Prepared for:

MONTANA DEPARTMENT OF ENVIRONMENTAL QUALITY
Remediation Division
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REVIEWS AND APPROVALS

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ACRONYMS AND ABBREVIATIONS

AAS	Outdoor Ambient Air Study
AHERA	Asbestos Hazard Emergency Response Act
ASTM	American Society for Testing and Materials
BNSF	Burlington Northern and Santa Fe
CAMA	Computer Assisted Mass Appraisal
CDC	Centers for Disease Control and Prevention
CDM	Camp, Dresser, and McKee
CERCLA	Comprehensive Environmental Response, Cleanup, and Liability Act
CIC	Community Involvement Coordinator
COC	Chain of Custody
CUA	Common Use Area
DEQ	Montana Department of Environmental Quality
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EPA	U.S. Environmental Protection Agency
ERS	Environmental Resource Specialist
FS	Feasibility Study
FSDS	Field Sample Data Sheet
GIS	Geographic Information System
GPS	Global Positioning System
LA	Libby Amphibole Asbestos
LUA	Limited Use Area
MBMG	Montana Bureau of Mines and Geology
NIOSH	National Institute for Occupational Safety and Health
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 (form)
PPE	Personal protection equipment
QA/QC	Quality Assurance/Quality Control
RDI	Removal design inspection
RI	Remedial Investigation
ROW	Right-of-Way
s/cm^2	Structures per square centimeter
SOP	Standard Operating Procedure
SUA	Specific Use Area
TAAD	Troy Ambient Air Database
TAPE	Troy Asbestos Property Evaluation
Tetra Tech	Tetra Tech EM Inc.
TEM	Transmission Electron Microscopy
TFO	Troy Field Office
TOAD	Troy Owner Access Database
UCL	Upper Confidence Limit

1.0 INTRODUCTION

Tetra Tech EM Inc. (Tetra Tech) has been tasked by the Montana Department of Environmental Quality (DEQ) under DEQ Contract No. 407026, Task Order No. 45, to prepare a Remedial Investigation (RI) report for Operable Unit 7 (OU7) of the Libby Asbestos Superfund Site. The United States 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 presents the data collected under the Troy Asbestos Property Evaluation (TAPE) Work Plan (Tetra Tech 2007) and Outdoor Ambient Air Study (AAS) Work Plan (Tetra Tech 2009a). These investigations were undertaken to assess the nature and extent of Libby Amphibole asbestos (LA) contamination within OU7. The data presented in this RI report are a culmination of TAPE, AAS, and associated activities that occurred from April 1, 2007 to December 31, 2009. The TAPE and AAS data will be used in the preparation of a Feasibility Study (FS), remedy selection, and the eventual remediation of OU7. Tetra Tech is the contractor for the DEQ and has assisted with all OU7 investigations to date.

1.1 REMEDIAL INVESTIGATION 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. The purpose of this RI report is to describe the approach used during the TAPE and AAS investigations, 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 removal design inspections (RDI) and removal actions;
- Gather sufficient data to support a future site-wide risk assessment;
- Allow for the effective development and evaluation of alternative remedies to be included in the FS.

Tetra Tech has 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 report will be updated annually as additional TAPE and AAS data are obtained, as removal activities are performed, as EPA residential and commercial removal action levels and clearance criteria are amended, and when a risk assessment is completed. 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 current 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 may influence on-going TAPE and AAS sampling requirements and protocols and could affect the number of properties requiring removal actions. Also, the results of additional TAPE investigations may impact the delineation of LA contamination within OU7.

It should be noted that a risk assessment has not been completed as part of this RI. However, once LA toxicity studies have been completed by the EPA, a site-wide risk assessment will be included in the appropriate annual addendum to this RI. The initial draft and the annual addenda to the RI will be combined with the risk assessment to form a future final RI for OU7.

1.2 REPORT ORGANIZATION

This RI report is organized into seven sections of text, which are followed by literature references, figures, and appendices. Tables are presented within the text. The contents of Sections 1.0 through 7.0 are briefly described below.

Section 1.0, Introduction - describes the report purpose and objectives, the report organization, and the site background and history.

Section 2.0, Physical Characteristics and Environmental Setting - describes the climate, geology, soils, surface water hydrology, hydrogeology, and population and land use of OU7.

Section 3.0, Libby Amphibole Asbestos - describes the historical use, physical and chemical properties, and general health effects of LA. This section also presents fate and transport information for LA and the EPA removal action levels.

Section 4.0, OU7 Asbestos Property Evaluation (TAPE) Investigation - describes the investigative objectives and procedures, field methods and activities, data management, quality assurance (QA) and quality control (QC) methods and samples, management of investigation-derived waste, opportunistic sampling, deviations from the TAPE Work Plan, analytical methods, and provides an assessment of data quality.

Section 5.0, OU7 Outdoor Ambient Air Study (AAS) - describes the investigative objectives and procedures, field methods and activities, data management, QA and QC methods and samples, management of investigation-derived waste, opportunistic sampling, deviations from the AAS Work Plan, analytical methods, and provides an assessment of data quality.

Section 6.0, Additional Removal Activities – summarizes the remediation of vermiculite along Callahan Creek, the removal of vermiculite from an abandoned foundation and stockpile on a portion of the Burlington Northern Santa Fe (BNSF) railroad right-of-way (ROW), and the number of residential parcels selected for removal action due to considerable contamination.

Section 7.0, Field Results and Nature and Extent of Contamination - summarizes the current understanding of the nature and extent of contamination based on field observations and data gathered during the TAPE and AAS investigations. This section also discusses response differences to visible vermiculite between OU7 and the other operable units within the Libby Asbestos Superfund Site.

Section 8.0, Conclusions and Recommendations - presents conclusions and recommendations based on this draft final RI report and describes the FS process.

1.3 DESCRIPTION OF OU7

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 operable units (OU) for 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. A description of the individual OUs for the Libby Asbestos Superfund Site is provided in Section 1.4 below.

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 upon 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.

1.4 HISTORY OF OU7

Vermiculite was mined and processed at the Rainy Creek vermiculite mine and associated processing operations located near Libby, Montana from the 1920's 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.

The EPA eventually split the Libby Asbestos Superfund Site into eight OUs to further focus the remedial activities in each unit of the site. Figure 1-2 shows the location of each OU; they are 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 BNSF railroad lines and ROW to the south.

- OU2 – The area of 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). 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.

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 clothes 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.

The 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. Approximately two-thirds of OU7 properties had a TAPE inspection performed between 2007 and 2009. Tetra Tech continues to complete TAPE investigations at eligible parcels. Figure 1-3 displays the boundary of OU7.

In 2009, Tetra Tech was tasked by DEQ to begin an assessment of LA contamination in outdoor ambient air. An AAS Work Plan was prepared and the monitoring began in the fall of 2009. The assessment of outdoor ambient air is scheduled to continue through fall 2010.

2.0 PHYSICAL CHARACTERISTICS AND ENVIRONMENTAL SETTING

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

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 °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 (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 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) 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, stramatolitic 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).

2.2.1 The Rainey 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 Rainey Creek Complex. The Rainey 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 <1 millimeter to >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).

2.2.2 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°C. When heated, the vermiculite plates expand at right angles to the cleavage (accordion-like) as the contained water is converted to steam (USGS 2005). Vermiculite was mined for 67 years from the Rainy Creek Complex and used in numerous consumer products in its expanded form.

2.3 SOILS

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.

2.4 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 8 miles of OU7, entering from the north near the neighborhood of Kootenai Vista and continuing through the site until exiting at the south near the Wilderness Plateau neighborhood.

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.

2.5 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 (US 2000 Census [Census] 2000). Of this total, approximately 1,982 live within the boundaries of OU7, which is about 10.5% of the county's total population. Of this total, 957 reside within the Troy city limits (Census 2000).

The median household income in Troy is $19,635. The primary occupation categories in Troy are service (24.3%) and professional or management (19.5%). Unemployment is high in Troy; of those persons aged 16 years and over, only 52.5% are in the labor force. The labor force is comprised of 55.6% private wage and salary workers, 27.8% government workers, and 15.7% self-employed workers. Approximately

eighty percent of the population 25 years and older have high school diplomas or higher (i.e. Associate degree) and 13.7% 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 government entities. Only 513,371 acres (22%) are privately owned. Of the total area, 1,759,003 acres (75%) is owned by the Federal government and 74,461 acres (3%) is owned by the State. The US Forest Service and Plum Creek Timber are the largest landowners in Lincoln County owning 1,727,772 acres (73.5%) and 294,603 acres (12.5%) respectively (Census 2000).

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

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 term specifically refers to six fibrous minerals that have been commercially exploited: tremolite, actinolite, anthophyllite, amosite, chrysotile, and crocidolite, although there are several naturally occurring minerals that are capable of producing asbestos like fibers. The asbestos minerals are categorized into two groups based on morphology, serpentine and amphibole. Of the six asbestos minerals listed previously, all but chrysotile fall into the amphibole asbestos group. 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] 2009a).

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 located 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 expands 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 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 (Center for Asbestos Related Disease 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 will address the issue of LA toxicity; until this toxicity has been determined, the risks associated with exposure to LA cannot be fully assessed. At the time this RI report was prepared, the Libby Action Plan studies were still in progress and toxicity of LA had not yet been determined.

3.4 EPA REMOVAL ACTION LEVELS

The EPA has established removal action levels and clearance criteria for the Libby Asbestos Superfund Site based on screening-level risk calculations that were 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 allow investigators to determine whether or not a property is contaminated to the point that a removal action is warranted. The EPA clearance criteria are used to determine when a removal action is sufficiently complete (EPA 2003). The EPA has been conducting removal actions in OU1 (export plant) and OU2 (screening plant) since 2000 and in OU4 (Libby) since 2002. It is expected that the EPA will issue a Record of Decision for OU4 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 dust when disturbed, and hence, removal or isolation of the source greatly reduces the potential exposure from one of the most significant pathways. 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 are used to determine if a removal action for the contaminated portion of the property in OU4 is 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". 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 a specific use area or other yard soils by any analytical method greater than or equal to 1% 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%)
- Bin B2 = Between 0.2% and 1%
- Bin C = LA greater than or equal to 1% by weight (EPA action level)

TAPE results are compared to the EPA removal actions and discussed in greater detail in Section 7.2.

4.0 TROY ASBESTOS PROPERTY EVALUATION (TAPE) INVESTIGATION

This section describes the objectives, methods, and procedures utilized during the TAPE investigations. The information presented in this section is a condensed version of 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 references that document as necessary for greater detail. The TAPE process was initiated in 2007 and is presently on-going. The majority of TAPE investigations for OU7 were completed between 2007 and 2009.

4.1 TAPE OBJECTIVES

As discussed in Section 3.3 above, previous investigations in Libby determined that LA was present in multiple environmental media including indoor air, outdoor ambient air, attic insulation, and soil. Due to: (1) the proximity of Troy to Libby; (2) the likely historical transport of asbestos-contaminated dust on the clothing and equipment of mine workers living in Troy; (3) limited investigations indicating the vermiculite insulation found in Troy is similar in morphology and mineralogy to vermiculite found in Libby; and (4) information indicating the Libby mine material was transported to Troy in the past, it was determined that the magnitude and potential exposure of Troy residents to LA was unknown and that additional data were needed to define the nature and extent of LA to support removal action decisions.

In response to this decision, the TAPE data collection process was formulated and specific TAPE Data Quality Objectives (DQO) were established to ensure adequate data were collected and under the appropriate conditions. The DQOs also specified the tolerable limits on decision errors that would be used as the basis for establishing the quantity and quality of data needed to support decision-making. A full description of the TAPE DQOs can be found in Section 3 of the Final TAPE Work Plan (Tetra Tech 2007).

The primary objective of the TAPE is to determine the eligibility of properties for removal actions and to obtain sufficient information to evaluate the properties if the eligibility criteria should change. This objective is supported by the *Draft Final Libby Asbestos Site Residential/Commercial Cleanup Action Level and Clearance Criteria Technical Memorandum* (EPA 2003), which outlines the criteria a property must meet in order to be eligible for removal activities and serves as the basis for the TAPE sampling methodology.

4.2 FIELD INVESTIGATION METHODS

The following sections present information on TAPE logistics and site access, inspection and sampling procedures, QA and QC samples, field documentation methods, and sample management.

4.2.1 Logistics and Site Access

Logistics

The TAPE investigation process focuses on residential, commercial, and rural properties within OU7. Road and alley parcels were also inspected as part of the TAPE. Because the work relies heavily upon owner access to each property, Tetra Tech has provided public outreach support (public meetings, fact sheets, newspaper articles and notices, and radio announcements) since 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. The Information Center is centrally located at 303 N. Third St. The DEQ Troy Information Center also serves as the Tetra Tech field office and is 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.

Tetra Tech staffs the DEQ Troy Information Center with a TAPE Field Team Leader, Community Involvement Coordinator (CIC), and Sample Database Coordinator. The TAPE Field Team Leader and CIC are responsible for obtaining site access agreements, assisting with public outreach, scheduling daily field activities, and providing quality control and oversight of the TAPE field teams. The Tetra Tech Sample Database Coordinator has the primary responsibility of checking and cataloging all field and quality assurance/quality control (QA/QC) samples at the end of each day and for working closely with the EPA sample coordinator to ensure that complete, adequate, and secure sample information is collected and transferred to EPA.

In addition to the staff discussed above, Tetra Tech also provides TAPE field team members stationed in Troy during the field effort. The average field team size has been two to four persons. All field team members are certified in hazardous waste operations and trained to properly handle the health and safety protocols for the project. At least one field team member must hold a current AHERA asbestos inspector training certificate. All field personnel were trained on sample collection, community

relations, health and safety, and the overall objectives of the TAPE. The field team leader accompanied the field teams on a rotational basis to ensure and verify that the teams were conducting the TAPE activities as described and outlined in the work plan.

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. Unique two-letter, six-digit address numbers (AD numbers) were assigned to each parcel for tracking purposes. The unique AD numbers begin at 200001 (AD-200001) and go up; this was done so 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 4.5.1.

Approximately one month before the TAPE field activities began in 2007, Tetra Tech assisted the DEQ with mailing access agreements 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, MT. 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 is required for each parcel of land. This process was repeated prior to 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 by telephone to schedule a TAPE inspection and sampling visit. 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 any pertinent questions. Tetra Tech will provide 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 TAPE field activities.

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 continues currently. Procedures and activities in support of community outreach for OU7 are summarized below.

The DEQ Troy Information Center office is staffed with a full-time CIC that has been 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 from spring 2007 through 2010, 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 have also been sent property reports summarizing inspection results at their parcel(s). In addition, as new information regarding properties or events occur in OU7, the DEQ sends the public informational mailings by letter or postcard.

The DEQ and the EPA also notify the public in OU7 about scheduled meetings in Libby through local newspapers and radio announcements. Meetings may include 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

The following sections briefly outline the methods and procedures of the TAPE inspection process. For a more thorough description of TAPE procedures, please refer to the Final TAPE Work Plan (Tetra Tech 2007).

At the start of each field day, Tetra Tech field teams received a packet of information and a hand-held GeoXT personal digital assistant (PDA) with specific information for each property to be inspected and sampled that day. The PDAs were loaded with required field forms and were used to record the majority of inspection data. The packet of information included:

- Confirmation that the office has a signed access agreement or blank access agreement if occupant provided prior verbal agreement.
- Details of the scheduled inspection date and time, and the name and telephone number of the property owner or the person who will be present for inspection and sampling, if different than the property owner.
- Preprinted property-specific, building, sample point, and sample identification labels.
- Numbered field logbook.
- One copy of the property parcel map on an aerial photo.
- Graph paper for field sketch documentation.
- Hard copy of all field data collection forms as backup in case the PDA fails.

4.2.3.1 Interior Inspection and Sampling

The TAPE investigation commenced with a verbal interview with the property owner or resident. 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 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 and exterior use areas located on the property. Each building on the property was assigned a unique building identification number (BD-2XXXXX) for tracking purposes.

Visual Inspection

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. 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 insulation. Visual inspections did not involve opening up walls or ductwork to inspect for vermiculite insulation within the building wall cavities. 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. Details on ERS activities can be found in Section 7.1. 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

Dust samples were collected from primary buildings and secondary buildings with finished floors (i.e. material other than dirt) as part of the TAPE inspection process during 2007 and 2008. The collection of dust samples was ceased after the 2008 field season when it was determined 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 Final TAPE Work Plan (Tetra Tech 2007); deviations from the work plan and the rationale behind them are discussed in Section 4.3.

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 microvacuum (microvac) sampling techniques. The dust

samples were collected following the procedures detailed in CDM-LIBBY-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) was 100 square centimeters 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 complete, 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 five-digit number.

Soil Sampling

Soil samples were collected from all 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. 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.

4.2.3.2 Exterior Inspection and Sampling

Visual Inspection

All areas of OU7 properties not covered with buildings were inspected for vermiculite product in soil and surface materials. These exterior areas were grouped into four general 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 use area identification number (UA-2XXXXX) was assigned to each individual exterior use area that was delineated and inspected. For the TAPE, visual point inspections correlated exactly with soil sample aliquot locations. There was a minimum of 5, and a maximum of 30, visual point inspections per use area.

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 use area. Each composite was obtained from either a depth of zero to three inches for CUAs and LUAs or from a depth of zero to six inches for SUAs. No soil samples were collected from non soil-covered areas such as parking lots or other outdoor paved areas. Each aliquot was examined for the presence of visible vermiculite. The amount of visible vermiculite was categorized as none, low, intermediate, or high using the procedures defined in CDM-LIBBY-06 (Appendix A). The visible vermiculite was also described as being expanded or unexpanded. Once all of the soil aliquots had been obtained and examined for visible vermiculite, 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, Tetra Tech was requested by DEQ to add OU7 road and alley parcels 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 4-1 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 unique AD numbers 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 the 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 visible vermiculite. Similar to the exterior use area procedure, the visible vermiculite 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 visible vermiculite 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 surface 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 adjacent road edge/borrow ditch. Each aliquot was inspected for visible vermiculite. 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 (TT number).

4.2.5 QA/QC Samples

Various QA/QC were collected as part of the TAPE investigation to satisfy data quality objectives. The basis for the TAPE QA/QC sampling (type, frequency, etc) is outlined in the *Draft Site-Wide Quality Assurance Project Plan* (CDM 2007a), which was prepared for the Libby Asbestos Superfund Site. The procedure for the collection of dust field blank samples was modified as necessary for OU7. All QA/QC samples collected during the TAPE were submitted to the laboratory as "blind" samples (labeled as a collected sample).

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 1 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. Data for dust lot blank samples will be 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 5 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 (Monday through Sunday) 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 method EPA 100.2, modification 20. 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.

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. The following paragraphs provide a brief explanation of TAPE field documentation procedures. Additional information on the handling of field documentation materials can be found in Section 4.5.2.

All TAPE data, including answers 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 provided a means for limiting the occurrence of data entry errors. Use of the PDA also allowed for compilation of TAPE data into the TAPE Scribe Database. A description of Scribe is located in Section 4.5.3.

The field teams were also supplied with TAPE field logbooks. Any additional information that was not recorded on the PDA field forms was recorded in the 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 sketch showed 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 visible vermiculite detected during the exterior visual inspection. Requirements for field sketches are discussed in greater detail in the Final TAPE Work Plan (Tetra Tech 2007).

Each TAPE field team was provided with a digital camera for photo-documenting primary characteristics of inspected parcels. The field teams collected photographs of the primary and secondary buildings, attic insulation, any vermiculite insulation or visible vermiculite discovered on the premises, use areas, and any other notable features. The home owner or resident was asked for permission before any photographs were taken. The photographs were recorded in the field logbook.

4.2.7 Decontamination and Investigation-Derived Waste Handling

Decontamination

Dust sampling activities required minimal decontamination as a new cassette and a disposable paper template was 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 paper towels were placed in a labeled asbestos waste bag.

Re-usable 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 on the ground surface within the area just sampled and the paper towels were placed in a labeled asbestos waste bag.

Field team members were required to wear 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, were outlined in the project health and safety plan (Appendix A of the Final TAPE Work Plan). Personal protection equipment (PPE) included disposable gloves, disposable protective outerwear, work boots, disposable boot covers, and respirators. Respirators were decontaminated in accordance with the project health and safety plan.

Investigation-Derived Waste Management

Investigation-derived waste 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 contained some asbestos. Therefore, all investigation-derived waste was double-bagged in appropriate asbestos bags, labeled with asbestos labels, and stored in an approved containment area at the Tetra Tech Troy field office where it was later transferred to an approved landfill (Lincoln County outside of Libby) for final disposal. Non-sampling waste generated by the TAPE field teams, such as food containers and waste paper, was separately bagged and properly disposed of as solid waste.

4.2.8 Sample Management

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 Troy field office/equipment shed and wrote the corresponding number of samples on a specific form for the Troy sample coordinator. The sample coordinator then printed chain of custody (COC) forms 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. The sample coordinator stored the samples in a secure area until the samples were transferred to the off-site analytical laboratory (dust) or the sample preparation laboratory (soil) under a COC.

4.2.9 Record Keeping and Chain of Custody

At the end of each field day, the TAPE field teams returned to the Tetra Tech Troy field office to download the PDA, catalog and store all field and QC samples, and turn in the appropriate logbook and paperwork to the Tetra Tech sample coordinator (or the coordinator's designee). The field teams were also responsible for accurately labeling and downloading the day's digital photographs to a computer at the Troy field office/equipment shed. Management of field documentation (logbook, property sketch, etc) is discussed in Section 4.5.2.

The TAPE Scribe Database is capable of generating COC information. Once inspection data on the PDA has been reviewed for accuracy and imported into Scribe (Section 4.5.3), the Sample Database Coordinator created COCs for all of the samples collected (water, soil, air). Scribe automatically assigns a number to the COC so it can be tracked in the database. COCs are reviewed by the field teams that collected the samples, and then signed, dated, and placed with the samples in a bin in the Troy field office/equipment shed.

4.3 DEVIATIONS FROM THE TAPE WORK PLAN

The following sections discuss modifications in TAPE procedure and the collection of opportunistic field samples.

4.3.1 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 close proximity to the erroneous entry where

possible. Errors encountered after the document had been scanned were corrected using electronic comments attached to the PDF file using Adobe Acrobat.

Project-wide modification was necessary on occasion and required the DEQ Project Officer to consult with the EPA Remedial Project Manager for the modification. When a modification was required, a Troy Field Office (TFO) Record of Modification form was filled out by the DEQ Project Officer. The Tetra Tech project team did not implement the modification until verbal or written approval was granted by the DEQ Project Officer. Table 4-1 below summarizes the modifications for the TAPE project; there have been fourteen TFO modifications through December 31, 2009. Copies of the applicable modification forms are provided in Appendix B.

TABLE 4-1
TROY FIELD OFFICE (TFO) RECORD OF MODIFICATIONS THROUGH DECEMBER 31, 2009

TFO Record of Modification Number	Date of TFO Record of Modification	Modification to TAPE
TFO-00001	5/8/2007	Required number of dust sampling aliquots changed 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 detected, 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 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%.
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.

4.3.2 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 is low. This is why 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 teams determined that an NUA was disturbed, the use area was re-categorized (i.e. LUA or SUA) and sampled accordingly.

In some cases, property owners requested that a soil sample be collected from the NUA on their parcel. Between April 1, 2007 (project inception) and December 31, 2009, five NUAs were sampled on developed parcels and three entirely NUA parcels (undeveloped parcels) were sampled.

4.4 TAPE ANALYTICAL METHODS

The analytical methods used for TAPE interior dust sample analysis and soil sample analysis are outlined in the sections below. Copies of the referenced analytical methods, SOPs, and Libby modifications are provided in Appendix A.

4.4.1 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 ≥ 0.5 µm; aspect ratio ≥ 3:1) and sample preparation techniques per Libby Asbestos Superfund Site modifications for investigative samples.

4.4.2 Soil Sample Analysis

Soil samples were analyzed using PLM-VE. Sample preparation was performed in accordance with Libby standard operating procedure (SOP) SRC-Libby-01 and sample analysis was performed in accordance with Libby SOP SRC-Libby-03, Analysis of Asbestos Fibers in Soil by Polarized Light Microscopy. Soil

samples from OU7 were not analyzed using the additional PLM-Grav method, as the results from testing soils using this method would not alter whether or not a property was eligible for removal.

4.5 TAPE DATA MANAGEMENT

The following sections describe the management of data associated with parcel inspections, sample collection, and sample analyses. Additional information on TAPE data management can be found in the Troy Data Management Plan Version 3.0 (Tetra Tech 2009c).

4.5.1 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. Periodically since that time, the State of Montana has released updates to the cadastral (geography) and CAMA databases. The Tetra Tech Troy Geographic Information System (GIS) coordinator processes revisions from both sources before they are incorporated into the parcel geodatabase. Parcels that have been subdivided, combined, or had any other boundary changes are updated and the changes noted in the OU7 parcel geodatabase.

Occasionally parcels are legally combined or subdivided. If parcels have been legally combined, then one or more AD numbers must be removed from the parcel geodatabase. When a parcel is subdivided, a new (unique) AD number must be assigned, and if any data are associated with the subdivided parcel, it must be 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.

Sometimes a field team would discover an adjacent parcel owned by the same landowner that shared a building or major use area with the parcel scheduled for inspection. The parcels were listed as separate properties in CAMA, but because they shared a structure or major use area (e.g. house, driveway) with another parcel, realistically they would not be considered separate. In these cases, the landowners had not legally combined the two parcels, and the field teams had to designate one of the parcels as a reference parcel. As of December 31, 2009, a total of 85 inspected parcels have been referenced to an adjacent parcel since the beginning of the 2007 field season. Ten of these are road and alley parcels.

GPS Data

In 2007, field teams attempted to collect global positioning system (GPS) geographical coordinates (latitude and longitude) from each exterior use area to demarcate 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 recorded only one set of coordinates for each inspected parcel. The GPS reading was collected approximately 20 feet outside the main entrance to the primary building on the parcel, on a defined use area (yard, driveway, etc). The information was captured using the field forms programmed onto the PDA. The latitude, longitude, and other GPS data are exported from the handheld computer and imported into the TAPE Scribe Database.

4.5.2 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 are required prior to any field activities on a subject property. Access agreements are mailed to parcel owners prior to the field season. The CIC schedules inspections with property owners who have returned signed access agreements to the DEQ Troy Information Center. Some landowners provide verbal approval and schedule an inspection over the phone. In this case, the field team obtains a signature immediately prior to conducting the inspection. The original, signed, access agreements are maintained in file folders for each parcel (by AD number) in the DEQ Troy Information Center. The original access agreements are also scanned into a portable data format (PDF) and are included in the electronic data archive for each property.

Logbooks

Information pertaining to the field inspection is recorded in TAPE field logbooks. Each field team maintains 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. Once a field team arrives at a scheduled parcel (AD number), they assign BD, UA, and TT numbers. These numbers are the core of the field data. Each assigned number, as well as any sampling information that pertains to it, must be recorded in the logbook.

A new page is started in the field logbook for each property. 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 are scanned into individual PDF files and stored as part of the electronic data archive for each property. The hard copy original logbooks are stored in the DEQ Troy Information Center.

Property Sketches

A property sketch is generated for each property inspected. The sketch depicts 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) are 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 visible vermiculite. The sketches are prepared on 8.5- by 11-inch graphing paper and are mandatory for each property inspected. Original hard copy property sketches are maintained in the file folders in the DEQ Troy Information Center. Property sketches are 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 is required for every occupied property inspected (with the exception of properties that are entirely non-use), regardless of whether it is occupied at the time of inspection by

the owner, a tenant, or an employee. Information requested on the POC form includes 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 are maintained in the project files in the DEQ Troy Information Center. A scanned copy (PDF format) of the POC form is maintained in the electronic data archive for the property.

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

Photographs

The field teams take photographs during TAPE inspections 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 are asked for permission before any photographs are taken, unless the photographs are taken from public rights-of-way. All photographs are taken using digital cameras and a description of each photograph is recorded in the logbook. The photographs are downloaded at the DEQ Troy Information Center the same day as the inspection, and saved in a directory of field photographs. The images are 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.

Management of Field Documentation

Field forms and photographs, along with other information (including access agreements, logbooks, property sketches, and POC forms) generated during the inspection process, are archived in two ways. Original field forms and other hard copy documentation are filed, or archived, according to AD number in the DEQ Troy Information Center. The CIC is responsible for archiving these documents. The second type of archiving is electronic. Field forms and other hard copy documentation are scanned into PDF format. The PDF files are stored, according to AD number, in the electronic data archive for the parcel. The Sample Database Coordinator maintains the electronic data archive in the DEQ Troy Information Center during the field season. Backup copies of the archive are prepared on a weekly basis and sent to the Tetra Tech office in Helena, Montana for safekeeping. At completion of the field season, the master

electronic data archive is transferred to the Tetra Tech office in Helena, where it is maintained by the Electronic Data Archive Coordinator.

4.5.3 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.

PDA Setup and Modification

Each field team was provided a handheld computer (PDA) for data and GPS point acquisition. The geographic software ArcPad was loaded onto each of the PDA handheld computers to support parcel GIS layers. In addition, aerial photographs of OU7 were uploaded onto the PDA 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 TAPE 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 modification.

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, how many attics, what types of buildings – sheds, garages, homes, etc.), sample information (number of aliquots, location of samples, etc), and the presence or absence of visible vermiculite 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 is 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 gives the handheld computer to the Sample Database Coordinator, who reviews 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 computer are exported as an extensible markup language file and then imported into a desktop Access database where the Sample Database Coordinator verifies them. After verification, the data are imported into the TAPE Scribe Database where they are 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 is 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 are 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).

4.5.4 Analytical Data

Analytical data were delivered to Tetra Tech from the laboratories responsible for analyzing TAPE dust and soil samples in an electronic format. Tetra Tech data validation and management personnel were copied on the messages and an assessment of data quality for each deliverable was subsequently initiated. Section 4.6 below describes the data quality assessment process. Each electronic data deliverable (EDD) and associated bench sheet file was filed on the Tetra Tech network in Helena, Montana and served as a backup copy.

4.6 TAPE DATA QUALITY ASSESSMENT

Tetra Tech assessed the quality of both field and laboratory data generated during the TAPE to ascertain whether they satisfied project data quality objectives specified in the Final TAPE Work Plan (Tetra Tech 2007). The field data verification process is summarized in Section 4.6.1 and laboratory data verification process is summarized in Section 4.6.2.

4.6.1 Field Data Verification

Tetra Tech verified the accuracy and completeness of TAPE field documentation collected and recorded during the 2007, 2008, and 2009 field seasons in accordance with the procedures outlined in the Data Management Plan Version 3.0 (Tetra Tech 2009c), developed for the TAPE project.

TAPE field documentation included all data compiled and recorded on field forms, on PDAs, and in photographs taken during property assessments conducted pursuant to the TAPE project. Verification of field documentation was done to ensure, to the greatest extent possible, that all necessary information was entered completely and accurately into logbooks and handheld computers; that photographs were correctly and adequately cataloged; and that no discrepancies existed amongst the various records as documented in the TAPE Scribe Database and electronic data archive for each property. Resolution of field documentation issues was done to rectify inaccuracies and discrepancies, so that the final record for each property was as accurate and complete as possible.

The verification process was two tiered. The tiers were referred to as Category 1 and Category 2. The verification tiers were primarily designed to eliminate incorrect (in particular, false negative) determinations of visible vermiculite from record, as well as to ensure the highest level of accuracy of the information recorded in the TAPE Scribe Database and electronic data archive.

Individual Category 1 and Category 2 errors were tracked for various purposes, and assigned to various groupings as needed. For example, the number of errors where the presence of vermiculite was misidentified in the database, referred to as high-profile errors, was tracked. In addition, the number of errors that may cause a no-removal-required property to require removal actions, referred to as critical errors, was tracked. Critical errors are a subset of high-profile errors.

Category 1 Verification

Category 1 verification was done to identify inconsistencies within the TAPE Scribe Database. It was conducted on 100 percent of the data in the TAPE Scribe Database generated during each field season. Category 1 verification involved two general types of queries of the TAPE Scribe Database: global queries and relational queries. Global queries were conducted first, followed by relational queries. Global queries were done sequentially, so that the broader-based discrepancies were eliminated first. Example global query topics include: (1) identification of blank fields, (2) identification of missing TT or UA numbers, and (3) identification of obvious errors, such as the area of a primary residence listed as 50 square feet. After the global queries were run and any issues were resolved, relational queries were conducted. Relational queries were also completed on 100 percent of the data in the TAPE Scribe Database generated during each field season. Similar to the global query process, relational queries

were completed sequentially, with discrepancies being resolved before moving on to subsequent queries.

Examples of relational queries used for Category 1 verification include:

- The land use description must correspond appropriately to the land use category. For example, if the value in the database for land use description is "C – Decorative Gravel/Rock," then the land use category should be "Common Use Areas."
- If the value in the database for "DOES THE INTERIOR HAVE VERMICULITE ATTIC INSULATION?" is "*No attic*," then the value in the database for "EXTENT OF FINISHING IN THE ATTIC AREA?" should be "*No attic.*"

The Scribe Database Administrator ran the global and relational queries and depending on the issue, either addressed it directly or designated a Data Verification Analyst to resolve it. The verification analyst documented how to resolve each issue in a modification tracking (ModTrack) form. This form was an Excel spreadsheet that allowed the verification analyst to record and track necessary changes by identifying specific items requiring resolution as well as the corresponding modification. The Scribe Database Administrator compiled each of the ModTrack Excel files into a master ModTrack Access database file as a record of the changes. The Scribe Database Administrator then made the corrections to the TAPE Scribe Database as indicated in the ModTrack forms.

Category 2 Verification

Category 2 verification was done to identify errors in the written documentation (e.g., logbook entries and sketches) as well as inconsistencies between the TAPE Scribe Database and written documentation. Category 2 verification was performed on at least 25% of field forms, logbooks, and photo documentation during and after the 2007, 2008, and 2009 TAPE field seasons. In addition, many property database queries and written documentation entries were reviewed due to specific project needs, resulting in greater than 50% Category 2 verifications on many of the properties. The properties verified were inclusive of properties reviewed during the Category 1 verification process. Category 2 verification entailed cross-checking data contained in the TAPE Scribe Database with the various documents contained in the electronic data archive.

Upon review and verification of all field information for a given property, the Data Verification Analyst recorded findings for the property on the verification checklist on Tetra Tech's internal TAPE web portal. Resolution of issues identified during verification sometimes required modifications to the TAPE Scribe Database and/or the electronic data archive. Modifications to the TAPE Scribe Database were performed by the Scribe Database Administrator based on the information presented in the ModTrack forms. Modifications to the electronic data archive were done by the Data Verification Analyst by adding electronic comments to the affected PDF files such that the original document was not altered, but the modifications were clearly indicated in comments that could be viewed in both the electronic file and on a hard copy printout of the file. Typically, verification analysts made these modifications directly to the associated PDF documents and coordinate updates to the master archive with the Electronic Data Archive Coordinator.

4.6.2 Analytical Data Review and Verification

Tetra Tech verified the accuracy and completeness of TAPE laboratory results generated during the 2007, 2008, and 2009 field seasons in accordance with procedures outlined in the TAPE DMPs (Tetra Tech 2008a, 2009d, 2009c).

Analytical data were received from the laboratory in two formats: (1) sample receipt documentation and laboratory 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 results.

The analytical 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 Final Troy Data Management Plan Version 3.0 (Tetra Tech 2009c). The verification steps were conducted concurrently with data validation, which, while closely associated with data verification, was the mechanism to ensure the quality of the data by verifying that laboratory procedures were consistent with analytical method requirements and were consistent between laboratories and analysts.

Dust Analytical Data Validation

Tetra Tech conducted data review and data entry verification of the dust sample analytical data in accordance with SOP EPA-LIBBY-09 (revision 1). 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's review and verification process involved three steps: (1) the selection of data records for review and verification, (2) a review of the original laboratory bench sheets, and (3) verification of the transfer of results from the bench sheets into the TAPE Scribe Database.

Selection of TEM Records for Review

To ensure that a representative subset of TAPE dust sample results were reviewed and verified, a minimum of 10 percent of the TEM dust sample results were selected for review and verification. These were selected through a query of the TAPE Scribe Database. The query was designed to randomly select a representative number of results by laboratory analyst as well as result type (detected/nondetected).

Consistency Review of Laboratory Bench Sheets

Consistency reviews of laboratory bench sheets were done to identify the occurrence of any data omissions, apparent inconsistencies, and/or potential errors in structure. The bench sheets include the laboratory COC form, sample check-in form, preparation log, and data record sheets. Scanned copies of the original hand-written bench sheets were reviewed to determine whether the raw structure data were recorded in accordance with AHERA/ASTM and applicable laboratory modifications. Information reviewed includes verifying (1) whether recorded structure types are consistent with the counting rules, (2) whether the total column is populated with non-zero numbers for all countable structures and zeros for all non-countable structures (3), whether all recorded non-asbestos mineral structures are indentified as non-countable structures, (4), whether the recorded structures meet counting rule requirements (e.g., length, width, and aspect ratio), whether the recorded dimensions for matrices are the protrusion dimensions, not the matrix dimensions, (5) whether the mineral class is populated for all structures, (6) whether the mineral type and appropriate spectra code is recorded in the structure comment field for all recorded LA, other asbestos, and non-asbestos mineral structures, (7) whether the structure comments are supported by recorded data, and (8) whether the stored values in the TAPE

Scribe Database for primary total structure type, length, width and mineral class match the original bench sheet.

Verification of Data Transfer from Bench Sheet to Database

Verification of data transfer from bench sheets to the TAPE Scribe Database was done to ensure that data from the bench sheets were transferred into the TAPE Scribe Database without error or omission. Tetra Tech compared the analysis-specific information in the TAPE Scribe Database to that on the bench sheets. This included verifying consistency of analytical method, analysis date, laboratory name, laboratory job number, laboratory sample number, preparation method, filter status, primary effective filter area, secondary effective filter area, grid opening area, F-factor, dust sample area, and analysis comments. Tetra Tech recalculated the effective filter area for all samples verified and the F-factor for all samples that underwent indirect preparation, recounted the total number of grid openings evaluated on the hand-written bench sheets and compared that to the AnalysisGOCounted entry in the TAPE Scribe Database, recounted the total number of countable LA structures and compared it to the binned LA values in the EDD.

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 Validation

Tetra Tech conducted data review and data entry verification of the soil sample analytical data in accordance with Libby PLM validation SOP (version 1). The SOP describes a standardized method for review of raw PLM data and verification of PLM 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.

The Analytical Data Validation Analyst completed a validation 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 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.

4.6.3 Reconciliation with Data Quality Objectives

DQOs are developed to help clarify study objectives, define the most appropriate data to collect and the conditions under which to collect it, specify tolerable limits on decision errors, and establish required analytical sensitivities. This information is used to determine the quantity and quality of data needed to support project-related decision making, and, later on, to assess whether the data collected are of adequate quality and quantity to satisfy project needs. DQOs for the TAPE project were presented in the Final 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* (EPA 2003) document. Three questions and associated decision rules were established for the TAPE project.

1) Does vermiculite insulation and/or visible vermiculite need to be removed from specific properties in OU7? If uncontained, migrating vermiculite insulation or visible vermiculite is observed in indoor living spaces or working spaces, or uncontained vermiculite insulation or visible vermiculite is observed in attics, the property will require a response action to remove the vermiculite insulation and /or visible vermiculite.

2) Do specific properties in OU7 contain levels of LA in soil that need to be addressed by short-term response actions? If visible vermiculite is observed within a specific use area on the property, a short-term response action will be conducted to remove it from the specific use area. If the average concentration of LA in soil within any use area on the property exceeds the action level for soil established by EPA in a decision document for OU7, any detectable LA in soil on the property will be removed in a short-term response action.

3) Do specific properties in OU7 contain levels of LA in dust that need to be addressed by short-term response actions? If the average concentration of LA in dust over an interior building level (floor) exceeds the action level for indoor dust established by EPA in a decision document for OU7, the entire building level from which the dust sample was collected will be subject to removal action.

4.6.3.1 Sampling Design

To address the first question, interior inspections for vermiculite insulation and visible vermiculite 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 within each use area. One composite sample consisting of 30 subsamples was collected from each use area. The sampling depth depended on the type of use area, to account for anticipated exposure to deeper soil horizons in specific use areas (e.g., gardens).

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 within each building level. One composite sample consisting of 10 subsamples was collected from each building level (floor). A specific number of subsamples was collected from each of 3 designated sub-area types, as applicable to the building: accessible areas (4 subsamples), infrequently accessed areas (4 subsamples), and inaccessible areas (2 subsamples).

4.6.3.2 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* (EPA 2003) 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 vermiculite or vermiculite-containing insulation is observed during a visual inspection of a property, there should be no more than a 10 percent chance that vermiculite or vermiculite-containing insulation is, 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 visible vermiculite was as accurate as possible.

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

Soil. If the measured average concentration of LA in soil within any use area on the property 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 appears to overestimate rather than underestimate the true concentration of LA in soil, so the risk of false negative decisions is presumed to be low. In addition, both field and laboratory duplicate samples were included in the TAPE analytical program, and the highest of the original and duplicate sample result was reported, 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 and the highest of the original and duplicate sample result was reported. 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 (supporting documentation for TFO-012, Appendix B). Since dust sample collection program was discontinued, the sample variability (based on field duplicate pair results) was not determined.

4.6.3.3 Analytical Sensitivity

OU7 dust and soil sample analytical sensitivities are 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* (EPA 2003). These removal criteria are LA asbestos levels of 1 percent for soil and 5,000 LA asbestos structures per square centimeter (s/cm^2)(using AHERA counting rules) for dust. The dust and soil analytical sensitivities used for the TAPE are low enough to detect LA at levels below these removal criteria.

4.6.3.4 Summary

In summary, the objective of the TAPE was to identify those parcels in Troy that meet the removal criteria identified in EPA's *Action Level and Clearance Criteria Technical Memorandum for the Libby Asbestos Site Residential/Commercial Cleanup* (EPA 2003) document. This objective was met through (1) visual inspections for vermiculite; (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 above the EPA removal action levels (EPA 2003). A total of 102 properties meeting removal criteria were identified during the TAPE (see Section 7.2 for details).

5.0 OU7 OUTDOOR AMBIENT AIR STUDY

This section describes the objectives, methods, and procedures utilized during the AAS investigation. The information presented in this section is a condensed version of the *Final RI Work Plan, Outdoor Ambient Air Study, for OU7 of the Libby Asbestos Superfund Site* (Tetra Tech 2009a) and references that document as necessary for greater detail. The AAS process was initiated in 2009 and is presently on-going. The AAS is scheduled to continue through October 2010.

5.1 OUTDOOR AMBIENT AIR STUDY OBJECTIVES

As discussed in Section 4.1 above, previous investigations by EPA in Libby determined that LA was present in multiple environmental media. The potential exists for similar LA contamination in the OU7 area due to its proximity and historical mining connection to Libby. The DEQ requested that Tetra Tech formulate a data collection process to assess outdoor ambient air as a potential exposure pathway and prepare the AAS Work Plan.

The primary objective of the AAS is to measure LA concentrations in outdoor ambient air and combine the collected data with discrete data from other exposure routes to support human health risk assessment and the evaluation of future remedial actions. Specifically, as part of the DQOs, the AAS seeks 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 Final AAS Work Plan (Tetra Tech 2009a).

5.2 FIELD INVESTIGATION METHODS

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

5.2.1 Site Access and Logistics

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 (at least a year and perhaps longer), and were informed of the importance of obtaining samples consistently over that extended period. Tetra Tech personnel explained the program and the potential impact to the property (e.g., installation of the housing unit, sample technicians visiting the property 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 office.

The DEQ Troy Information Center continues to provide public information for both the TAPE and AAS investigations.

5.2.2 Pre-Sampling Activities

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

Field Planning Meeting/Equipment Inventory and Procurement

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 require procurement.

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 close 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 Troy to measure LA concentrations in downtown Troy. One station was placed in the Kootenai Vista area in the northern portion of OU7 and the last station was placed along Iron Creek Road in the southwestern portion of OU7 (Figure 5-1).

Outdoor Ambient Air Sample Station Installation

The boxes used for housing the equipment were formerly used during the OU4 outdoor ambient air sampling; each was thoroughly inspected for defects prior to installation. The equipment housing boxes were thoroughly decontaminated in OU4 prior to bringing them to OU7. After decontamination, each station was labeled with the appropriate OU7 AAS station number. Other information recorded included the landowner name for the property on which the station was erected, the DEQ Troy Information Center phone number and address, and a telephone number to call in case of an emergency. Equipment housing boxes were installed at each of the seven locations and were locked to a stationary object (such as a tree or fence post). Tetra Tech personnel locked the stations with padlocks whenever they were not present at the station site.

Tetra Tech used 12-volt deep cycle batteries to power the stations (pumps and heaters). As batteries were the only source of power, an electrical connection, grounding wire, or conduit to the residence or building were not required.

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) was 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 consists 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 AC power from the field office.

Sampling Equipment Setup and Calibration

The pumps used for sampling were SKC, Inc. Model AirCheck2000. The pumps were battery powered so that sampling was not interrupted due to loss of an AC power source. The pumps were calibrated prior to each sampling event to ensure proper flow rate.

5.2.4 QA/QC Samples

Three types of QA/QC samples were collected as part of this investigation and include: lot blanks, field blanks, and co-located samples. All QA/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. 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 (6 samples through December 31, 2009). Field co-located samples were collected from the same location throughout the project (Station Number T4QC). Station T4QC is located approximately seven feet from sampling station T4 at the DEQ Troy Information Center. All co-located samples were handled and sent for analysis using the same TEM method used for field samples.

5.2.5 Field Documentation

Field documentation generated during this sampling program included the following: field sample data sheets (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 5.2.8.

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.

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 other pertinent site features. The photographs were each assigned a unique number and are included in the electronic data archive.

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 are regularly scanned into PDF files and stored in the electronic data archive. The original field logbook is maintained in the DEQ Troy Information Center office.

5.2.6 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, 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 a labeled asbestos waste bag for later disposal.

5.2.7 Containment and Disposal of Investigation-Derived Waste

Investigation-derived waste 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 and replacement and repair of any defective or worn tubing. As it was possible that these investigation-derived waste materials contained asbestos, all investigation-derived waste was double-bagged in appropriate asbestos bags, labeled with asbestos labels, and stored in an approved containment area at the DEQ Troy Information Center field office until it was properly disposed of at the Lincoln County Landfill, outside of Libby. 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.

5.2.8 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 office 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 office. 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.

5.2.9 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 field 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 office 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 OUTDOOR AMBIENT AIR ANALYTICAL METHODS

OU7 outdoor ambient air samples were analyzed in accordance with International Organization for Standardization Method 10312, Ambient Air – Determination of Asbestos Fibers – Direct-transfer Transmission Electron Microscopy Method. A copy of the analytical method is provided in Appendix A.

5.4 OUTDOOR AMBIENT AIR 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) on a weekly basis for submittal to the electronic data archive. Field staff also downloaded digital photographs to a computer at the DEQ Troy Information Center field office. Scanned access agreements, FSDS, 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 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 are able to download a complete database that contains all station, sample, analytical, and custom queries.

After the data is successfully downloaded and the OU7 Outdoor Ambient Air Scribe Database is published to the internet, the air sampling pumps are 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 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 validation process is summarized in Section 5.6.2.

5.5 DEVIATIONS FROM OUTDOOR AMBIENT AIR STUDY WORK PLAN

All minor FSDS entry modifications were made 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 close proximity to the erroneous entry where possible. Errors encountered after the FSDS had been scanned, were corrected using electronic comments attached to the PDF file using Adobe Acrobat, explaining or clarifying the erroneous entry.

Between inception of the AAS investigation and December 31, 2009, one circumstance arose where modification of the procedures described in the AAS Work Plan was necessary to complete project objectives. The Tetra Tech Project Manager requested a modification to the approved procedures to change the sample cassette filter size.

Outdoor Ambient Air TFO-00001 requested a change in sample cassette filter size from 0.45 µm to 0.8 µm to accommodate the high sampling pump air flows of up to 3 liters per minute. Changing to the larger filter opening was intended to prevent overloading of the filter media due to the relatively high volume of air being passed through the filter (21,600 total liters at 3 liters per minute). Changing to the larger filter size was also consistent with filter size employed during the OU4 sampling activities in Libby.

The modification was documented on a TFO Record of Modification form; form TFO-00001 is provided in Appendix C. The TFO form was submitted to the DEQ project manager for review and approval and a signed copy was kept in the DEQ Troy Information Center office.

5.6 OUTDOOR AMBIENT AIR DATA QUALITY ASSESSMENT

The primary objective of data quality assessment is to ascertain the accuracy and usability of the data collected in meeting the DQOs of the project. This objective is achieved in two steps; by verifying the accuracy and completeness of the field data collected, as described in Section 5.6.1, and by verification and validation of the analytical data, as described in Section 5.6.2. It should be noted that AAS analytical data was not available as of December 31, 2009. The field data verification process was performed on AAS field data as it was collected, but the 2009 AAS analytical data will need to be assessed by the process described in 5.6.2 once it is available.

5.6.1 Field Data Verification

Following each round of outdoor ambient air data collection, field data associated with that round were reviewed for completeness, accuracy, and consistency to ensure the quality of the outdoor ambient air samples collected. First, electronic pump history files were reviewed for each station to ensure that pump run times, flow rates, and total volumes were within acceptable criteria. Next, the pump history data were compared to data manually recorded on FSDS sheets to ensure they were consistent. Also, the FSDSs and outdoor ambient air logbook were reviewed to determine if any notable events occurred during the sampling round. Data from the pump history files, FSDS and logbook were then checked against the TAAD database to ensure that data were once again consistent between the data summary locations and that notable events were documented throughout. When discrepancies were noted on the FSDS forms or in the logbook, the field team was contacted and changes to the appropriate files were made with electronic notes on 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 Outdoor Ambient Air Scribe Database was completed after data entry to ensure that the transfer of data from TAAD was complete and that no errors were present.

5.6.2 Analytical Data Review and Verification

Tetra Tech will conduct data review and data entry verification of the outdoor ambient air analytical data in accordance with Libby SOP EPA-Libby-09, Revision 1 (Appendix A). The SOP describes a standardized approach for review of raw TEM data and verification of TEM data entry into the project database. However, some Libby-specific details were modified as appropriate for OU7 (for example, the SOP refers to the Libby2 Database, but for OU7, it is the OU7 Outdoor Ambient Air Scribe Database).

The data review and entry verification process involves three primary steps: (1) the selection of data records for review and verification, (2) review of the original laboratory bench sheets for consistency with the method, and (3) verification of the transfer of results from the bench sheets into the OU7 Outdoor Ambient Air Scribe Database. Each step is described below.

Selection of TEM Records for Review

To ensure that a representative subset of the ambient air sample results were validated, Tetra Tech validated will validate all 54 field samples collected during sampling periods 1 through 6. For subsequent sampling rounds, approximately 25 percent of the field samples will be validated; records reviewed were selected on the basis of (1) result type (detected LA), (2) field duplicate pairs, and (3) field blanks.

Consistency Review of Laboratory Bench Sheets

Tetra Tech will inspect the information recorded on the original hand-written laboratory bench sheets in accordance with EPA-Libby-09 (Revision 1).

Scanned copies of the original hand-written bench sheets will be reviewed to verify the data entered and to identify the occurrence of any data omissions, apparent inconsistencies, or potential errors in structure identification. The bench sheets will be reviewed to verify (1) whether the recorded structure types are consistent with the counting rules, (2) whether disperse complex structures were broken down in accordance with ISO 10312 counting rules and compact complex structures were not broken down, (3) whether the primary and total columns were populated with non-zero numbers for all countable structures and a zero for all non countable structures, (4) whether all recorded non-asbestos mineral structures were identified as non-countable structures, (5) whether all recorded fibers meet the appropriate aspect ratio requirement, (6) whether the mineral class is populated for all structures, (7) whether the mineral type and appropriate spectra code are recorded in the structure comment field for all recorded LA, Chrysotile, OA , and non-asbestos mineral structures, (8) whether the structure comments are supported by recorded data, and (9) whether the stored values in the TAPE Scribe Database for primary, total, structure type, length, width, and mineral class match the original bench sheet.

Verification of Data Transfer from Bench Sheet to Database

Verification of data transfer from bench sheets to the Outdoor Ambient Air Scribe Database will be done to ensure that data from the bench sheets were transferred into the database without error or omission. Tetra Tech will compare the analysis-specific information in the database to that on the bench sheets. This includes verifying consistency of analytical method, analysis date, laboratory name, laboratory job number, laboratory sample number, preparation method, filter status, primary effective filter area, secondary effective filter area, grid opening area, F-factor, air volume, and analysis comments. Tetra Tech will recalculate the effective filter area for all samples verified and the F-factor for all samples that underwent indirect preparation, recount the total number of grid openings evaluated on the hand-written bench sheets and will compare that to the "Analysis Grid Opening Counted" entry in the database, and recount the total number of countable LA structures and compare it to the binned LA values in the EDD.

Corrective Action

Tetra Tech will summarize any apparent inconsistencies, omissions, and other suspected errors and provide them in spreadsheet format to the laboratory liaison, who will relay relevant issues to the contract laboratories. The contract laboratories will address the issues and the revised EDD and/or bench sheet will be submitted to the laboratory liaison, who will forward them to Tetra Tech for final review and archiving.

5.6.3 Reconciliation with Data Quality Objectives

DQOs are developed to help clarify study objectives, define the most appropriate data to collect and the conditions under which to collect it, specify tolerable limits on decision errors, and establish required analytical sensitivities. This information is used to determine the quantity and quality of data needed to support project-related decision making, and, later on, to assess whether the data collected are of adequate quality and quantity to satisfy project needs.

The objectives of the AAS were to collect the data necessary 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 risk range 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. The DQOs developed for the AAS are presented in the AAS work plan (Tetra Tech 2009a).

5.6.3.1 Sampling Design

The data needed to answer the above questions must be reliable and representative (over location and time) of the long-term average concentration of LA in outdoor ambient air within OU7. Since there are several alternative strategies for specifying the concentration of LA in air, as well as several alternative strategies for using those data to estimate human health exposure and risk, and final decisions have not been made regarding which approaches will be used, the analytical data obtained must be as comprehensive and representative as possible, so that they can be used to compute the appropriate concentration values for use in whatever alternative risk models are selected for the site (Tetra Tech 2009a).

Tetra Tech followed procedures in the sampling and analysis plan for outdoor ambient air monitoring at OU4 (EPA 2006a) to calculate an exposure point concentration for LA in outdoor ambient air. The approach used for determining sample minimum size requirements necessary for meeting the exposure point concentration is described in Appendix A of the AAS Work Plan (Tetra Tech 2009a).

In order to assure a minimum of 20 to 25 detected results, a target sample size in the range of at least 150 to 200 discrete samples was planned. A total of 65 outdoor ambient air samples had been collected as of December 31, 2009.

5.6.3.2 Limits on Decision Errors

The EPA is most concerned about guarding against the occurrence of Type I errors, because an error of this type may leave humans exposed to unacceptable levels of LA in outdoor ambient air. For this reason, it is anticipated that the exposure assessment for this pathway will be based on the best estimate and the 95 percent upper confidence limit (UCL) of the long-term average concentration of LA in the area being evaluated. Use of the UCL to estimate exposure and risk helps account for limitations in the data, and provides a margin of safety in the risk calculations, ensuring that risk estimates are unlikely to be too low.

EPA is also concerned with the probability of making Type II (false positive) decision errors. Although this type of decision error does not result in unacceptable human exposure, it may result in unnecessary expenditure of resources. For the purposes of this effort, the strategy adopted for controlling Type II errors was to ensure that, if the risk estimate based on the 95 percent UCL is above EPA's level of concern for this pathway, then the UCL is not larger than 3-times the best estimate of the mean. If the 95 percent UCL is at or above the range that is of potential concern, and the UCL is greater than 3 times the best estimate of the mean, then more data may be needed.

5.6.3.3 Analytical Sensitivity

The analytical sensitivity must be sufficient to ensure reliable detection and quantification of risks from LA. For the AAS specifically, the analytical sensitivity must detect an excess of cancer risk of 1E-05 (1 in 100,000) or a non-cancer hazard quotient of 0.1 in outdoor ambient air.

For cancer, a simplified equation for computing the risk associated with some specified concentration is:

Risk = C x TWF x UR

Where:

Risk =	risk of lung cancer or mesothelioma from the exposure being evaluated
C =	long-term average concentration of asbestos (fibers per cubic centimeter [f/cc])
TWF =	time weighting factor (percent of full time that exposure occurs)
UR =	unit risk for lifetime exposure

The target analytical sensitivity is then computed by rearranging the equation as follows:

Target Analytical Sensitivity $\leq$ 1E-05 / (TWF x UR)

For planning purposes, Tetra Tech made the conservative assumption that the time weighing factor equals 1.0 (this value was used in the outdoor ambient air analysis conducted in OU4 (EPA 2006a). This corresponds to an outdoor ambient air exposure of 24 hrs/day for a lifetime (actual exposures are expected to be lower than this for most people). Based on EPA's currently recommended risk model (EPA 2006b), the unit risk factor for lifetime exposure is 0.23. Thus, the level of concern for LA in air would be about:

Target Analytical Sensitivity $\leq$ 1E-05 / 0.23 = 0.00004 f/cc

For non-cancer effects, the basic risk equation is:

HQ = C x (ET/24 x EF/365 x ED) / RfC

Where:

HQ = hazard quotient (dimensionless)
C = long-term average concentration of asbestos in air (f/cc)
ET = exposure time (hrs/day)
EF = exposure frequency (days/yr)
ED = exposure duration (yrs)
RfC = Cumulative reference concentration (f/cc-yrs)

To date, no cumulative reference concentration (RfC) has been established for evaluating non-cancer effects from inhalation of LA, so one cannot compute an analogous level of concern for this endpoint. In the absence of such data, the target analytical sensitivity that is adequate for evaluating cancer risk is assumed to also be sufficient for evaluating non-cancer risks. This assumption will be re-visited when an RfC is developed. Thus, the target analytical sensitivity for outdoor ambient air samples was $\leq$0.00004 f/cc. This analytical sensitivity was achieved for the AAS sample analysis.

5.6.3.4 Summary

In summary, the objective of the AAS was to collect the data necessary 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 risk range 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 total of 54 outdoor ambient air samples had been collected as of December 31, 2009. The samples were analyzed in accordance with ISO 10312 (modified per applicable Libby laboratory modifications). At the time this draft final RI was prepared, AAS analytical data was not yet available, and as a result, had not been validated or reviewed by project chemists. Results for all 54 samples will be reviewed and verified in accordance with SOP EPA-LIBBY- 09, Revision 1 (Appendix A) and the process outlined in Section 5.6.2 to determine if the data are of adequate quality and quantity to be use in a risk assessment. The AAS sampling program is expected to continue at least through 2010.

6.0 ADDITIONAL REMOVAL ACTIVITIES

The following sections discuss removal activities associated with the identification of visible vermiculite and/or LA source materials along Callahan Creek, on a portion of the BNSF ROW, and at OU7 parcels with considerable vermiculite contamination. This draft final RI covers investigation and removal activities within OU7 from April 2007 to December 31, 2009. Any previous investigations or removal actions occurring in OU7 and vicinity prior to 2007 are captured in pre-existing reports.

6.1 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).

6.2 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 personnel observed a stockpile and concrete foundation on the BNSF ROW containing visible vermiculite. Although the BNSF ROW is part of OU6, the presence of vermiculite was deemed critical due to the amount of contamination and evidence of frequent visitation to the site. Tetra Tech contacted DEQ and EPA regarding the contamination. After an inspection with Tetra Tech and DEQ, 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 on two separate days (June 6 and 12, 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. A copy of the report can be found in the electronic data archive.

6.3 RESIDENTIAL REMOVAL ACTIONS

On occasion, Tetra Tech would conduct a TAPE inspection on a property with a considerable amount of vermiculite. It was determined that these properties required immediate attention to reduce exposure from material continually leaking into the living space and/or from exposure to heavily contaminated soil. When such a property was identified, the field team would alert the Tetra Tech Field Team Leader to the situation. Once the Field Team Leader confirmed that the building was in need of attention, the EPA was contacted so that a removal action of the vermiculite could occur under the contract for Libby removal actions, as removals were not yet being conducted in Troy.

In 2008 and 2009, thirteen individual properties underwent an interior removal action, an exterior removal action, or a combination of both. The removal actions were performed by either Department of Transportation John A Volpe Center or the United States Army Corps of Engineers.

7.0 FIELD RESULTS AND NATURE AND EXTENT OF CONTAMINATION

This section summarizes the TAPE and AAS field activities and the data obtained from these investigations, from their respective project inception dates through December 31, 2009. The TAPE Work Plan was implemented in April 2007 and the AAS Work Plan was implemented in September 2009; both investigations are currently on-going. The intent of these investigations is to gather sufficient and reliable data such that the nature and extent of LA contamination in OU7 can be evaluated and the associated risks to human health assessed. In addition, TAPE data has been used to identify parcels meeting the EPA removal action levels (EPA 2003). Data obtained from the AAS investigation will primarily be used to determine whether levels of LA in outdoor ambient air contribute a risk of cancer or non-cancer effects. The TAPE and AAS data will eventually be used in support of a site-wide risk assessment and determination of remedial alternatives.

The historical connection between OU4 and OU7 with regards to mining and transport of LA-contaminated vermiculite make the EPA removal action levels appropriate for OU7 as well as OU4. The EPA removal action levels are presented in Section 3.5 above. Section 7.1 provides field results associated with the TAPE investigation and defines the nature and extent of LA contamination within OU7 as it relates to EPA removal criteria and the DEQ selection process for removal action. Section 7.2 summarizes the analytical results from the TAPE, compares TAPE results to EPA action levels, and discusses the selection of OU7 parcels for removal. Section 7.3 presents the sampling activities, data acquisition, and analytical data from the AAS investigation.

7.1 TROY ASBESTOS PROPERTY EVALUATION RESULTS

TAPE investigation results and associated activities are presented in the following sections organized by year. Between April 1, 2007 and December 31, 2009, a total of 1,239 parcels were inspected, including all parcels within the OU7 boundary shown on Figure 7-1, for which property owners' granted permission to inspect. Additionally, in response to requests by individual property owners, 28 properties located outside the OU7 boundary were also inspected under the TAPE. These inspections 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 if the owner had knowledge of such an event. For the purposes of this RI, these parcels will be considered as part of OU7 and are included in the total inspection count of 1,239 parcels as of December 31, 2009. Also, of the 1,239 parcels inspected, 302 are

road and alley parcels. Road and alley parcels were inspected during the 2009 field season; these results will be discussed exclusively and are not included in general soil counts provided in the following sections. Between April 1, 2007 and December 31, 2009, Tetra Tech performed 28 ERS actions and CDM performed two ERS actions. ERS actions are discussed below according to the year in which they were conducted. The total number of parcels in the TAPE geodatabase as of December 31, 2009 was 1,578, including 31 parcels outside of the OU7 boundary and 316 road and alley parcels.

As discussed in Section 4.5.3, the TAPE Scribe Database is used to store, manage, and retrieve TAPE data. The TAPE Scribe Database is dynamic; data have been corrected or updated over the course of the project as a result of modifications to TAPE sampling objectives, additional information gained from parcel revisits (i.e. visible vermiculite re-inspections, aggressive attic entry), and as part of the TAPE data verification process (Section 4.6.1). In order to present TAPE data by year, database queries were developed using the initial inspection date for each parcel. This eliminated the possibility of counting a parcel or data record twice. The data presented in the sections below have been extracted from the TAPE Scribe Database. Appendix D presents a summary of the TAPE investigation results.

7.1.1 2007 Sampling

Results of the 2007 TAPE investigation are grouped into five categories: site access, dust sampling, attic and interior inspection, soil sampling, and ERS activities and are presented in the following sections.

7.1.1.1 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 (Section 4.2.1 and 4.5.1). At the time of the 2007 access agreement mailing, there were 1,092 mapped parcels within 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 the owners for each of these parcels a cover letter and access agreement. 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 mailed access agreements in 2007, 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 inspections only, or secondary building inspections 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).

It should be noted that the status of parcels (i.e. granted, limited, denied) is not static and fluctuates as parcel ownership changes. New owners may reverse a previous owner's limitations on access or revoke previously granted access. A review of the parcel status for all OU7 parcels as of December 31, 2009 can be found in Section 7.1.3.1.

7.1.1.2 Dust Sampling

The Tetra Tech project team attended a week-long intensive training on all aspects of the TAPE in May 2007. As part of the training, field teams performed test TAPE inspections at a few parcels to facilitate understanding of TAPE sampling equipment, protocols, and data collection. The original dust sampling protocol outlined in the TAPE Work Plan followed the CDM-Libby-10 SOP (Appendix A), which specified 30 dust aliquots for each dust sample collected. Through the test parcel sampling during training week, it was determined that 30 aliquots caused cassette overloading and pump failure. A Record of Modification was initiated to address this issue. TFO-00001 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).

In July 2007, TFO-00003 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 were outlined in the SOP *Libby Asbestos Project Draft Pilot Study30-point Dust Composite Sampling* document (CDM 2007b) (Appendix A).

In total, 536 individual parcels underwent an initial TAPE inspection in 2007. The inspections covered 508 primary and 730 secondary buildings. Dust samples were obtained from both primary buildings and secondary buildings without dirt floors. Tetra Tech field teams collected 1,211 dust samples, excluding dust field duplicate samples (42) and dust field blank samples (195). Six hundred and seventy-eight dust samples were collected from primary buildings and 533 dust samples were collected from secondary buildings. Of the total dust field blanks, 159 were obtained from primary buildings and 36 were collected in secondary buildings. Likewise, 24 dust field duplicates came from primary buildings and 18 came from secondary buildings.

In August 2007, TFO-00005 was instituted that requested dust samples only be collected from parcels with a 'dust trigger', i.e., any parcel with observed visible vermiculite in interior or exterior areas, or if the resident was a former miner or had an asbestos-related disease. 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. A copy of the TFOs can be found in Appendix B.

7.1.1.3 Attic and Interior Inspection

As part of the TAPE inspection process, the living areas and attics of primary and secondary buildings were inspected for the presence of visible vermiculite or vermiculite insulation. The attic visual inspection was not completed in buildings without attics or if the attic could not be accessed.

As stated above, 508 primary buildings and 730 secondary buildings were initially 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 of the parcels initially inspected in 2007 had vermiculite insulation in a building attic. Field teams identified 28 properties that had visible vermiculite in an indoor living space.

7.1.1.4 Soil Sampling

The TAPE inspection soil sampling protocol was modified at the on-set of TAPE activities on May 8, 2007 through TFO-00002 to set the maximum number of visual point inspections for an individual use area at 30. The modification required visual point inspections be evenly distributed throughout the use area and corresponded to the sample aliquot locations (Appendix B). This modification altered the protocol outlined in the CDM-Libby-06 SOP, which specified that visual point inspections be performed at a minimum of 1 point inspection per 100 square feet for each use areas 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 use area was deemed sufficient for detecting LA, if it was present. Follow up investigations would identify the exact extent of the visible vermiculite prior to any removal action.

Tetra Tech identified and visually inspected the following use areas 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 visible vermiculite. Although not all vermiculite contains asbestos, historical sampling and analytical results from OU4 showed a strong correlation between visible vermiculite in SUAs and LA contamination. Given the similarities between OU4 and OU7, it was believed that visible vermiculite in SUAs in OU7 would contain LA and that it would be cost effective to forego the collection of a soil sample from SUAs with visible vermiculite, as visible vermiculite is a removal action criteria. Tetra Tech field staff did perform a 5-point minimum, 30-point maximum, visual point inspection for SUAs with visible vermiculite and classified the presence of visible vermiculite as none, low, intermediate, or high (DEQ 2009a).

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

Tetra Tech also visually inspected and sampled interior crawlspace areas (if safe and accessible). A total of 316 interior 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 visible vermiculite seen 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 was purchased from a store recently would not contain LA. In response to this evaluation, TFO-00007 was instituted on November 15, 2007 and requested that field crews sample all SUAs with visible vermiculite. This modification went into effect in the 2008 field season.

7.1.1.5 Environmental Resource Specialist Activities

ERS 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 actions was to temporarily isolate potential LA-containing material from residents or workers until removal activities could be conducted. ERS 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 high efficiency particulate air vacuums, removing bags of Zonolite (i.e., vermiculite attic insulation) from attics or storage areas, and providing advice during remodeling activities.

Tetra Tech and CDM conducted ERS activities at 10 parcels in 2007, including one property outside of the OU7 boundary. Each ERS action is documented in an ERS report. ERS reports were prepared by the Tetra Tech or CDM Field Team Leader. Each report was scanned into a PDF file and added to the electronic data archive folder for the property where the ERS response occurred. A note indicating the occurrence of the ERS activity was also added to the TAPE Scribe Database.

7.1.2 2008 Sampling

Results of the 2008 TAPE investigation are grouped into five categories: site access, dust sampling, attic and interior inspection, visible vermiculite re-inspections and soil sampling, and ERS activities. Each topic is presented in the following sections.

7.1.2.1 Site Access

Tetra Tech initiated the 2008 TAPE field investigation process by mailing access agreements to 538 property owners within OU7. This number included property owners that received an access agreement in 2007, but did not respond, and new property owners. Property owners who returned a signed access agreement in 2007, but did not get scheduled for a TAPE inspection in 2007, were contacted by the CIC at the beginning of the 2008 field season for scheduling.

Of the parcel owners mailed an access agreement 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. The reasons for limited access in 2008 were similar to those in 2007.

7.1.2.2 Dust Sampling

Tetra Tech continued to collect dust samples in primary and secondary buildings (without dirt floors) in 2008. There were 261 parcels that were initially inspected as part of the TAPE in 2008. The inspections covered 226 primary buildings and 349 secondary buildings. The field teams collected 573 dust samples, excluding 21 dust field duplicate samples and 97 dust field blank samples. Of the total dust samples obtained, 313 were collected from primary buildings and 260 were collected from secondary buildings. Eighty-two dust field blanks and 13 field duplicates came from primary buildings. Fifteen dust field blanks and 8 field duplicate samples came from secondary buildings.

In December 2008, following review of TAPE data for 2007 and 2008, modification TFO-00012 was prepared, specifying the cessation of dust sampling as part of the TAPE investigation process. The comparison of inspection results for visible vermiculite in interior living areas to dust analytical results showed that none of the parcels with visible vermiculite in interior living areas had dust results above the 5,000 s/cm^2 removal action level. The DEQ concluded the dust analytical results did not contribute meaningful information from which removal action decisions could be made (i.e. the presence of visible vermiculite signifies interior removal despite dust analytical results). For this reason, TFO-00012 was approved on December 22, 2008 and was implemented at the start of the 2009 field season. A copy of TFO-00012 can be found in Appendix B.

7.1.2.3 Attic and Interior Inspection

The visual inspection of interior living areas and attic spaces (when accessible) continued in 2008. As stated above, 226 primary and 349 secondary buildings underwent a TAPE inspection in 2008. Tetra Tech recorded that 82 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 initially inspected in 2008 had vermiculite insulation in a building attic. Field teams identified 20 properties that had visible vermiculite in an indoor living space.

7.1.2.4 Visible Vermiculite Re-Inspections and Soil Sampling

Visible Vermiculite Re-Inspection

At the beginning of the 2008 field season, Tetra Tech was requested to revisit all UAs identified in 2007 to confirm, semi-quantify, and describe visible vermiculite in exterior soils. The reason for the re-inspection and additional sampling was to address 1) poor correlation between soil analytical results and the visible vermiculite observations recorded at UAs , 2) the need for soil samples from all SUAs as specified in TFO-00007, and 3) an accurate description of visible vermiculite that was reported as 'store purchased potting soil' to confirm the relationship between vermiculite observed in 'potting soil' and analytical results from OU7 that may differ from that relationship as observed in OU4 through documentation of such descriptions during re-inspection and additional sampling (Tetra Tech 2008b). TFO-00008 specified that Tetra Tech return to 462 parcels to perform visible vermiculite re-inspections on 1,743 UAs. TFO-00008 also required Tetra Tech to return to each SUA inspected in 2007 where visible vermiculite was identified and collect a soil sample, which was calculated as 115 parcels affecting 144 SUAs. Detailed information on how the parcels and UA were chosen, the sampling methodology, and the data management for visible vermiculite re-inspections is included with TFO-00008 in Appendix B.

To ensure accurate identification and quantification of visible vermiculite, nine Tetra Tech field personnel were trained by experienced CDM field persons for approximately four days on the recognition of visible vermiculite in exterior soils. Subsequently, Tetra Tech placed at least one of the additionally trained individuals on the TAPE inspection teams during 2008 to accurately identify visible vermiculite.

Between May 20 and July 24, 2008, Tetra Tech re-inspected the UAs on 101 parcels for the identification and possible quantification of visible vermiculite in exterior soils. A total of 527 UAs were re-inspected; the type and number of UA 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 visible vermiculite re-inspection process. The first audit was completed in July 2008 on 8 parcels for which Tetra Tech had previously conducted visible vermiculite re-inspection activities. CDM independently assessed visible vermiculite on each of these 8 parcels and prepared a technical memorandum detailing the findings (CDM 2008b). The identification and quantification of visible vermiculite by both parties was similar, 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 visible vermiculite 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 visible vermiculite, which were attributable to several factors (CDM 2008b); however, the results of the audits demonstrated that visible vermiculite was being adequately identified during the TAPE investigations. As a result, Tetra Tech was not required to pursue visible vermiculite re-inspections at any additional properties beyond the first one hundred.

The second objective of TFO-00008 (to sample all 2007 SUAs with visible vermiculite) 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).

On December 22, 2008, DEQ and EPA approved TFO-00010, ceasing the visible vermiculite re-inspection requirement set forth in TFO-00008. Copies of both TFOs are included in Appendix B.

Soil Sampling

As part of regular TAPE inspection activities in 2008, Tetra Tech identified and visually inspected the following use areas:

- Common Use Areas = 257
- Limited Use Areas = 83
- Specific Use Areas = 655
- Non-Use Areas = 103

A total of 1,000 soil samples were collected in 2008 from the use areas listed above, not including 44 field duplicate and 152 field split samples (split samples are discussed below).

Tetra Tech also visually inspected and sampled interior crawlspace areas (if safe and accessible). A total of 95 interior soil samples were collected in 2008. In addition, 2 field soil duplicate samples and 12 field soil split samples were collected from interior crawlspaces.

On June 3, 2008, DEQ approved TAPE modification TFO-00009, requesting that Tetra Tech field crews collect split soil samples from UA 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 through TFO-00011 once the laboratories determined that Tetra Tech could provide additional soil volume in one bag and perform the split once the sample arrived at the laboratory. Modification TFO-00011 eliminated the collection of field split samples and required an increase in soil volume by 50% (Appendix B).

7.1.2.5 Environmental Resource Specialist Activities

Tetra Tech conducted ERS activities at 12 parcels in 2008, including four properties outside of 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 ERS actions in 2008 were similar to those in 2007 and involved temporarily isolating potential LA-containing material from residents or workers. CDM did not perform any ERS activities in OU7 in 2008. A copy of each ERS report was scanned into a PDF file and added to the appropriate electronic data archive folder.

7.1.3 2009 Sampling

Results of the 2009 TAPE investigation are grouped into six categories: site access, attic and interior inspection, soil sampling, road and alley sampling, aggressive attic entry and attic revisits, and ERS activities. Each topic is presented in the following sections.

7.1.3.1 Site Access

Tetra Tech initiated the 2009 TAPE field investigation process by mailing access agreements to 313 property owners within OU7. This number included owners that were mailed an access agreement in 2007 or 2008, but did not respond, and 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 the CIC at the beginning of the 2009 field season for scheduling.

Of the 313 parcel owners that were mailed an access agreement 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. The reasons for limited access in 2009 were similar to those in previous years.

As mentioned in Section 7.1.1.1, the access status (i.e. granted, limited, denied) fluctuates as property ownership changes. In addition to the access agreements received from yearly access mailings, access agreements have also been obtained from walk-ins to the DEQ Troy Information Office and from door-to-door solicitations of owners whose addresses were listed incorrectly (or were unknown) in the CAMA database. TOAD tracks each of the changes to parcel status. As of December 31, 2009, the parcel status for all parcels (those that were part of the access agreement mailing and those obtained by other means) was the following:

Granted = 941
Limited = 15
Denied = 34

7.1.3.2 Attic and Interior Inspection

The visual inspection of interior living areas and attic spaces (when accessible) continued as part of the TAPE 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 vermiculite insulation in a building attic. Field teams also identified 5 properties with visible vermiculite in an indoor living space.

7.1.3.3 Aggressive Attic Entry and Attic Revisits

To address the possibility that vermiculite insulation was not identified in attics with limited access, or in buildings where the inspector's view of the entire attic was obstructed from the point of access, a modification was made to the TAPE Work Plan allowing for more aggressive attic entry procedures. TFO-00013 was approved on May 27, 2009, stating that aggressive attic inspections would be conducted to adequately identify vermiculite insulation, and that these procedures would be implemented in future TAPE inspections and re-visits when possible. Tetra Tech prepared a SOP entitled *TAPE Aggressive Attic Inspection Activities to Verify Presence or Absence of VCI*, which was submitted along with TFO-00013 (Appendix B).

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 vermiculite insulation in the Attic?". If the structure was built after 1980 (greater than or equal to), then it was removed from the list of potential attics to revisit, as the construction year made it unlikely that the attic would contain vermiculite insulation. 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. The Tetra Tech Field Team Leader identified eight buildings where an aggressive attic entry revisit may be warranted.

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 vermiculite insulation, despite the inability to access the attic.

7.1.3.4 Soil Sampling

As part of the TAPE inspection activities in 2009, Tetra Tech identified and visually inspected the following use areas:

- 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 use areas listed above.

Tetra Tech also visually inspected and sampled interior crawlspace areas (if safe and accessible). A total of 75 interior soil samples were collected in 2009. In addition, 2 field soil duplicate samples were collected from interior crawlspaces.

7.1.3.5 Road and Alley Sampling

The TAPE was modified on June 17, 2009 with TFO-00014 to require TAPE inspections on road and alley parcels in OU7 during the 2009 field season. Tetra Tech prepared the *Protocol Modification to the TAPE Project Work Plan, Inspection and Sampling of Roads and Alleys for visible vermiculite* in support of TFO-00014. This protocol modification outlined the procedures and techniques that would 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. Copies of TFO-00014 and Tetra Tech's protocol modification for road and alley inspections are located in Appendix B.

Tetra Tech inspected and sampled (as appropriate) all accessible road and alley parcels within OU7 during 2009. In total, 302 road and alley parcels were inspected for visible vermiculite and 271 soil samples were collected. In addition, 8 field duplicate samples were collected as part of this effort. Occasionally the field team would attempt to complete an inspection on a road and alley parcel, but were unable to gain access due to a private gate or refusal by the property owner. These attempts account for 14 of the 316 road and alley parcels.

Out of 302 inspected road and alley parcels, visible vermiculite was identified on 20 parcels. 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 road and alley parcels were found to be adjacent to residential parcels contaminated with vermiculite and eligible for removal action. The visible vermiculite detected 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 discussion of road and alley soil analytical results is presented in Section 7.2.1.

7.1.3.6 Environmental Resource Specialist Activities

Tetra Tech and CDM conducted ERS activities at 8 parcels in 2009, including four properties outside of the OU7 boundary. The ERS actions in 2009 were similar to those from previous years and involved temporarily isolating potential LA-containing material from residents or workers. Copies of the ERS reports were scanned and placed in the individual electronic archive folders for the pertinent properties. A note indicating that an ERS response had been conducted at the property was entered into the TAPE Scribe database.

7.2 LIBBY AMPHIBOLE ASBESTOS CONTAMINATION IN OU7 AND THE SELECTION OF PARCELS FOR REMOVAL ACTION

This section presents TAPE analytical results and reviews the results with reference to the EPA and DEQ documents guiding removal actions in OU7; specifically, the EPA *Libby Asbestos Site Residential/Commercial Cleanup Action Level and Clearance Criteria Technical Memorandum* (EPA 2003), the DEQ *Libby Asbestos Site Troy OU7 Residential/Commercial Cleanup Criteria Specific Use Area visible vermiculite Action Level Technical Memorandum* (DEQ 2009a), and the DEQ *Libby Asbestos Site Troy OU7 Removal Parcel Status* (DEQ 2009b) memorandum.

7.2.1 TAPE Analytical Results

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

Dust

Indoor dust sampling was conducted during 2007 and 2008 as part of the TAPE inspection process. Table 7-1 below summarizes the analytical results for all field dust samples collected. It does not include results for QA/QC samples (field blanks or field duplicates).

In total, 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 with a LA concentration greater than 5,000 s/cm^2 (EPA 2003). One hundred and eighty-five samples had detected LA concentrations below 5,000 s/cm^2. LA was not detected in 1,022 indoor dust samples from 2007.

A total of 573 indoor dust samples were collected during TAPE inspections in 2008. Of these, only 2 samples exceeded the EPA removal action level for indoor dust with a LA concentration greater than 5,000 s/cm^2. Ninety-seven samples had a detectable level of LA less than the EPA indoor dust removal action level and 474 samples did not have any detected LA.

TABLE 7-1
TAPE DUST ANALYTICAL RESULTS

Analytical Value	2007	2008
> 5,000 s/cm^2	4	2
< 5,000 s/cm^2	185	97
Non-Detect	1,022	474
Total Field Dust Samples	**1,211**	**573**

Notes:
> = greater than
< = less than
s/cm^2 = structures per square centimeter

Use Area and Interior Soil

Exterior soil samples have been collected from UAs at all TAPE inspected properties since the project inception in 2007. In addition, Tetra Tech field crews collect indoor soil samples from safely accessible crawlspaces and dirt floors in primary and secondary buildings. Soil analytical results for all field soil samples, excluding road and alley samples, for April 1, 2007 through December 31, 2009, are presented in Table 7-2 below by year and in accordance with bin categories that outline the percentage of LA

detected in a given soil sample. The table does not include results for QA/QC samples (equipment blanks or field duplicates).

The bin categories for LA concentration in soil are:

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

It should be noted that no Bin C results have been detected for any of the exterior or interior soil samples collected as part of the TAPE.

A total of 1,924 exterior and interior soil samples were collected in 2007. There were 1,274 samples with a Bin A designation (non-detect). A trace amount of LA, less than 0.2%, was identified in 637 soil samples (Bin B1). Only 13 soil samples had LA concentrations between 0.2% and 1% (Bin B2).

Of the 1,226 soil samples collected in 2008, LA was not detected in 894 samples (Bin A). Three hundred twenty-seven samples were placed in Bin B1 for having a detectable level of LA of less than 0.2%. There were only 5 soil samples with Bin B2 results in 2008.

There were 656 soil samples collected in 2009, excluding road and alley samples. Of these, 426 samples received a Bin A designation due to no detectable LA. Two hundred twenty-eight samples had a trace amount of LA (less than 0.2%) and two samples had a LA concentration between 0.2 and 1%.

TABLE 7-2
TAPE SOIL ANALYTICAL RESULTS

2007		**2008**		**2009**	
Bin Result	**Number of Samples**	**Bin Result**	**Number of Samples**	**Bin Result**	**Number of Samples**
Bin A	1,274	Bin A	894	Bin A	426
Bin B1	637	Bin B1	327	Bin B1	228
Bin B2	13	Bin B2	5	Bin B2	2
Bin C	0	Bin C	0	Bin C	0
Total	**1,924**		**1,226**		**656**

Notes:
Results include interior soil samples.

Road and Alley Soil

The analytical results for soil samples collected from road and alley parcels in 2009 are presented in Table 7-3 below. This table does not include results from QA/QC samples such as equipment blanks or duplicates.

Of the 271 total soil samples collected from road and alley parcels in 2009, 197 were placed in Bin A, as LA was not detected in any of these samples. Seventy-four samples had a LA concentration of less than 0.2%. There were no Bin B2 (LA concentration between 0.2 and 1%) or Bin C (concentration of LA greater than or equal to 1%) results for soils collected from road and alley parcels.

TABLE 7-3
TAPE ROAD AND ALLEY SOIL ANALYTICAL RESULTS

	2009
Bin Result	**Road and Alley Parcels**
Bin A	197
Bin B1	74
Bin B2	0
Bin C	0
Total	**271**

7.2.2 Comparison of TAPE Results to EPA Removal Action Levels

As discussed in Section 3.4, the EPA has established removal action levels that allow investigators to determine whether a removal action is warranted (EPA 2003) and has been utilizing these action levels in OU4 (Libby) since 2002. Removal is 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).

A summary of TAPE investigation data between April 1, 2007 and December 31, 2009 meeting the EPA removal action levels is presented below:

OU7 TAPE Attic/Walls

- Total number of parcels with visual confirmation of vermiculite insulation = **74**

OU7 TAPE Interiors

- Total number of parcels with visual confirmation of vermiculite (visible vermiculite) in an indoor living space = **38**
- Total number of indoor dust samples collected with a LA concentration greater than 5,000 (s/cm^2) = **6**

OU7 TAPE Soils

- Total number of SUAs with visual confirmation of vermiculite (visible vermiculite) = **278**
- Total number of exterior soil samples with an analytical LA concentration greater than or equal to 1% = **0**

7.2.3 DEQ Evaluation of Visible Vermiculite in Specific Use Area Criterion

As discussed in Section 7.1.1.4, 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 visible vermiculite in soil and the presence of LA fibers in soil samples had been observed in OU4, and therefore, DEQ did not request that soil samples be obtained from SUAs containing visible vermiculite in 2007. It was noted at the end of the 2007 field season 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) to include soil sampling from SUAs with visible vermiculite starting in 2008.

DEQ performed an evaluation of the soil samples collected from SUAs with visible vermiculite in 2008 and found that of the 392 soil samples collected from SUAs with visible vermiculite, only 3 samples had LA fibers detected above 0.2% (Bin B2). There were no Bin C results for SUAs with visible vermiculite in OU7 (or for any OU7 soil sample). From this evaluation, DEQ recommended that field crews continue to collect soil samples from all OU7 UAs, that the semi-quantification of visible vermiculite in all OU7 UAs

be maintained, 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 visible vermiculite in a specific use area, be considered for removal decisions. Those are discussed in the next section.

7.2.4 OU7 Parcel Selection for Removal Actions

Two of the primary objectives of the TAPE investigations are to identify parcels that meet EPA removal action levels and to provide reliable data on LA contamination in support of OU7 RDI and removal activities. The goal is to address those parcels with LA contamination at levels that present a sufficient health risk and minimize that risk by completing a removal action.

In November 2009, the DEQ asked Tetra Tech to query the TAPE Scribe Database to determine which inspected parcels in OU7 met one or more of the following EPA removal action levels (DEQ 2009b):

- Visible Vermiculite in a Living Space = Yes
- Visible Vermiculite in an Attic = Yes
- Visible Vermiculite in a 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

The DEQ also requested that Tetra Tech query the TAPE Scribe Database for positive answers to the following interview questions asked of residents during TAPE inspections, with the thought that the information from these answers could be used as additional lines of evidence for the identification of parcels needing soil removed:

- Any knowledge of former miners, close relatives of miners, or any highly exposed persons living or visiting the building = Yes
- Is the resident, past or present, 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 there Libby vermiculite additives in any of the building materials = Yes

The query returned 379 parcels meeting the above conditions (DEQ 2009b). The DEQ Project Officer reviewed each parcel individually for all possible lines of evidence (as recommended in DEQ 2009a) that a removal action was warranted. 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 TAPE Scribe Database. Based on the parcel review, the DEQ identified parcels eligible for interior removal only (71), exterior removal only (16), and both interior and exterior removal actions (15), for a total of 102 parcels eligible for removal actions. DEQ then provided the EPA with the list of parcels to plan for the 2010 OU7 RDI and removal activities. Figure 7-2 spatially depicts the parcels identified for removal action in OU7.

Table 7-4 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 proposed OU7 lines of evidence (DEQ 2009a, 2009b). The number of parcels presented in Table 7-4 will not directly sum to the number of OU7 parcels identified for removal (102) due to several parcels satisfying more than one EPA removal action level (i.e. parcel has both vermiculite insulation in the attic and a dust sample greater than 5,000 s/cm^2).

TABLE 7-4
SUMMARY OF OU7 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	74	74
		Visual confirmation of vermiculite in living area	38	38
		Concentration of LA in an indoor dust sample > 5,000 s/cm^2	6	6
		Concentration of LA in a soil sample ≥ 1% LA (Bin C)	0	0
		Visual confirmation of vermiculite in SUA	278	31

Notes:
Numbers reflect data through December 31, 2009

LA	Libby Amphibole asbestos	>	greater than
≥	greater than or equal to	s/cm^2	structures per square centimeter
SUA	Specific Use Area		

A RDI will be conducted at each property before removal activities are initiated. The primary objective of the RDI is 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 are designed to fill remaining data gaps at these properties by further delineating the extent of LA. RDI and removal activities will commence in spring 2010.

7.3 OUTDOOR AMBIENT AIR STUDY RESULTS

This section describes AAS sampling and data acquisition activities for the period October 30, 2009 through December 23, 2009. Section 7.3.1 summarizes sample collection and data recording procedures and schedule. Section 7.3.2 summarizes outdoor ambient air analytical results for samples collected through December 23, 2009.

7.3.1 Sample Collection and Data Recording Procedures and Schedule

Outdoor ambient air monitoring began with Period 1, on October 30, 2009, and continued through Period 6, which ended on December 23, 2009. Periods 1-6 represent two-thirds of the first quarter of AAS sampling. The first quarter consists of nine similar 5-day sampling periods, separated by five non-sampling days between each sample period. Table 7-5 provides a summary of the first six sample periods.

TABLE 7-5
OU7 OUTDOOR AMBIENT AIR SAMPLE DATES
FOR SAMPLING PERIODS 1 THROUGH 6

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

The first sampling period, October 30, 2009 to November 3, 2009, was a full-length test run. The pumps ran successfully during this period, so the data were considered valid and similar methods were employed for subsequent sampling periods. The pumps were set to run at 3 liters of air per minute for 5 days, for a total of 21,600 liters of air. Tetra Tech monitored the 12-volt batteries daily and recorded minimal voltage loss during this first period. Only one sample, TA-0006, collected at station T-2, the

Kootenai Fire Station (Figure 5-1), did not meet the air volume goal. This error was due to a programming malfunction and not battery failure, therefore, the test run was deemed successful and samples were submitted for analysis (Tetra Tech 2010b).

Sampling data and observations were recorded daily on FSDSs. FSDSs for the samples collected through Sample Period 6 (December 23, 2009), are provided in Appendix A of the AAS first quarter memorandum (Tetra Tech 2010b).

Pump performance during winter temperature drops was still a concern since the operating temperature range of the AirChek2000 sampling pump is 32 to 100 degrees Fahrenheit. To maintain temperatures above 32 degrees Fahrenheit, Tetra Tech purchased 12-volt deep cycle batteries and operated miniature heaters inside the sample station boxes during the winter months (Tetra Tech 2010b).

The air sampling pumps were programmed to record atmospheric pressure in the "INS" mode (inches of mercury [Hg]); however, the FSDSs incorrectly referenced "mm/Hg" (millimeters of Hg) mode. The FSDS sheets were corrected after period 7 to reference "INS" (Tetra Tech 2010b).

Sampling Schedule

At each of the seven outdoor ambient air sampling stations, sampling occurred on a regular 10-day schedule (5 days on and 5 days off). This results in the collection of 36 field samples per year, per station. Sample collection at each station was programmed to begin at the same time on a predetermined day of the week.

Samples were collected continuously during the 5-day (120-hour) sampling event until the target sample volume was reached. The pump was programmed to record and store flow data on a regular interval of once every hour. The AirCheck2000 pumps were programmed to shut off after the target volume was reached. When target volumes were reached, samples were retrieved and prepared for shipment to the laboratory. Pump data were downloaded to a computer and stored in an electronic format.

7.3.2 Outdoor Ambient Air Analytical Results

As of December 23, 2009, six rounds of outdoor ambient air samples totaling 54 individual samples (including field QC samples), had been collected from 8 stations in OU7. As of December 31, 2009, no analytical results were available.

8.0 CONCLUSIONS AND RECOMMENDATIONS

8.1 CONCLUSIONS

The objectives of the RI for OU7 of the Libby Asbestos Superfund Site were to adequately characterize the nature and extent of LA contamination by investigating building interiors, soil, and outdoor ambient air; to provide community support during the investigations; identify parcels that meet the EPA removal action levels; collect and provide reliable data on LA contamination to support RDI and removal action activities; gather sufficient data for a future site-wide risk assessment; and allow for effective development of alternative remedies to be included in an FS.

Tetra Tech conducted TAPE investigations on 1,239 out of 1,578 parcels between April 1, 2007 and December 31, 2009. Three hundred two (302) of the inspected properties were public road and alley parcels. Twenty-eight (28) of the inspections occurred at parcels outside of the OU7 boundary. The TAPE investigations included the visual inspection of 2,149 primary and secondary buildings, and the collection of 1,784 interior dust samples, 3,320 exterior soil samples, and 486 interior soil samples (excluding QA/QC samples). The AAS had completed 6 sampling periods as of December 31, 2009. No analytical data were available at that time.

The DEQ Troy Information Office was opened in April 2007 and continues to serve OU7 residents and community members by providing information on LA and TAPE/AAS procedures. A full-time CIC is available to the public and is responsible for scheduling properties for TAPE inspections, answering property owner questions, providing informational pamphlets on LA, supporting the Tetra Tech field inspection staff, filing and maintaining all hard-copy field paperwork, maintaining all records in TOAD, and preparing scheduling packets for properties undergoing an initial TAPE inspection. Over the course of the project, the DEQ Project Officer, Tetra Tech Field Team Leader, and Tetra Tech management personnel have been alerted to various issues at properties because of the open line of communication between the CIC and OU7 residents.

Of the 1,239 parcels that underwent a TAPE inspection, Tetra Tech and the DEQ were able to identify 396 parcels that met at least one of the EPA removal action levels (Section 7.2.2). The DEQ used the EPA removal action levels in conjunction with OU7 secondary triggers and full parcel reviews to further evaluate the 396 parcels meeting EPA removal action levels and determine which were eligible for removal action within OU7. A total of 102 parcels within OU7 were identified for interior removal,

exterior removal, or a combination of both interior and exterior removal action (Section 7.2.4). The removal actions will be initiated after RDI activities have been completed at each parcel; this work is anticipated to begin in April 2010.

The TAPE investigations have generated an extensive amount of data on the location of visible vermiculite and vermiculite insulation within and around the buildings in OU7. In addition, information has been compiled on the history of the buildings and parcels, resident health and previous exposure to Libby vermiculite (to some degree), and how the parcels are utilized by the residents (i.e. use area information). It is presumed these data will be pertinent to any future site-wide risk assessments that will be conducted once the toxicity of LA is determined. These data will be sufficient for beginning the FS process.

8.2 RECOMMENDATIONS

Tetra Tech makes the following recommendations to the DEQ regarding investigations in OU7:

- Address the data gap of uninspected parcels by conducting TAPE inspections on the 339 unaddressed parcels within OU7.
- Perform removal actions for parcels identified based on EPA removal action levels, as amended for OU7, to minimize or eliminate resident exposure to LA;
- Continue to evaluate OU7 parcels for possible removal action as remaining TAPE investigations are performed;
- Finish the first year of AAS sample collection and review all data for accuracy, completeness, and validity;
- Evaluate the need to continue the outdoor ambient air sampling program beyond the first year and continue the program if deemed appropriate;
- Maintain community support and outreach in OU7;
- Once toxicity of LA has been calculated, perform a risk assessment using OU7 TAPE and AAS data;
- Develop appropriate remedial action alternatives.

Additional data gaps and/or recommendations may depend on activity-based sampling in OU4.

8.3 FEASIBILITY STUDY PROCESS

After the OU7 contaminant characterization has been completed through the RI process, EPA toxicological studies completed, and risks to human health and the environment evaluated, the FS process will begin to develop and evaluate remedial alternatives that can be used to remediate those areas within OU7 that are identified as posing an 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 Record of Decision document, development of the remedial design, and implementation of the remedial action.

9.0 REFERENCES

Base Map Service Center. 2010. Montana Cadastral Codes. Accessed on July 2, 2010. http://giscoordination.mt.gov/data.asp#Cadastral

Boettcher, A.L. 1966. The Rainy Creek igneous complex near Libby, Montana. Ph.D. dissertation, 155p.: Pennsylvania State University, University Park.

Boettcher, A.L. 1967. The Rainy Creek alkaline-utramafic igneous complex near Libby, Montana. I: Ultramafic rocks and fenite. Journal of Geology, v. 75, p. 526-553.

Bush, A.L. 1976. Vermiculite in the United States, in Eleventh Industrial Minerals Forum: Montana Bureau of Mines and Geology Special Publication 74, p. 145–155.

Camp, Dresser, and McKee (CDM). 2007a. Draft Quality Assurance Project Plan, Libby Asbestos Site, Libby, Montana. January.

CDM. 2007b. Libby Asbestos Project Draft Pilot Study30-point Dust Composite Sampling. Standard Operating Procedure. April.

CDM. 2008a. Addendum to the Response Action Work Plan, Callahan Creek Removal Plan, Troy, Montana. July 25.

CDM. 2008b. Technical Memorandum. Troy Visual Vermiculite Investigation, First Event. July 31. From Nicole Bein, CDM to Mark Raney, Volpe.

CDM. 2009a. Final Remedial Investigation Report for Operable Unit 1 – Former Export Plant Site. August.

CDM. 2009b. Technical Memorandum. Troy Visual Vermiculite Investigation, Second Event – Revision 1. April 16. From Nicole Bein, CDM to Mark Raney, Volpe.

Center for Asbestos Related Disease (CARD). 2010. Information pertaining to the health effects of asbestos. Accessed 7/8/2010 online at: http://www.libbyasbestos.org/libby/understanding.cfm

Ground Water Information Center. 2010. Well Construction Data for OU7. Accessed 6/28/2010 online at: http://mbmggwic.mtech.edu/sqlserver/v11/menus/menuData.asp

Harrison, J.E., Cressman, E.R. and Whipple, J.W. 1992. Geologic and structure maps of the Kalispell 1 degree x 2 degrees quadrangle, Montana, and Alberta and British Columbia: U.S. Geological Survey, Miscellaneous Investigations Series Map I-2267, scale 1:250000.

Harrison, J.E. and Cressman, E.R. 1993. Geology of the Libby thrust belt of northwestern Montana and its implications to regional tectonics. U.S. Geological Survey.

Meeker, G.P., A.M. Bern, I.K. Brownfield, H.A. Lowers, S.J. Sutley, T.M. Hoefen, and J.S. Vance. 2003. The Composition and Morphology of Amphiboles from the Rainy Creek Complex, Near Libby, Montana. American Mineralogist. November-December. v. 88 no. 11-12. P.1955-1969.

Montana Bureau of Mines and Geology (MBMG). 1963. Mineral and Water Resources of Montana. Special Publication No. 28. May.

MBMG. 1978. Ground-Water Resources in the Libby Area, Northwestern Montana. Arnold J. Boettcher and Kathleen R. Wilke. Bulletin 106. June.

Montana Department of Environmental Quality (DEQ). 2009a. Final Libby Asbestos Site, Troy Operable Unit 07, Residential/Commercial Cleanup Criteria, Specific Use Area Visible Vermiculite Action Level Technical Memorandum. April 23.

DEQ. 2009b. Libby Asbestos Site, Troy Operable Unit 07, Removal Parcel Status. November 30.

National Oceanic and Atmospheric Administration (NOAA). 2010. Accessed online June 13, 2010 at 3:00pm at: http://www.noaa.gov/index.html

Natural Resources Conservation Service. 2010. Web Soil Survey for Troy Area. Accessed 6/15/2010 at: http://websoilsurvey.nrcs.usda.gov/app/WebSoilSurvey.aspx

Natural Resource Information System. 2010. Montana Digital Atlas. Accessed 7/8/10 for wetland information at: http://maps2.nris.mt.gov/mapper/

Tetra Tech EM Inc. (Tetra Tech). 2007. Troy Asbestos Property Evaluation Work Plan. July.

Tetra Tech. 2008a. Final Data Management Plan for the Troy Asbestos Property Evaluation Project. March.

Tetra Tech 2008b. Record of Modification TFO-00008 to the TAPE Work Plan, Confirmation and Description of Visible Vermiculite in Use Areas and Sampling of Specific Use Areas. May 13.

Tetra Tech. 2009a. Remedial Investigation Work Plan, Outdoor Ambient Air Study (AAS). Final. October.

Tetra Tech. 2009b. Protocol Modification to the TAPE Work Plan, Inspection and Sampling of Roads and Alleys for Visual Vermiculite. July.

Tetra Tech. 2009c. Final Data Management Plan (Version 3.0) for the Troy Asbestos Property Evaluation Project. August.

Tetra Tech. 2009d. Data Management Plan (Version 2.0) for the Troy Asbestos Property Evaluation Project. March.

Tetra Tech. 2010a. Draft Report of Findings (Version 3) for Properties Inspected in 2009 for the Troy Asbestos Property Evaluation Project.

Tetra Tech. 2010b. Final First Quarter Memorandum, Outdoor Ambient Air Study, Operable Unit Number 7 of the Libby Asbestos Superfund Site. February.

US 2000 Census. 2000. Troy Demographics. Accessed 6/24/2010 online at: http://ceic.mt.gov/

U.S. Environmental Protection Agency (EPA). 1988. Guidance for Conducting Remedial Investigations and Feasibility Studies Under the Comprehensive Environmental Response, Compensation, and Liability Act. Interim Final. October.

EPA. 1993. Method for Determination of Asbestos in Bulk Building Materials. Test Method. July.

EPA. 2003. Libby Asbestos Site Residential/Commercial Cleanup Action Level and Clearance Criteria, Technical Memorandum. Draft Final. December 15.

EPA. 2006a. Final – Revision 1, Sampling and Analysis Plan for Outdoor Ambient Air Monitoring at the Libby Asbestos Site, Operable Unit 4, Libby, Montana. Region 8, Denver, Co. December.

EPA. 2006b. Integrated Risk Information System (IRIS). On-line database of toxicity information maintained by the EPA at http://www.epa.gov/iriswebp/iris/index.html

EPA. 2010. Dosimetric and Toxicologic Assessment of Amphibole Fiber-Containing Material. Accessed 7/28/2010 online at: http://www.epa.gov/Region8/superfund/libby/amphibole.html.

United States Geological Survey (USGS). 2005. Reconnaissance Study of the Geology of U.S. Vermiculite Deposits – Are Asbestos Minerals Common Constituents? U.S. Geological Survey Bulletin 2192. Van Gosen, B.S. et al. Version 2.0 May 7.

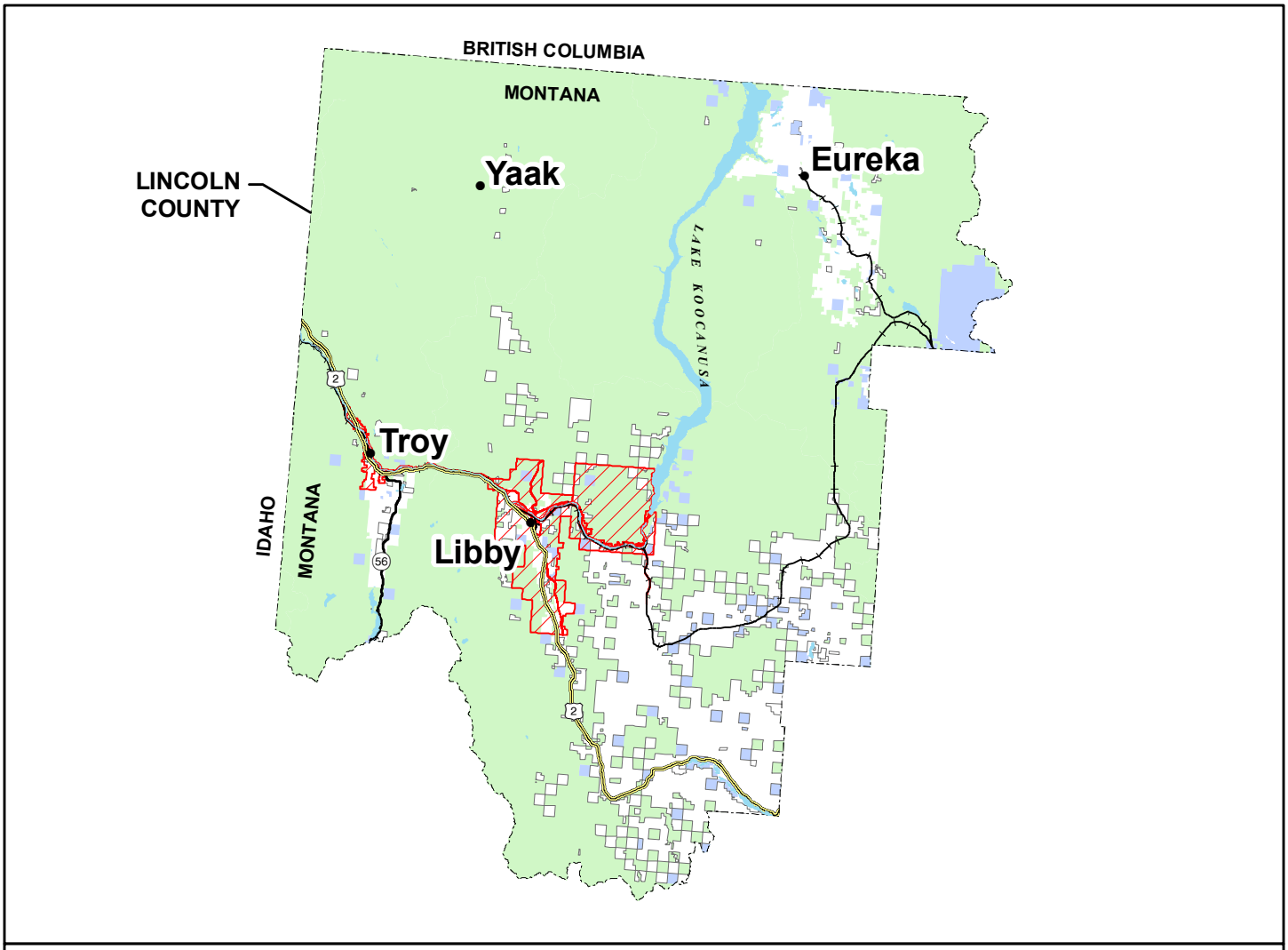
University of Idaho (modified from Bandli, B. R.) 2002. "An overview of the mining history, geology, mineralogy, and amphibole-asbestos health effects of the Rainy Creek igneous complex, Libby, Montana, U.S.A.: A case study in teaching environmental mineralogy". Characterization of amphibole and amphibole asbestos from the former vermiculite mine at Libby, Montana, U.S.A., M.S. Thesis, University of Idaho, Moscow, Idaho, U.S.A.

Van Gosen, Bradley. 2007. "*Geology of Asbestos in the United States and Its Practical Applications*". February.

Volpe 2009. Final RI Report for OU2

Western Regional Climate Center. 2010. Period of Record Monthly Climate Summary for Troy, Montana. Accessed online 6/24/2010 at: http://www.wrcc.dri.edu/summary/Climsmnidwmt.html

FIGURES



LOCATION OF LARGER MAP

TETRA TECH EM, INC.

LEGEND

- TOWNS
- STATE HIGHWAY
- US HIGHWAY
- RAILROAD
- OPERABLE UNITS
- NATIONAL FOREST LAND
- PRIVATE LAND
- STATE LAND
- WATER

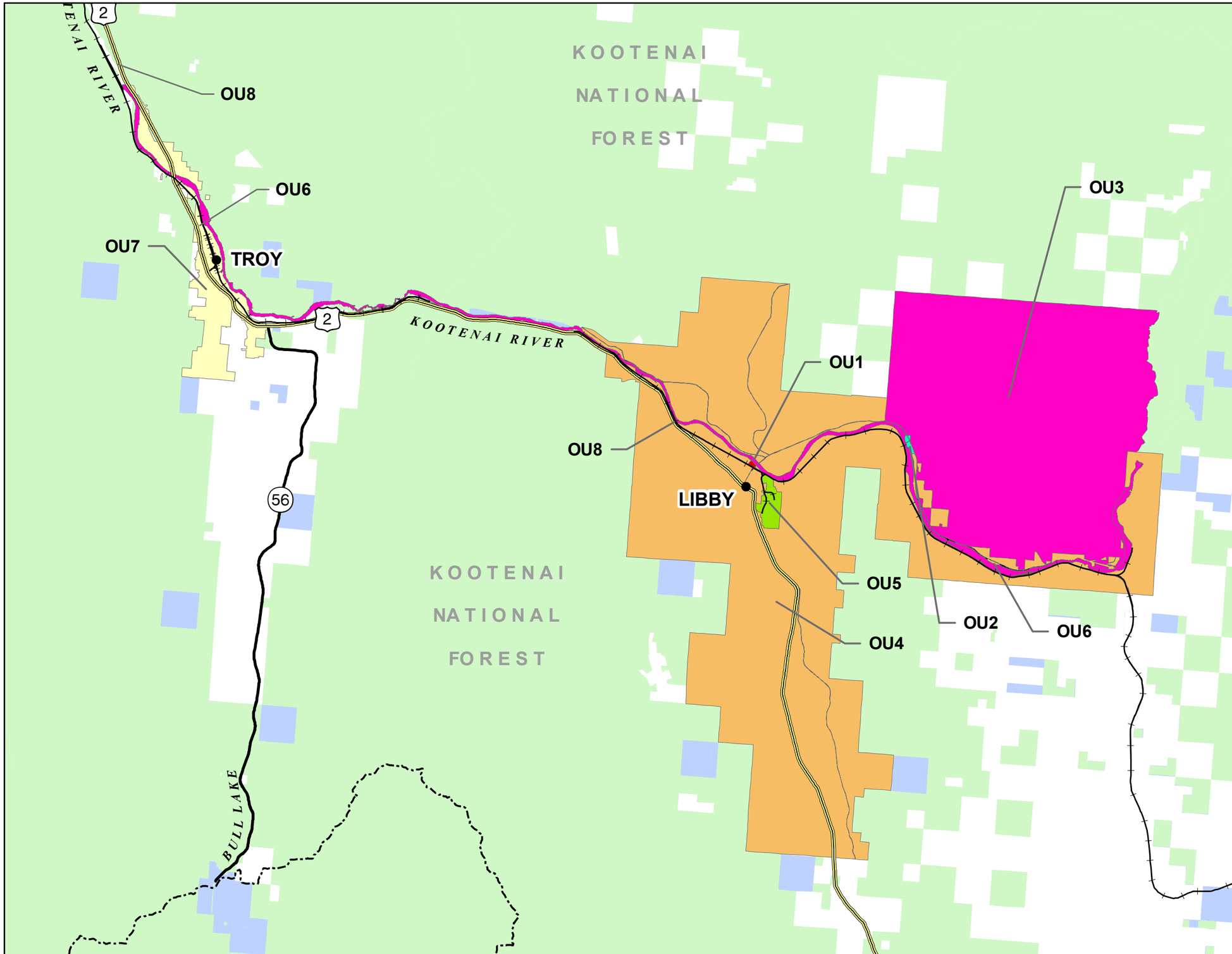
0 10 Miles

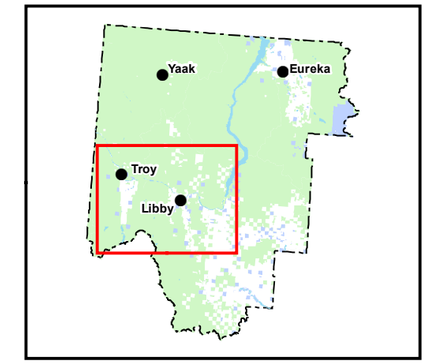
N

DRAFT OU7 RI
LIBBY ASBESTOS SUPERFUND SITE

FIGURE 1-1
LOCATION OF
LIBBY ASBESTOS SUPERFUND SITE
IN MONTANA

GIS map by Ed Madej TTEMI-HE
FIG1-1_OU7RI_LOCATION_MAP_081710.mxd





LOCATION MAP

LEGEND

OPERABLE UNITS

- OU1 - FORMER EXPORT PLANT
- OU2 - FORMER SCREENING PLANT
- OU3 - MINE SITE
- OU4 - RESIDENTIAL AND COMMERCIAL PROPERTIES IN AND AROUND LIBBY
- OU5 - FORMER STIMSON LUMBER MILL
- OU6 - BNSF RAIL LINE
- OU7 - RESIDENTIAL AND COMMERCIAL PROPERTIES IN AND AROUND TROY
- OU8 - US AND MONTANA STATE HIGHWAYS AND SECONDARY HIGHWAYS IN THE VICINITY OF LIBBY AND TROY MONTANA
- TOWNS
- STATE HIGHWAY
- US HIGHWAY
- RAILROAD
- NATIONAL FOREST LAND
- PRIVATE LAND
- STATE LAND
- WATER

0 2 Miles

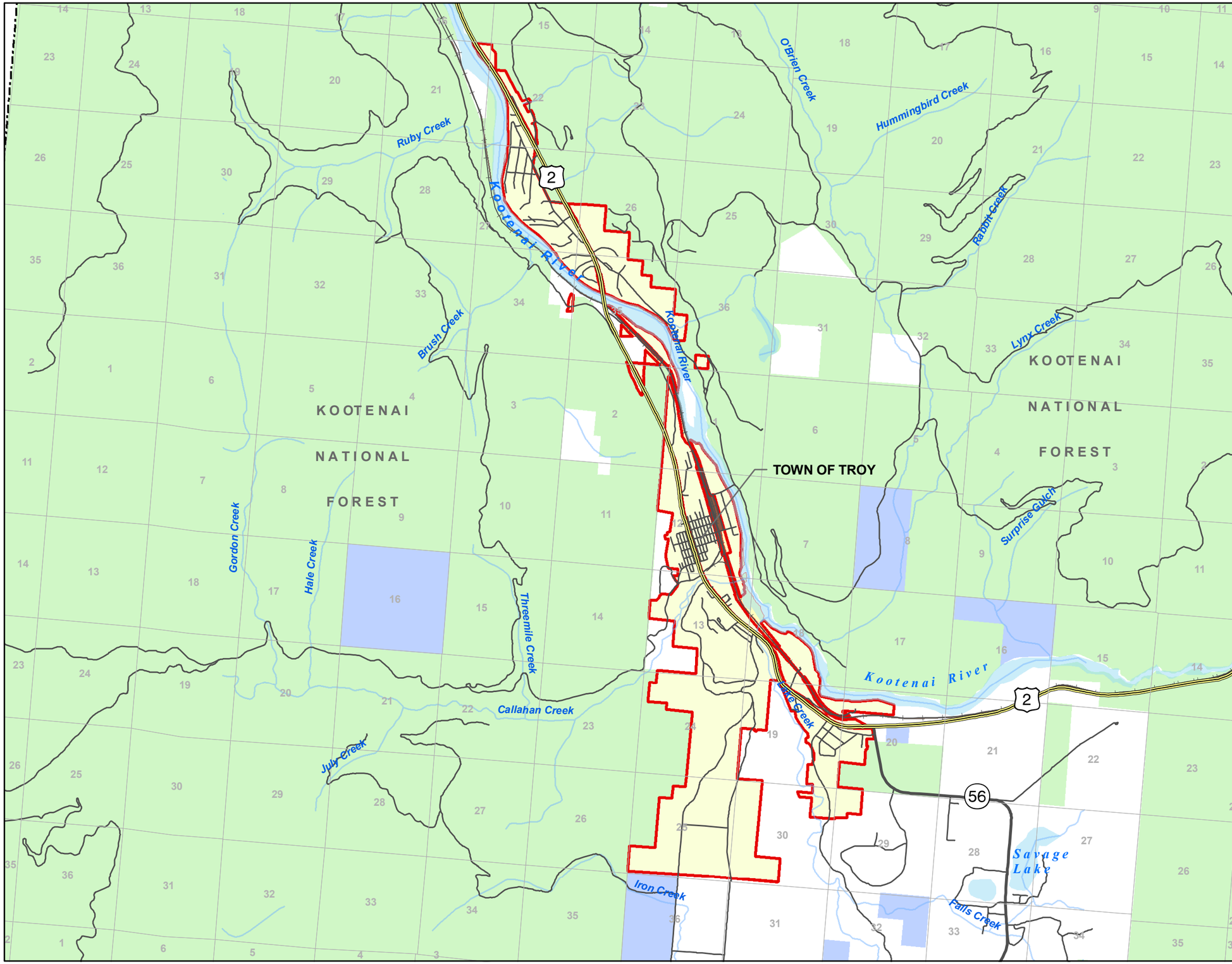
N

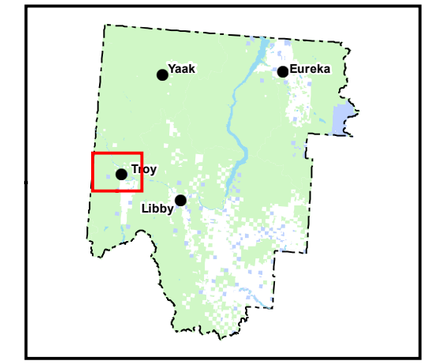
TETRA TECH EM, INC.

DRAFT OU7 RI
LIBBY ASBESTOS SUPERFUND SITE

FIGURE 1-2
LOCATION OF
LIBBY ASBESTOS SUPERFUND SITE
OPERABLE UNITS

GIS map by Ed Madej TTEMI-HE
FIG2_OU7RI_ALL_OUS_081710.mxd





LOCATION MAP

LEGEND

- OU7 BOUNDARY
- US HIGHWAY
- STATE HIGHWAY
- OTHER ROADS
- RAILROAD
- NATIONAL FOREST LAND
- PRIVATE LAND
- STATE LAND
- WATER

0 1 Miles

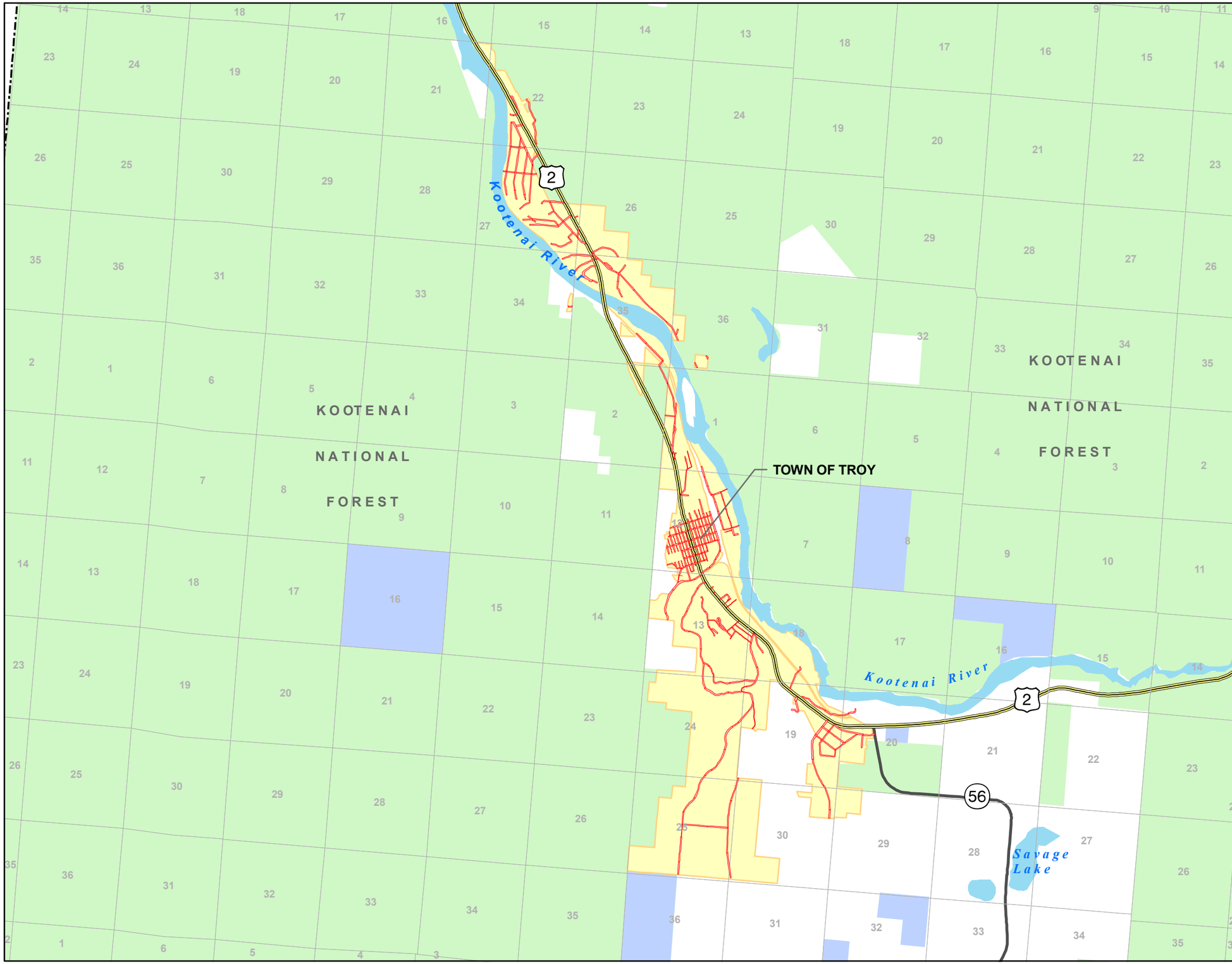
N

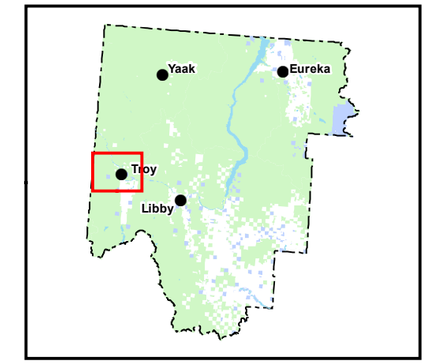
TETRA TECH EM, INC.

DRAFT OU7 RI
LIBBY ASBESTOS SUPERFUND SITE

FIGURE 1-3
OPERABLE UNIT 7 (OU7)
BOUNDARIES

GIS map by Ed Madej TTEMI-HE
FIG3_OU7RI_BOUNDARY_081710.mxd





LOCATION MAP

LEGEND

- OU7 BOUNDARY
- ROAD AND ALLEY PARCELS INSPECTED IN 2009 = 316 PARCELS
- US HIGHWAY
- STATE HIGHWAY
- NATIONAL FOREST LAND
- PRIVATE LAND
- STATE LAND
- WATER

0 1 Miles

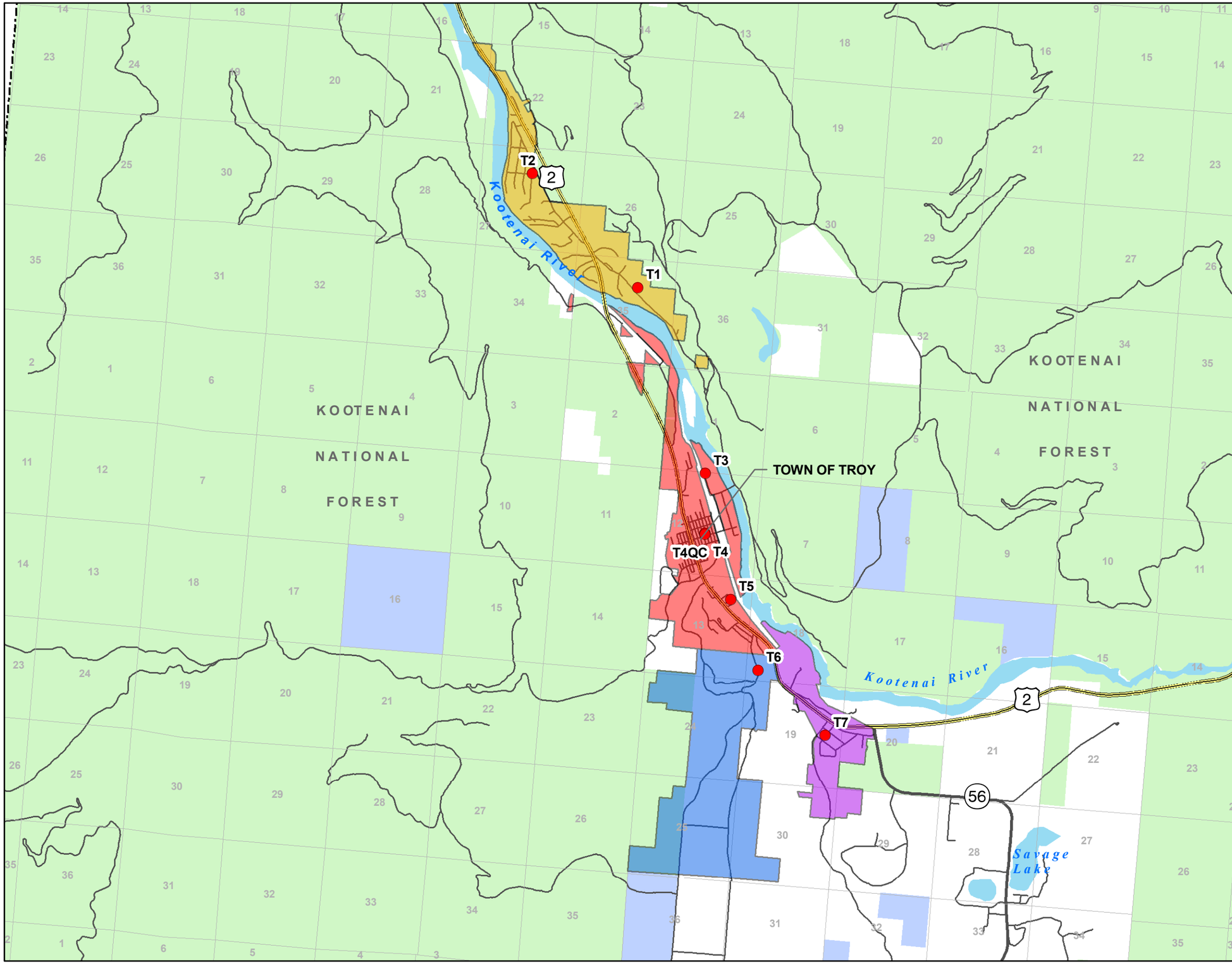
N

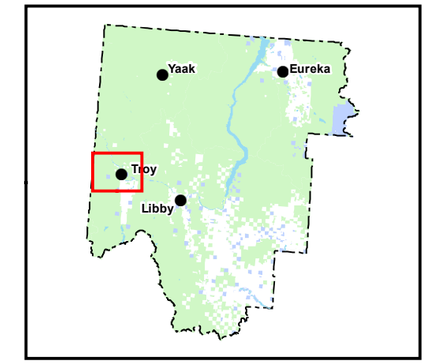
TETRA TECH EM, INC.

DRAFT OU7 RI
LIBBY ASBESTOS SUPERFUND SITE

FIGURE 4-1
ROAD AND ALLEY PARCELS

GIS map by Ed Madej TTEMI-HE
FIG4-1_OU7RI_ROAD_AND_ALLEY_PARCELS_081710.mxd





LOCATION MAP

LEGEND

- TROY AMBIENT AIR SAMPLE STATION

AIR ZONE

- ZONE 1
- ZONE 2
- ZONE 3
- ZONE 4
- US HIGHWAY
- STATE HIGHWAY
- OTHER ROADS
- NATIONAL FOREST LAND
- PRIVATE LAND
- STATE LAND
- WATER

0 1 Miles

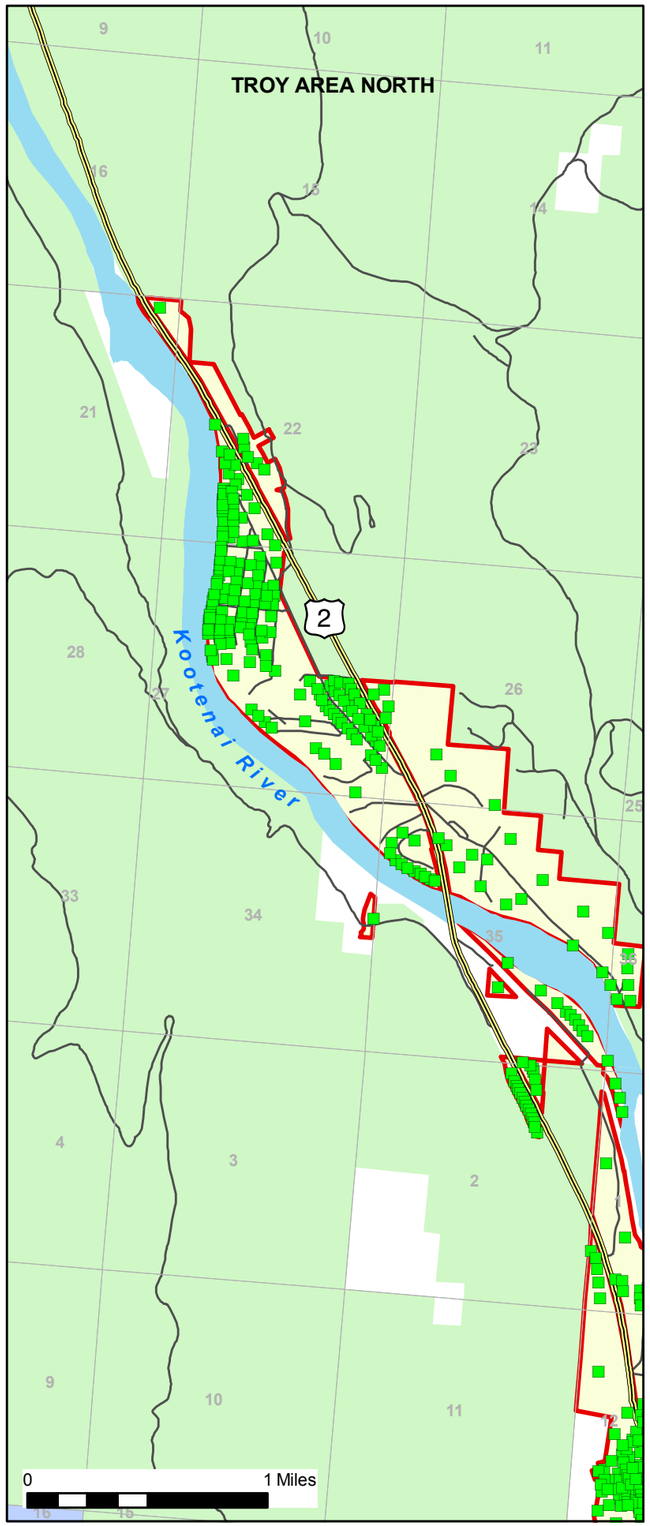
N

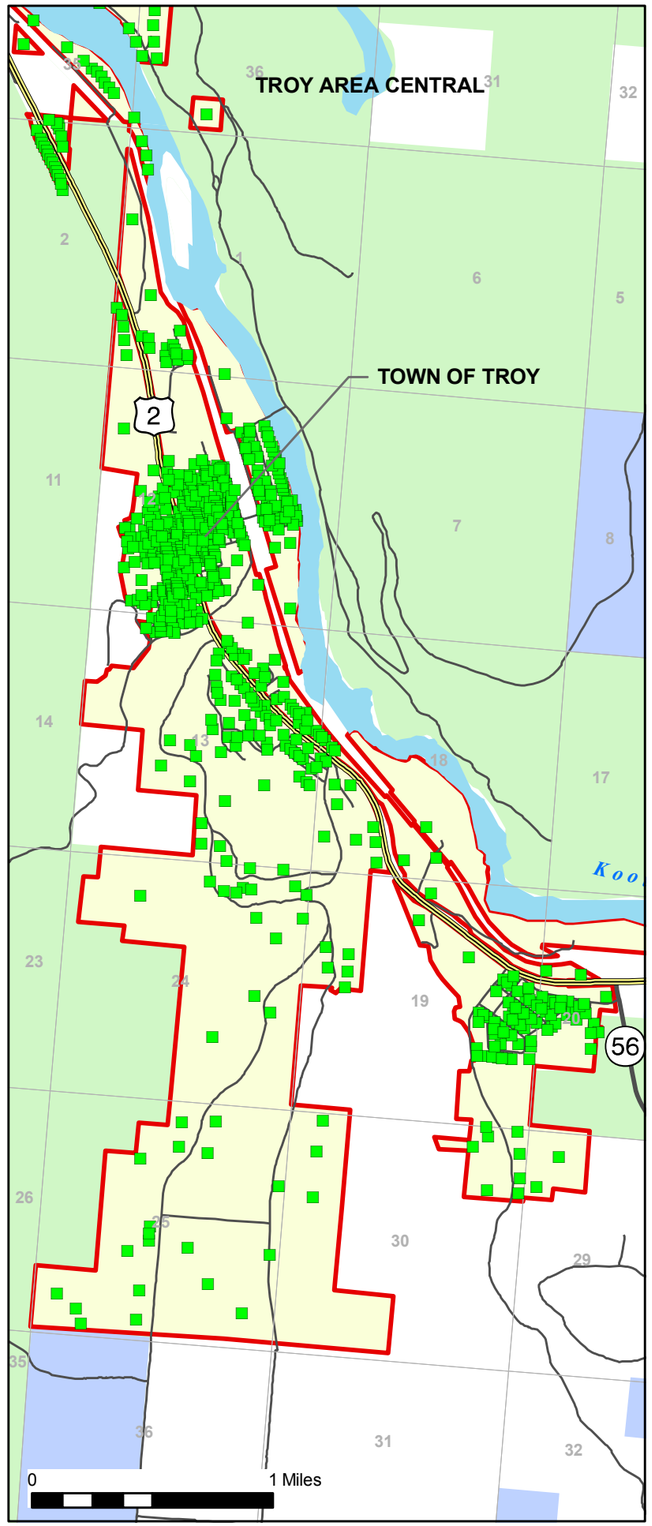
TETRA TECH EM, INC.

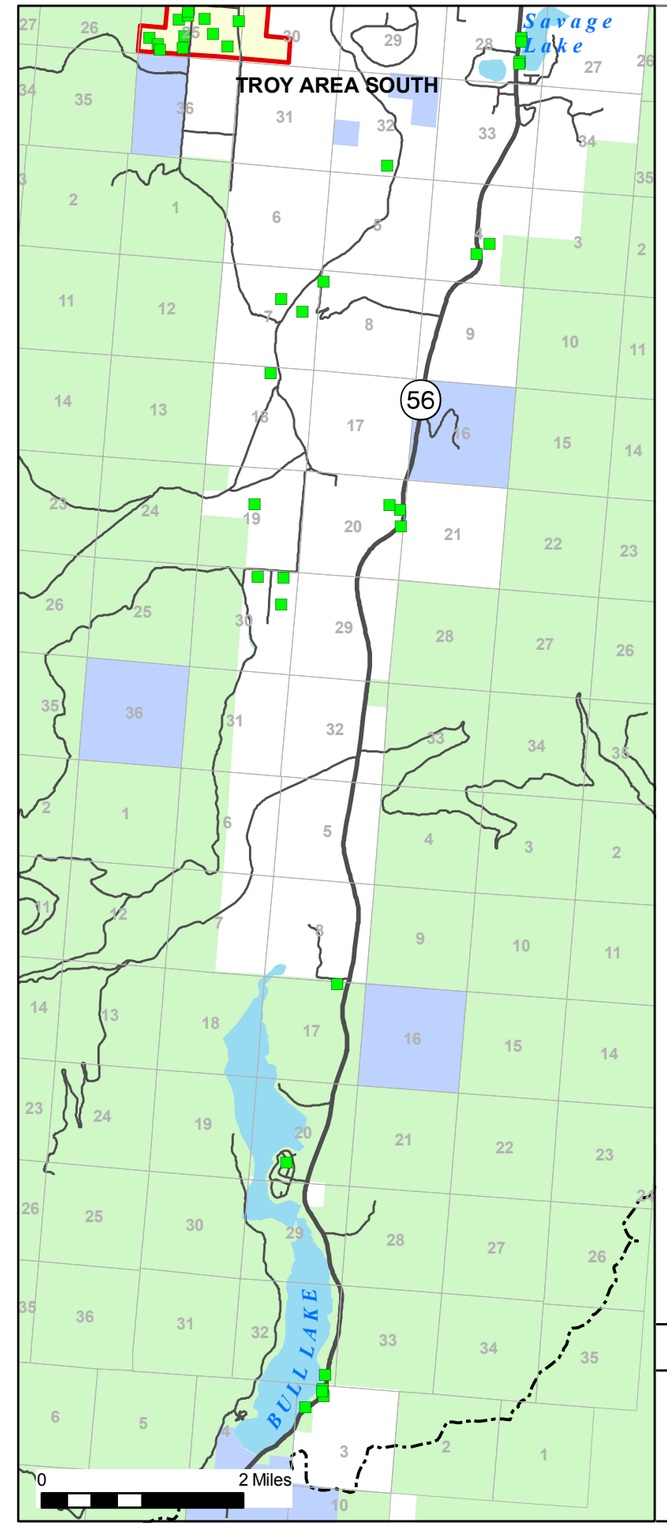
DRAFT OU7 RI
LIBBY ASBESTOS SUPERFUND SITE

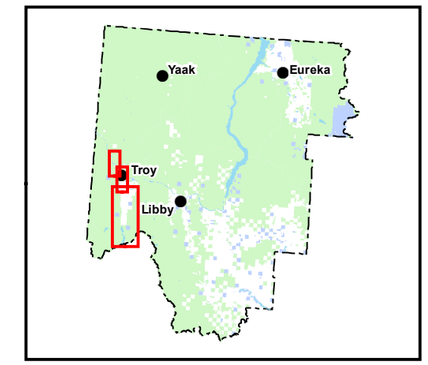
FIGURE 5-1
OUTDOOR AMBIENT AIR MONITORING STATION LOCATIONS

GIS map by Ed Madej TTEMI-HE
FIG5-1_OU7RI_AMBIENT_AIR_MONITORING_081710.mxd









LOCATION MAP

LEGEND

- INSPECTED PARCELS AS OF DECEMBER 31, 2009 = 937 PARCELS
- OU7 BOUNDARY
- US HIGHWAY
- STATE HIGHWAY
- OTHER ROADS
- NATIONAL FOREST LAND
- PRIVATE LAND
- STATE LAND
- WATER

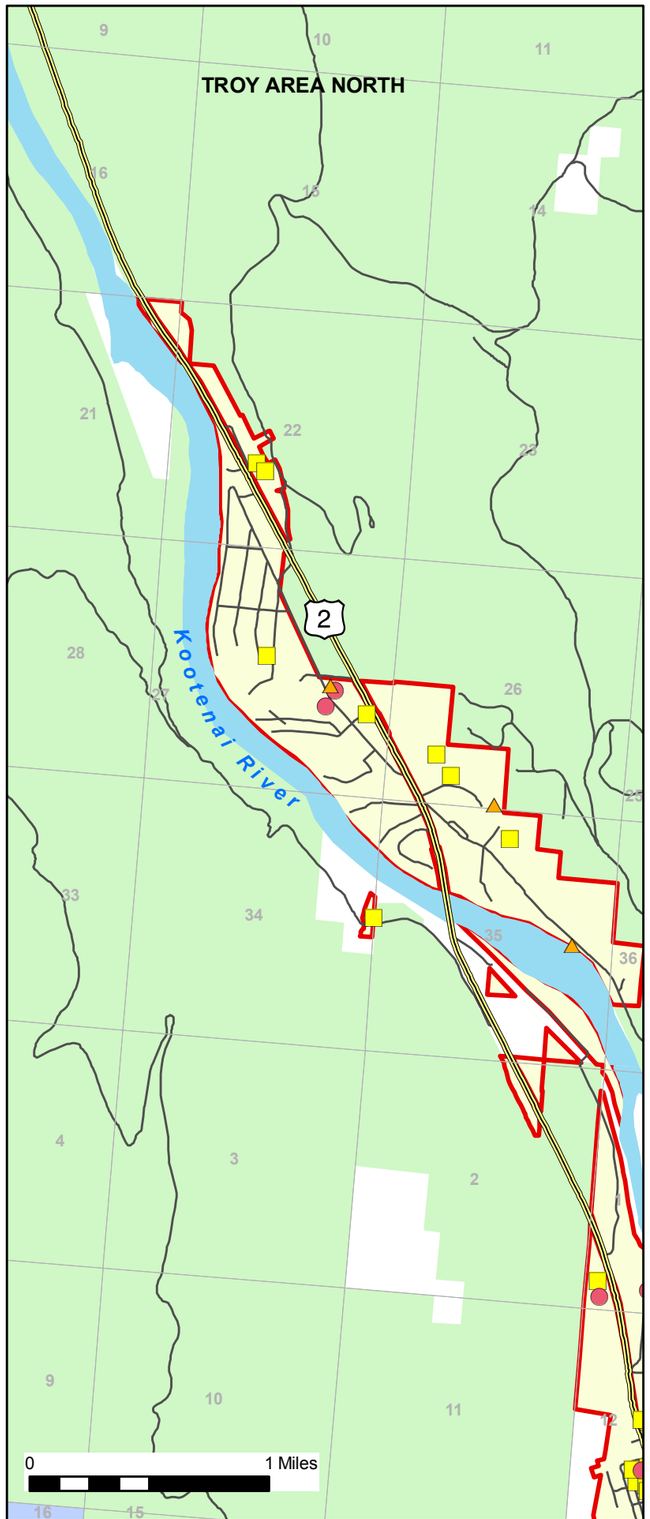
N

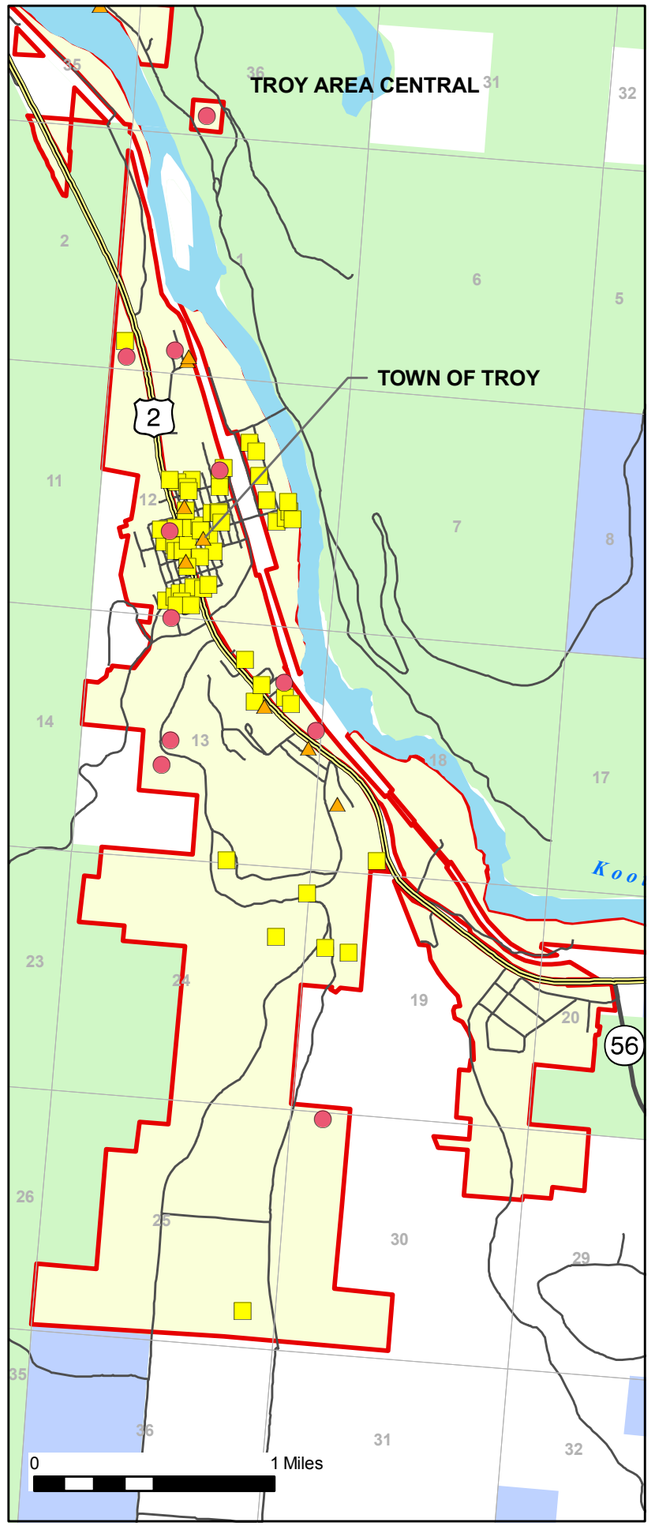
TETRA TECH EM, INC.

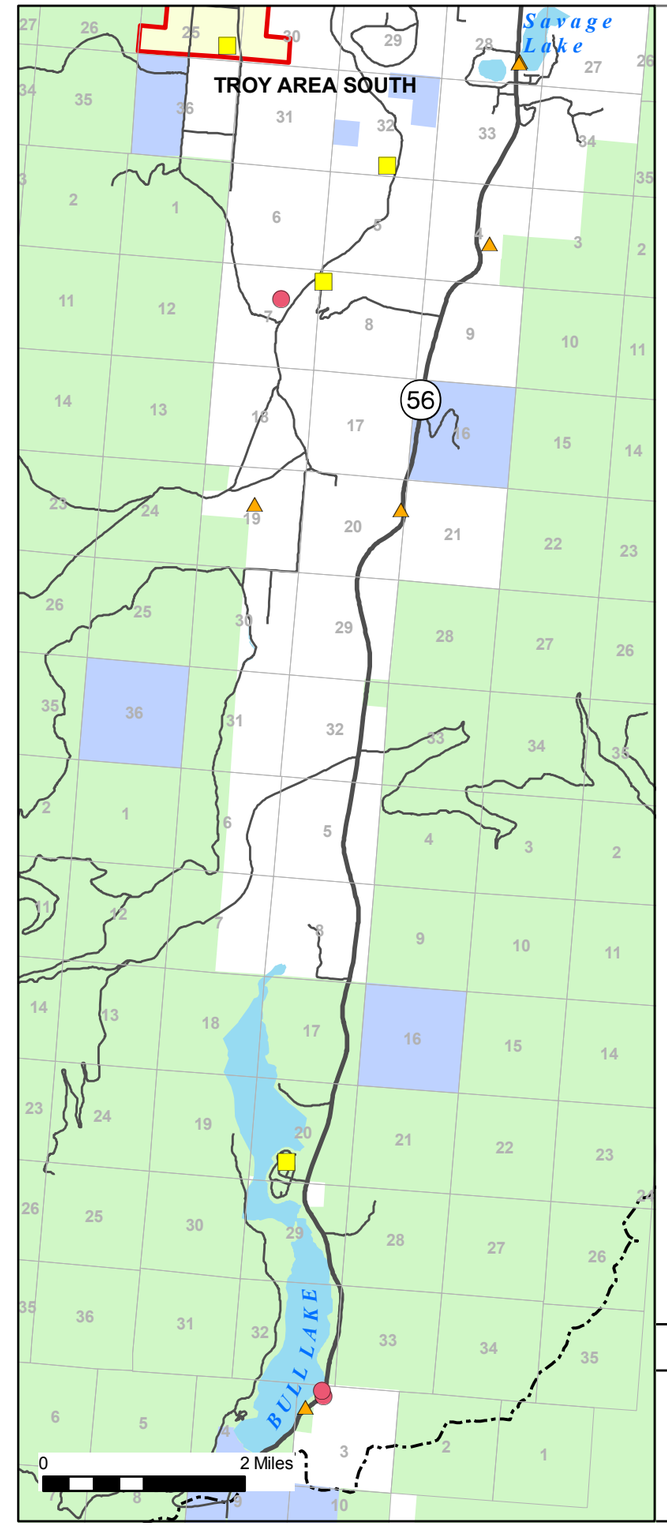
DRAFT OU7 RI
LIBBY ASBESTOS SUPERFUND SITE

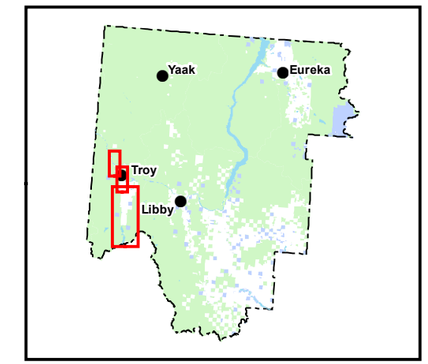
FIGURE 7-1
TAPE INSPECTED PARCELS IN OU7 AND VICINITY

GIS map by Ed Madej TTEMI-HE
FIG7-1_OU7RI_INSPECTED_PARCELS_081710.mxd









LOCATION MAP

LEGEND

- EXTERIOR AND INTERIOR REMOVALS = 17
- EXTERIOR REMOVALS ONLY =17
- INTERIOR REMOVALS ONLY = 71
- OU7 BOUNDARY
- US HIGHWAY
- STATE HIGHWAY
- OTHER ROADS
- NATIONAL FOREST LAND
- PRIVATE LAND
- STATE LAND
- WATER

N

TETRA TECH EM, INC.

DRAFT OU7 RI
LIBBY ASBESTOS SUPERFUND SITE

FIGURE 7-2
REMOVAL ACTION PARCELS
IN OU7 AND VICINITY

GIS map by Ed Madej TTEMI-HE
FIG6-2_OU7RI_REMOVAL_ACTION_PARCELS_102810.mxd

APPENDIX A

STANDARD OPERATING PROCEDURES (SOPs)

Site-Specific Sampling Guidance
Libby Superfund Site

Guidance No: **CDM-LIBBY-10, Revision 1**

Guidance Title: Collection of 30-Point Composite Microvacuum Dust Samples for Determining Nature and Extent of Libby Amphibole Asbestos (LA) in Indoor Dust

Approved by:

Technical Reviewer — Date: 5/10/07

QA Reviewer — Date: 5/10/07

Volpe Center Approval — Date: 05/10/07

EPA Approval — Date: 5/10/07

Section 1
Purpose

The purpose of this standard operating procedure (SOP) is to provide a consistent method for the collection of 30-point composite microvacuum dust samples. This SOP is to be used by contractors/subcontractors supporting EPA investigation activities at the Libby Superfund Site. This SOP describes the processes by which sample locations will be selected and the procedures used to collect samples. Samples collected according to this SOP can be used to determine the nature and extent of LA in indoor dust for assessing clean-up requirements.

Section 2
Responsibilities

Successful execution of this SOP requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role. All staff with responsibility for the collection of indoor dust samples is responsible for understanding and implementing the requirements contained herein as well as any other governing guidance documents.

Task Leader (TL) or Field Team Leader (FTL) - The TL or FTL is responsible for overseeing sample collection processes as described in EPA-approved governing guidance documents (i.e., site-specific sampling and analysis plans [SAPs], quality assurance project plans [QAPPs], etc.). The TL or FTL is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and all governing guidance documents. The TL or FTL will communicate with the field team members regarding the specific collection objectives and anticipated situations that require deviation from this SOP. It is also the responsibility of the TL or FTL to communicate the need for any deviations from the SOP with the appropriate EPA personnel (team leader or their designate), and document the deviations using a Field Modification Form provided in each SAP or QAPP.

Field team members - Field team members performing the sampling described in this SOP are responsible for adhering to the applicable tasks outlined in this procedure while collecting samples at properties associated with the Libby Superfund Site. The field team members should have limited discretion with regard to collection procedures but should exercise judgment regarding the exact location of sample points, within the boundaries outlined by the TL or FTL.

Section 3
Equipment

This section provides a list of equipment required to collect dust samples according to the site-specific protocols detailed in Section 4 and to meet the requirements of American Society for Testing and Materials (ASTM) method D5755-03 (ASTM 2003).

- Sampling pump – The sample pump used in the collection of microvacuum dust samples will be capable of flow rates typically used for dust sampling, 2.0 liters per minute (L/min). The pump must be capable of providing a non-fluctuating air-flow through the sampling media, and maintain the initial flow-rate volume to within ±10 percent (%) throughout the sampling period.

- Rotameter – A rotameter will be used as a secondary calibration standard as required for verifying the flow rate of the sampling pump used for sample collection. The rotameter will be calibrated such that the operator can measure flow rates to ±5% accuracy at the expected flow rate. Each rotameter in use should be calibrated against a primary standard as required according to manufacture recommendations and governing guidance documents.

- Sample cassettes – The sample cassettes used for the collection of microvacuum dust samples at the Libby Superfund Site are a commercially available 25-millimeter (mm), three-piece cassette with a 50-mm electronically conductive extension cowl loaded with a 0.45 micrometer (µm) mixed cellulose ester (MCE) filter. The sampling nozzle attached to the cassette inlet will meet the following specifications as described in ASTM D5755-03 (ASTM 2003): the sampling end will be cut at a 45° angle, and the length of tubing will be at least 25 to 37 mm in length and 0.25 inches in diameter.

- Inert tubing - Tygon® tubing with a 3/16-inch inner diameter and 5/16-inch outer diameter is used in the sample collection train to connect the outflow end of the sample cassette to the sampling pump.

- Sample ID labels (Index IDs) – pre-printed index ID number labels are placed on the sampling cassette to indicate the unique sampling number assigned to the sample. Index ID labels can also be used in logbooks/PDAs and on other field forms for sample identification. The specific index ID numbers used will be detailed in governing guidance documents.

- Collection area templates – 10 by 10 centimeter (cm) reusable plastic or disposable cardstock (paper) templates are used to delineate each sample point. When a plastic template is used, it will be wiped with a disposable wet towel between each individual sample (i.e., not between individual aliquots). When a paper template is used, a new template will be used after each sample.

- Zip-top plastic bag – after sample collection is complete, each sample cassette will be placed in an individual zip-top plastic bag. The index ID label will be placed on the outside of the zip-top bag and affixed with clear tape if necessary. The index ID may also be written on the outside of the bag using a permanent marker (preferred). Sample cassettes are placed into individual bag to mitigate the potential for cross-contamination in the event that a cassette should open during handling or shipping.

- Field logbook or PDA -used to record progress of the sampling effort and record any problems and field observations.
- Cooler or other ridged container - used to store samples while in the field.
- Custody Seals - aid in ensuring the integrity of samples during handling or shipping.
- Latex or Nitrile Gloves – Worn during dust sample collection to prevent cross-contamination.

Section 4
Selection of Sample Locations

Governing guidance documents should be consulted to determine when microvacuum dust sampling is required.

When sampling is required, one 30-point composite sample will be collected on a single sampling cassette per living floor, or as required for secondary buildings. Each dust sample will be collected from areas classified on a scale of accessibility as described below:

1. **Accessible** areas refer to locations where exposures are most likely to occur – places where dust accumulates and is encountered daily. This includes soft surfaces such as carpet (not including movable floor mats), upholstered furniture, floors, and waist-high hard surfaces such as counter tops and non-carpeted floors.

2. **Infrequently accessed** areas refer to locations where dust may accumulate, but exposures are likely to occur infrequently. This includes areas on tops of shelves, entertainment centers, and refrigerators, etc.

3. **Inaccessible** areas refer to locations where dust may accumulate but exposures occur only rarely, such as behind refrigerators or other large infrequently moved objects.

To the extent possible, the sample point locations will be collected from each type of accessibility area as indicated below:

1. **Accessible** target areas, if present (as indicated, some locations described should only be included when visible dust can be observed with the unaided eye of the field team members):
    a. Flooring (soft or hard surface) at the main entrance used by occupants
    b. Flooring at the secondary or less heavily used entrance to the home
    c. Flooring in the center of the living room or family room
    d. Flooring in the center of bedrooms

e. Flooring in an acknowledged or evident route of high traffic (i.e., hallway or other thoroughfare)
f. Flooring in the kitchen
g. Kitchen counter tops, ***only when visible dust is observed***
h. Table tops in the following rooms: dining room, living room, or family room, ***only when visible dust is observed***
i. Table tops (e.g., night stands, bureaus) in bedrooms, ***only when visible dust is observed***
j. Window sills in the dining room, living room, or family room
k. Window sills in the bedrooms
l. Upholstered furniture in the living room

2. **Infrequent** target areas, if present:
   a. Top of the refrigerator, when top is exposed
   b. Top of bookshelves
   c. Shelves of bookshelves
   d. Top of the hot water heater
   e. Top of wood stoves
   f. Fireplace mantels and/or hearths
   g. Beneath the sofa or other large pieces of furniture in the living room
   h. Beneath the bed or other large pieces of furniture in bedrooms
   i. Inside kitchen cabinets most frequently accessed

3. **Inaccessible** target areas, if present:
   a. Beneath infrequently moved heavy appliances when accessible without moving the appliance (e.g., refrigerator, washing machine, dryers, dishwashers, etc.)
   b. Inside forced air floor or ceiling vents in the living room
   c. Inside forced air floor or ceiling vents in the bedrooms
   d. Inside forced air floor or ceiling vents in the kitchen or bathroom
   e. Corners of closets or other similar small areas not frequently accessed or cleaned

The preferred distribution of the 30-sample points among the three target areas described above is as follows:

- 12-sample points collected from **Accessible** target areas
- 12-sample points collected from **Infrequent** target areas
- 6-sample points collected from **Inaccessible** target areas

Sampling Contingencies
The preferred sample distribution may not always be achievable given the varying conditions of buildings at the Libby Superfund Site. This section discusses situations

when the preferred distribution may not be achieved and provides guidance to the field team members for determining how sample points should be distributed.

1. When the preferred distribution cannot be achieved due the lack of locations in a specific target area category, the remaining number of sampling locations required to reach a total of 30-sample points should be distributed among other target areas according to the preferred distribution ratio (2:2:1).

   For example: If 12 **Accessible** and 12 **Infrequent** target areas are identified and sampled, and only 2 **Inaccessible** target areas are identified and sampled; 4 points remain to be sampled so the total number of sample points adds to 30. The four remaining locations should be distributed evenly among **Accessible** and **Infrequent** target areas, with 2 sample locations collected from each area type.

   If the preferred distribution cannot be achieved, the number of sub-sampling points collected for the composite sample will be recorded as specificed by project specific guidance.

   When unfurnished areas, primary buildings, or secondary building require dust sampling, the locations selected for **Accessible** and **Infrequent** target areas should include flooring and all available horizontal surfaces. It may be necessary to collect several sample points from flooring within the same room in order to meet the overall goal of collecting 30points. The potential issues discussed in Sampling Contingency #1 also apply to this situation.

2. In some cases secondary buildings may be so small that 30 discrete sample points do not exist in the building. This is most likely to occur when dust sampling in a pump house or other similarly sized structure. When this situation is encountered, the field team member will record the number of locations that were obtained and document this deviation according the governing guidance document. The potential issues discussed in Sampling Contingency #1 also apply to this situation.

Section 5
Sample Procedures

Once sampling cassettes have been deemed usable via submittal of lot blanks to the analytical laboratory (see Section 8.2), each sample will be collected, after calibration of the sampling pump and identification of individual increments (sub-samples), according to the following procedures modified from ASTM D5755-03 (ASTM 2003):

1. Set-up the sampling train by attaching the sampling cassette to the sampling pump at the outlet side of the cassette with the required tubing. The length of tubing between the sampling cassette and the sampling pump should be long enough to allow sampling locations to be reached without interfering with the

operation of the sampling pump. If a pre-assembled cassette is used, remove the end cap. If a an inlet nozzle must be assembled:

a. Attach an unused portion of tubing, approximately 25.4 mm in length with an internal diameter of 6.35 mm, directly to the inlet orifice.

b. Cut the sampling end of the tubing at a 45° angle leaving a length of tubing between 25 mm and 37 mm between the inlet orifice and the cut end of the tubing.

2. Don latex or nitrile gloves.

3. Place a sampling template on the area to be sampled. Turn the sampling pump on and begin timing using a stopwatch. Each template (sub-sample location) should be sampled for approximately 30 seconds at a flow rate of 2.0 L/min. The field team member should strive to make three orthogonal collection passes per template during the 30second interval. During the collection period, the surface being sampled should not be scraped or abraded with the collection nozzle.

4. When the 30-second collection period has been completed, invert the sampling cassette so the collection nozzle is pointed upwards. Turn the sampling pump off and stop the stopwatch (do not clear the time from the watch).

5. Repeat sample collection as described in Steps 2 and 3 for the remaining sampling points collecting a cumulative time of collection (approximately 15 minutes) on the stopwatch.

6. During the dust sampling pilot, verify the flow rate after every 5th sampling location (5th, 10th, 15th, 20th, 25th, and 30th) according to the following:

a. Turn the sampling pump off.

b. Connect the rotameter in a calibration train. Ensure the rotameter is within 6° of vertical.

c. Turn the sampling pump on.

d. Record the observed flow rates on the rotameter according to governing guidance document.

e. If required, adjust the flow rate back to 2.0 L/min according to instructions provided for the specific the sampling pump in use.

f. Turn the sampling pump off.

g. Record the value of the ending flow rate according to the governing guidance documents.

The verification frequency may be reduced after experienced is gained during the collection activities.

After the pilot phase, verify the flow rate at the beginning and at the end of each sampling event for each floor.

7. After the last location has been sampled and the final flow rate recorded as described in Step 5, turn the sampling pump off and seal each end of the cassette with a cassette end-plug. This can be done with either the sampling nozzle left in place (preferred) or removed. If the nozzle is removed it should also be sealed at both ends with an end-plug and placed in a separate zip-top bag for shipment to the laboratory. The nozzle is always saved and rinsed at the laboratory during sample preparation because a significant percentage of the dust drawn from a lightly loaded surface may adhere to the inside walls of the tubing.

8. Record the total elapsed sample collection time and total area sampled and other information as required according to governing guidance documents.

9. Wipe off the exterior surface of the cassette with disposable wet towel.

10. Place a sample label (index ID) on the cassette that clearly identifies the sample's unique identification number on the cassette.

11. Place a sample custody seal around both ends of the sampling cassette in a manner that does not obstruct the sample label.

12. Place each sample cassette in an individual plastic zip-top bag. Each bag should be labeled indicating the sample index ID. Do not put the sample cassette in a shirt or coat pocket as the filter can pick up fibers from clothing.

13. Decontaminate sampling equipment as required by the governing guidance document.

14. Transport the samples in a ridged container to the sample coordinator or designated recipient.

Dust field duplicate samples will be collected at the frequency required in the governing guidance documents. Field duplicate samples will be collected immediately adjacent to the locations of the parent sample. The duplicate will be collected from the same number of sub-samples as the parent sample, and be distributed across assess areas identically to the parent sample. For tracking purposes, the parent/duplicate sample relationship will be recorded in accordance with sample documentation requirements stated in the governing guidance document.

Section 6
Sample Custody and Shipment

Dust samples will be kept separate from other types of media sampled (i.e., soil, air, water, building materials, insulation, etc.) and should be transported in a ridged container until the field team can relinquish custody to the sample coordinator or designated recipient.

When dust samples are be shipped to on off-site analytical laboratory, a ridged sealed container will be used. Dust samples will be shipped separated from any other types of media. The cassettes must be tightly sealed and packaged in a material free of fibers or dust to minimize the potential for contamination. Plastic bubble wrap is an example of the appropriate material for this purpose. Examples of inappropriate materials are paper and packing peanuts.

Section 7
Documentation

As required by governing guidance documents, a field logbook/PDA will be maintained by each individual or team that is collecting samples as described in this SOP. The guidance documents will detail specific conditions which require attention and documentation, but at a minimum the following information should be collected:

- Project name
- Title of governing documents
- Property address
- Date
- Time
- Team members
- Weather conditions
- Locations of any samples or sub-samples that could not be acquired
- Descriptions of any deviations to the SOP or SAP and the reason for the deviation
- Relinquishment of samples to project sample coordinator or other recipient

In addition to logbook/PDA documentation, specifics regarding details of the sample collection will be recorded as required by governing guidance documents.

Section 8
Quality Assurance/Quality Control

8.1 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 manufacturer's instructions.

8.2 Collection of Field Quality Control Samples

Field quality control (QC) samples will consist of three types: lot blanks, field blanks, and field duplicates. The Site-Wide QAPP (CDM 2007) describes each of these samples, their corresponding acceptance criteria, and potential actions if acceptance criteria are not met. Governing guidance documents should be consulted to determine the required collection frequency for each sample type.

Section 9
Glossary

Governing guidance documents - The written document that spells out the detailed site-specific procedures to be followed by the project leader and the field personnel for completing specific investigations. These documents will clearly indicate specific requirements for the implementation of this SOP.

Sample Point - The actual location at which the dust sample is taken. The dimension of a sample point is 100 cm^2.

Composite Sampling - A sample program in which multiple sample points are compiled together and submitted for analysis as a single sample.

Libby Superfund Site – The Libby Superfund Site contains all buildings and land within the boundaries of each operable unit (OU) of the site and illustrated on the most recent version of the OU boundary map.

Section 10
References

ASTM. 2003. Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading. ASTM D5755-03.

CDM. 2007. Site-Wide Quality Assurance Project Plan. Draft in review.

Site-Specific Sampling Guidance
Libby Superfund Site

SOP No.: **CDM-LIBBY-06, Revision 1**

SOP Title: Semi-Quantitative Visual Estimation of Vermiculite in Soils at Residential and Commercial Properties

Approved by:

5/10/07
Technical Reviewer — Date

5/10/07
QA Reviewer — Date

05/10/07
Volpe Center Approval — Date

5/10/07
EPA Approval — Date

Section 1
Purpose

EPA will identify and delineate the extent of any visible vermiculite (VV) present in soils as part of all investigations conducted at the Libby Superfund Site and specified in governing guidance documents. The goal of this standard operating procedure (SOP) is to provide a consistent approach to identify and characterize any VV present in soils.

The semi-quantitative approach presented in this SOP for visually estimating VV in soil will be revised as required to optimize data collection as the sampling teams gain experience. This will be accomplished by expanding and/or improving this SOP, supporting pictorial standards, and additional electronic data acquisition efforts, as necessary.

Section 2
Definitions

Specific Use Area (SUA) – Discrete exterior parcels on a property with a designated specific use. Due to the nature of activities typically carried out in SUAs, residents may be especially vulnerable to exposures when Libby amphibole asbestos (LA) contaminated soil becomes airborne. SUAs may be bare or covered with varying amounts of vegetation. SUAs include:

- Flower Pot
- Flowerbed
- Garden
- Stockpile
- Play Area
- Dog Pen
- Driveway (non-paved)
- Parking Lot (non-paved)
- Road (non-paved)
- Alley (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:

- Walkway
- Yard (front, back, side, etc.)
- Former Garden
- Former Flowerbed

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:

- Pasture
- Maintained/Mowed Fields
- Underneath porches/decks[1]
- Overgrown Areas (with trails/footpaths, or between SUAs/CUAs)

Interior Surface Area (ISA) – Interior soil surfaces of buildings such as garages, pumphouses, sheds, and crawlspaces.

Non-Use Area (NUA) – Exterior parcels on a property with no current use (e.g., areas that are un-maintained and not accessed). NUAs may be bare or covered with varying amounts of vegetation. NUAs include:

- Wooded Lots
- Un-maintained Fields

Since NUAs are not currently accessed, they are not presently considered a complete exposure pathway. As such, semi-quantitative visual estimates of vermiculite in soil will not be captured at this time. However, to the extent that NUAs may become a complete exposure pathway in the future, EPA may revisit these NUAs at a later date.

Zone[2] – Parcels on a property that share a similar land use or subdivisions of a land use area based on site conditions (e.g., access, construction setup considerations, etc.) or sampling requirements. No area type may be combined with any other area type. For example, driveways and flowerbeds are both SUAs but will be separated into unique zones for visual inspection. Similarly, large CUAs such as yards may be subdivided into front yard, side yard, and back yard zones dependent on site conditions. Sectioning properties into additional zones will be at the discretion of the field team leader but consistent among the teams.

It is anticipated that SUAs and ISA zones will generally tend to be smaller parcels. Combining small, proximal SUAs into one zone will be at the discretion of the field team leader but consistent among teams. No ISA will be combined with any other ISA for visual inspection. There is not a maximum square footage restriction on any zone.

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[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.
[2] The restriction on the maximum square footage of SUA zones (1,000 ft2) and non-SUA zones (2, 500 ft2) was eliminated from the previous iteration of this SOP after the data were reviewed by EPA and determined to sufficiently characterize the presence of VV regardless of zone square footage. Additionally, this will allow the flexibility necessary for field teams to identify areas of zones most cost effectively for removal purposes.

Point Inspection (PI) – Used in SUA, CUA, LUA, and ISA zones. A PI is an intrusive visual inspection of the top portions of the soil at a randomly selected point within a zone. A PI consists of the active displacement of the surface soil with a small shovel and visual inspection of the displaced soil to determine if VV is present. If VV is observed during the PI, the location and a semi-quantitative estimate of VV contamination will be recorded.

Section 3
Applicability

This SOP applies to properties within the Libby Superfund Site at varying stages of the removal process including, but not limited to, all screening and risk-based investigations, pre-design inspections, and removal actions. Investigation-specific modifications to this SOP are outlined in the governing guidance document for each investigation. The following locations on a property will be evaluated for the presence/absence of VV:

- All parcels on a property where soil samples are being collected.
- All parcels on a property where soil was non-detect for LA during previous sampling activities.
- All SUA parcels on a property that have not been previously characterized as containing VV

Section 4
Procedure

Figure 1 illustrates the procedures and decision rules for this SOP. The three primary procedural steps are listed below:

- Establish zones
- Perform PI
- Perform semi-quantification of visual vermiculite

Each is described in the following subsections.

4.1 Establish Zones

Upon arrival at the property, the field team will locate all areas requiring sample collection (i.e., where previous soil sample results were non-detect for LA or SUAs have not been previously characterized for VV). Parcels will be identified as SUA zones, CUA zones, LUA zones, NUA zones, or ISA zones. The field team will measure the zone sizes and note them on the field sketch and/or design drawings. Zones will be assigned according to the definitions provided above.

4.2 Point Inspections[3]

As defined above, a PI is an intrusive visual inspection performed for the entire surface of a zone. Professional judgment may be used to determine the exact location of PIs; however, the following guidelines will be implemented to maintain consistency.

A minimum of 30 PIs will be evaluated per zone if sampling is required within that zone. If soil sampling is not required, a minimum of 5 PIs will be evaluated within each zone. Zones larger than 500 square feet (ft^2) will require evaluation at a minimum of 1 PI per 100 ft^2 (10 ft by 10 ft area). The PI locations will be randomly selected and will be spatially representative of the entire zone. Locations of the PIs and semi-quantitative estimates of VV (i.e., low, intermediate, or high) will be recorded on the field sketch for each PI. While a minimum of 5 PIs will be conducted per zone, there is no set maximum. Rather, the maximum number of PIs is variable—dependent upon the total area of the zone and achieving the minimum required frequency of 1 PI per 100 ft^2.

The following sections outline procedures for inspecting each use area (e.g., SUA, CUA, LUA, ISA). The procedure for semi-quantification of VV is provided in the next section.

<u>SUA Zone:</u>

- Visually inspect the PI point using a spade or trowel to remove any cover material, including excess debris (e.g., mulch, rock, etc.) and organic material, from the surface of the soil. Remove and visually inspect soil to a depth of 0-6 inches below ground surface[4].
- If a depth of 6 in. cannot be attained given the varying levels of compaction in driveways, roads, etc. the maximum depth attainable will be documented in the field logbook.
- Record semi-quantitative estimate of VV observed as described in the following section.
- Replace soil and cover material.
- Repeat as necessary employing procedure outlined above.

<u>CUA and LUA Zones:</u>

- Visually inspect the PI point using a spade or trowel, carefully removing organic material, including grass, from the surface of the soil. Remove and visually inspect soil to a depth of 0 - 3 inches below ground surface[5].

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[3] Surface Inspections- The non-intrusive visual inspection of the immediate surface of a zone was eliminated from the previous iteration of this SOP after their data were reviewed and determined by EPA to provide no additional information over that gained through Point Inspections.
[4] A soil depth of 6 inches for SUAs was chosen to approximate the depths to which digging would be expected during typical activities occurring in these SUA zones (e.g., gardening, child digging in dirt, etc.)
[5] A soil depth of 0-3 inches was chosen to approximate the depths to which soil disturbance would be most likely during typical activities occurring in these CUA and LUA zones (e.g., lawn mowing, etc.)

- Record semi-quantitative estimate of VV observed as described in the following section.
- Carefully replace all soil and organic material.
- Repeat as necessary employing procedure outlined above.

ISA Zone:

- Move items as necessary to access the soil surface.
- Visually inspect the PI points using a spade or trowel, remove and visually inspect soil to a depth of 0 - 3 inches below ground surface[6].
- Record semi-quantitative estimate of VV observed as described in the following section.
- Repeat as necessary employing procedure outlined above.

If during the PI, VV is observed to be localized within a zone, the portion with vermiculite will be denoted on the field sketch. If additional PIs are necessary to determine the boundaries of the area, approximately 10 to 20% additional PIs will be evaluated to determine the extent of localized vermiculite.

4.3 Semi-Quantification of Visual Vermiculite

During PI, the field team will estimate the quantity of vermiculite observed. Each PI location for all zones will be assigned a semi-quantitative estimate of visible vermiculite content using a 4-point scale: none (blank), low (L), intermediate (M), and high (H)[7]. For PI locations where VV is observed, semi-quantitative estimates (e.g., L, M, or H) will be recorded on the field sketch. PI locations where VV is not observed will not be recorded on the field sketch. Photographs illustrating these quantities are attached to this SOP as Figure 2. Additionally, jars of vermiculite-containing soils representing these three levels will be available for training and reference.

Under the current version of this SOP, there will be no effort to design an approach to combine vermiculite levels for PIs within or among zones. While the viability of combining semi-quantitative visual estimates within or among zones may be assessed as a pilot-scale evaluation, any PI with visible vermiculite qualifies as vermiculite-containing soil for the area represented by the inspection point or inspection zone.

[6] A soil depth of 0-3 inches was chosen to approximate the depths to which soil disturbance would be most likely during typical activities occurring in these IS zones (e.g., entering crawlspace, retrieving items from shed, etc.)

[7] Based on EPA's review of previous data, the 5-level scale VV identification scheme was not meaningful and will be reduced to a 4-level scale. As such the quantity of "Gross" VV in the previous iteration of this SOP was combined with High. Previously collected data of Gross VV should be considered analogous to High VV under this revised SOP.

Section 5
Health & Safety/Engineering Controls

All personnel will carry out visual inspections in accord with proper personal protective equipment (PPE) and other monitoring/governing requirements outlined in the most recent version of the Site Health and Safety Plan governing the work being conducted.

All visual inspections will employ appropriate engineering controls to minimize dust (e.g., wetting soil during inspection) as prescribed in the Site-Specific Standard Operating Procedure for Soil Sample Collection (CDM-LIBBY-05, Revision 2).

Section 6
Equipment Decontamination

Equipment decontamination is not required between each PI from the same zone, but is required before moving to another inspection zone. Decontamination of equipment will be conducted as required by the governing guidance documents.

Section 7
Documentation

As noted above, information about the presence of vermiculite will be recorded on the field sketch or design drawing for the property under investigation. Each zone will be marked with:

- Zone type (i.e., SUA, CUA, LUA, NUA, or ISA)
- Zone area in ft^2
- PI locations/points
- Semi-quantitative estimate of VV content for each PI (i.e., L, M, H)

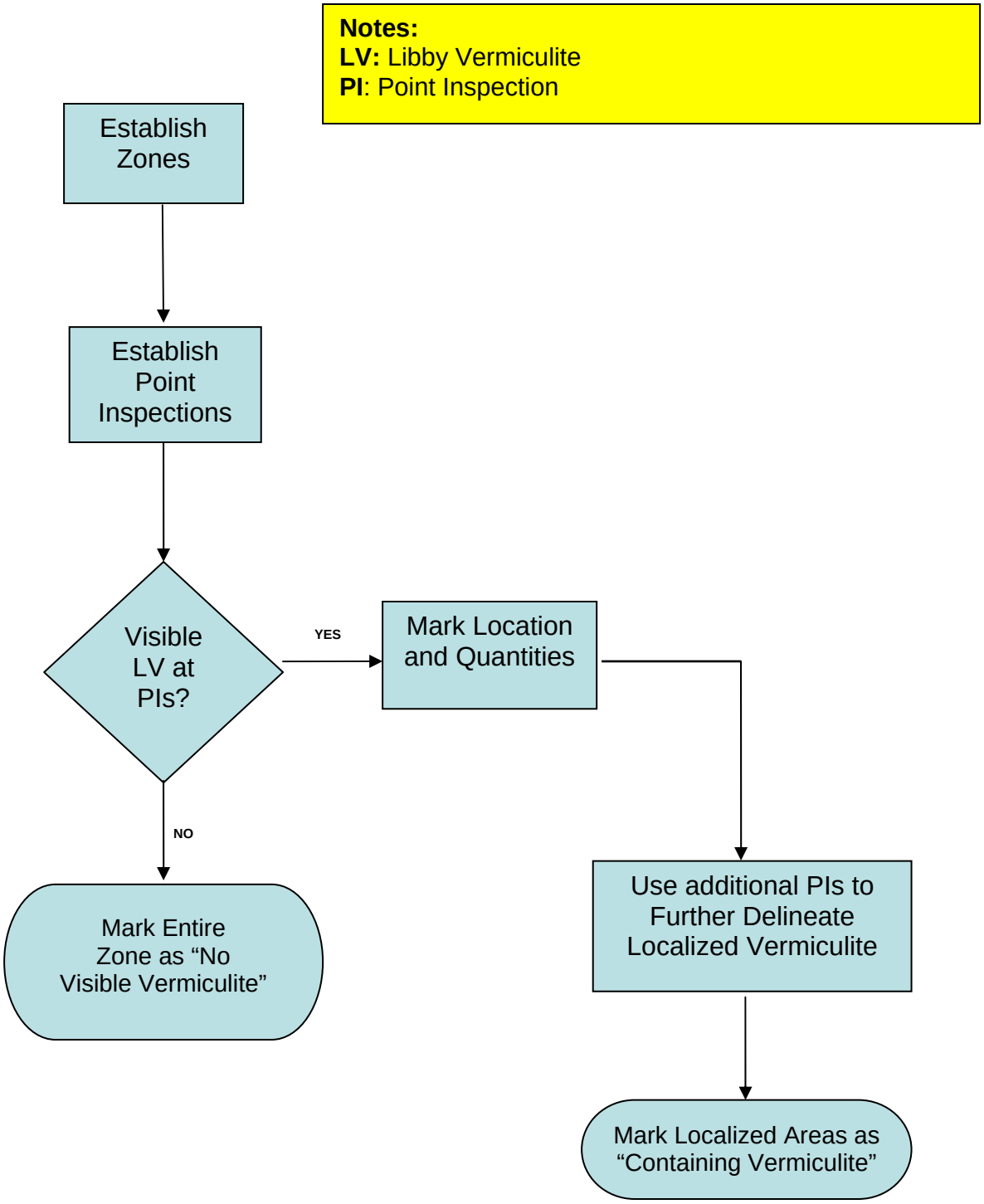
In addition to field sketch/design drawing documentation, each field team will generate a Visual Vermiculite Estimation Form (VVEF) (Figure 3) to document the semi-quantitative visual estimates of VV for each PI for possible future information use. This form will be managed according to governing guidance documents.

Section 8
Training

Every effort will be made to ensure consistency in the semi-quantitative evaluation of VV in soil to the extent possible. This will include training (e.g., field calibration), specimen examples (i.e., jars/photographs of low, intermediate, and high quantities of vermiculite, etc.), designated field staff, and oversight by the field team leader. Figures illustrating none, low, intermediate, and high quantities of vermiculite are attached to this SOP for reference (Figure 2).

To ensure consistency over time, the field team leader will verify semi-quantitative assignments at a rate of one property per team per week. The field team leader will sign off on those field sketches that were verified. If inconsistencies are noted, the field team leader will hold re-training with all teams participating simultaneously. Updates to the SOP and its attached specimen examples will occur as necessary and the EPA Project Team Leader and Technical Assistance Unit will be notified when these updates are recommended by the field team leader or field investigation manager.

Figure 1 – Visible Vermiculite Inspection Process



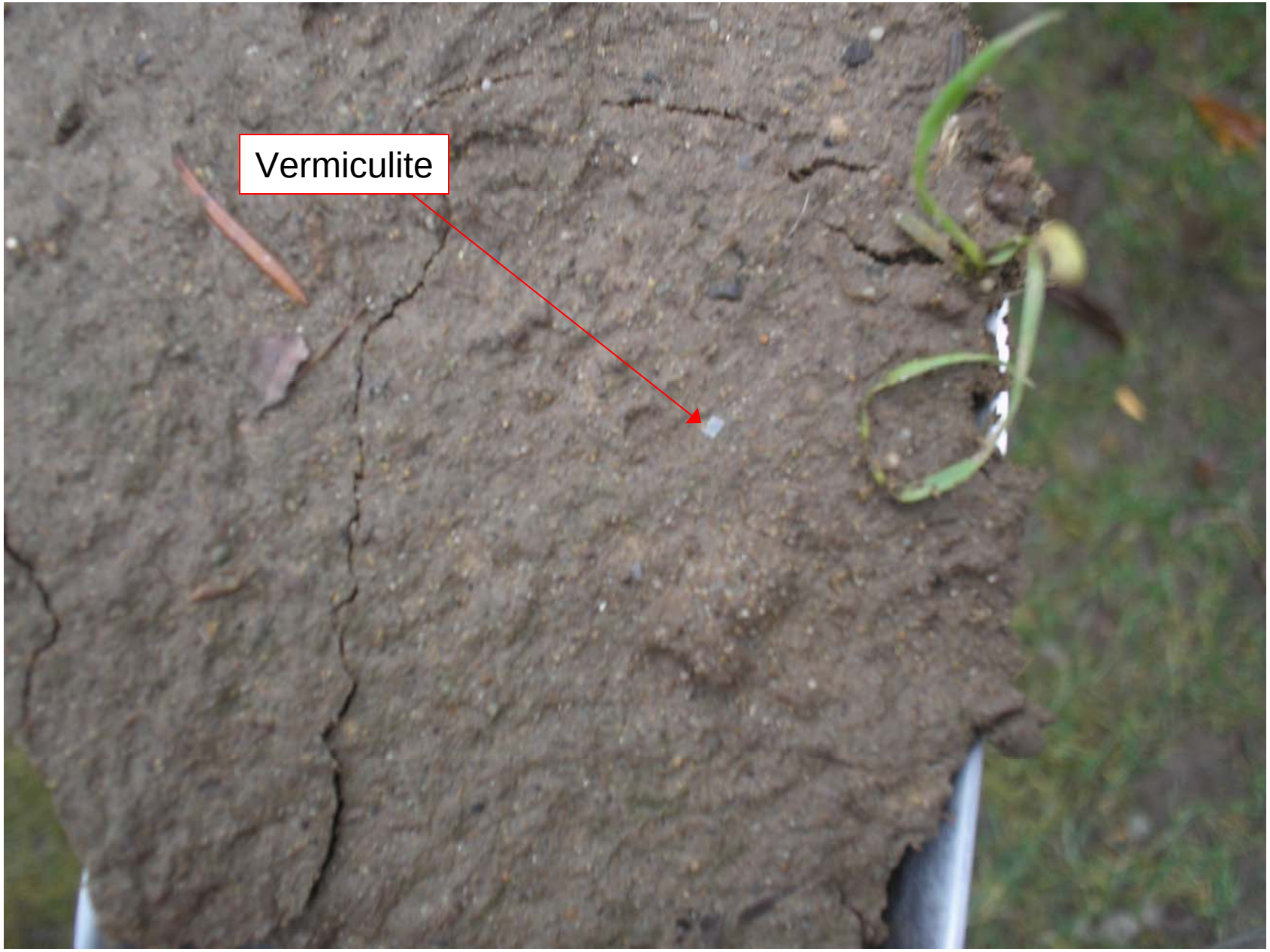


Figure 2a: Low Visible Vermiculite – A maximum of a few flakes of vermiculite observed within a given visual inspection point

Figure 2b: Intermediate Visible Vermiculite – Vermiculite easily observed throughout visual inspection point, including the surface.

Figure 2c: Intermediate Visible Vermiculite – Vermiculite easily observed throughout visual inspection point, including the surface.

Figure 2d: High Visible Vermiculite – Vermiculite easily observed throughout visual inspection point, including the surface.

LIBBY SUPERFUND SITE
Visual Vermiculite Estimation Form (VVEF)

Field Logbook No.: _____ Page No.: _____ Site Visit Date: _____ BD Number: _____

Address: _____ Structure Description: Property

Occupant: _____ Phone No.: _____

Owner (If different than occupant): _____ Phone No.: _____

Investigation Team: _____ Investigation Name: _____

Field Form Check Completed by (100% of Forms): _____ Visual Verification by Field Team Leader (10% of forms): _____

		Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6	Zone 7	Zone 8
Type (SUA/CUA/LUA/IS)									
Description									
Area Size (square feet)									
General Comment (Cover, etc.)									
PIs (X=None, L=Low, M=Intermediate, H=High)	**X**								
	L								
	M								
	H								
	Total	0	0	0	0	0	0	0	0

Areas previously identified for removal not inspected for visible vermiculite? **Yes** **No** **NA** **Location(s):** _____

Designation: D 5755 – 03

Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading[1]

This standard is issued under the fixed designation D 5755; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure to (*a*) identify asbestos in dust and (*b*) provide an estimate of the surface loading of asbestos in the sampled dust reported as the number of asbestos structures per unit area of sampled surface.

1.1.1 If an estimate of the asbestos mass is to be determined, the user is referred to Test Method D 5756.

1.2 This test method describes the equipment and procedures necessary for sampling, by a microvacuum technique, non-airborne dust for levels of asbestos structures. The non-airborne sample is collected inside a standard filter membrane cassette from the sampling of a surface area for dust which may contain asbestos.

1.2.1 This procedure uses a microvacuuming sampling technique. The collection efficiency of this technique is unknown and will vary among substrates. Properties influencing collection efficiency include surface texture, adhesiveness, electrostatic properties and other factors.

1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.

1.4 This test method is generally applicable for an estimate of the surface loading of asbestos structures starting from approximately 1000 asbestos structures per square centimetre.

1.4.1 The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregated asbestos into fundamental fibrils, fiber bundles, clusters, or matrices that can be more accurately quantified by transmission electron microscopy. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates nor destroys asbestos, but it may alter the physical form of the mineral fibers.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 3195 Practice for Rotameter Calibration[3]

D 3670 Guide for Determination of Precision and Bias of Methods of Committee D22[3]

D 5756 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Surface Loading[3]

D 6620 Practice for Determining a Detection Limit for Asbestos Measurements Based on Counts[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *asbestiform*—a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see Refs **(1)**,[4] **(2)** and **(3)**.

3.1.2 *asbestos*—a collective term that describes a group of naturally occurring, inorganic, highly fibrous, silicate dominated minerals, which are easily separated into long, thin, flexible fibers when crushed or processed.

[1] This test method is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

Current edition approved April 10, 2003. Published June 2003. Originally approved in 1995. Last previous edition approved in 2002 as D 5755 - 02.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] The boldface numbers in parentheses refer to the list of references at the end of this test method.

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3.1.2.1 *Discussion*—Included in the definition are the asbestiform varieties of: serpentine (chrysotile); riebeckite (crocidolite); grunerite (grunerite asbestos); anthophyllite (anthophyllite asbestos); tremolite (tremolite asbestos); and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to nomenclature of the International Mineralogical Association (3).

Asbestos	Chemical Abstract Service No.[5]
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Grunerite Asbestos	12172-73-5
Anthophyllite Asbestos	77536-67-5
Tremolite Asbestos	77536-68-6
Actinolite Asbestos	77536-66-4

3.1.3 *fibril*—a single fiber that cannot be separated into smaller components without losing its fibrous properties or appearance.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aspect ratio*—the ratio of the length of a fibrous particle to its average width.

3.2.2 *bundle*—a structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.

3.2.3 *cluster*—a structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers must have more than two points touching.

3.2.4 *debris*—materials that are of an amount and size (particles greater than 1 mm in diameter) that can be visually identified as to their source.

3.2.5 *dust*—any material composed of particles in a size range of <1 mm.

3.2.6 *fiber*—a structure having a minimum length of 0.5 µm, an aspect ratio of 5:1 or greater, and substantially parallel sides (4).

3.2.7 *fibrous*—of a mineral composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable. That is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance. The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.

3.2.8 *indirect preparation*—a method in which a sample passes through one or more intermediate steps prior to final filtration.

3.2.9 *matrix*—a structure in which one or more fibers, or fiber bundles that are touching, are attached to, or partially concealed by a single particle or connected group of nonfibrous particles. The exposed fiber must meet the fiber definition (see 3.2.6).

3.2.10 *structures*—a term that is used to categorize all the types of asbestos particles which are recorded during the analysis (such as fibers, bundles, clusters, and matrices). Final results of the test are always expressed in asbestos structures per square centimetre.

[5] The non-asbestiform variations of the minerals indicated in 5.1.3 have different Chemical Abstract Service (CAS) numbers.

4. Summary of Test Method

4.1 The sample is collected by vacuuming a known surface area with a standard 25 or 37 mm air sampling cassette using a plastic tube that is attached to the inlet orifice which acts as a nozzle. The sample is transferred from inside the cassette to an aqueous suspension of known volume. Aliquots of the suspension are then filtered through a membrane. A section of the membrane is prepared and transferred to a TEM grid using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using SAED and EDXA at a magnification of 15 000 to 20 000X.

5. Significance and Use

5.1 This microvacuum sampling and indirect analysis method is used for the general testing of non-airborne dust samples for asbestos. It is used to assist in the evaluation of dust that may be found on surfaces in buildings such as ceiling tiles, shelving, electrical components, duct work, carpet, etc. This test method provides an index of the surface loading of asbestos structures in the dust per unit area analyzed as derived from a quantitative TEM analysis.

5.1.1 This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings with asbestos-containing materials, or compliance with federal, state, or local regulations or statutes. It is the user's responsibility to make these determinations.

5.1.2 At present, no relationship has been established between asbestos-containing dust as measured by this test method and potential human exposure to airborne asbestos. Accordingly, the users should consider other available information in their interpretation of the data obtained from this test method.

5.2 This definition of dust accepts all particles small enough to pass through a 1 mm (No. 18) screen. Thus, a single, large asbestos containing particle(s) (from the large end of the particle size distribution) dispersed during sample preparation may result in anomalously large asbestos surface loading results in the TEM analyses of that sample. It is, therefore, recommended that multiple independent samples are secured from the same area, and that a minimum of three samples be analyzed by the entire procedure.

6. Interferences

6.1 The following minerals have properties (that is, chemical or crystalline structure) which are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials must be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 *Antigorite.*

6.1.2 *Palygorskite (Attapulgite).*

6.1.3 *Halloysite.*

6.1.4 *Pyroxenes.*

6.1.5 *Sepiolite.*

6.1.6 *Vermiculite scrolls.*

6.1.7 *Fibrous talc.*

6.1.8 Hornblende and other amphiboles other than those listed in 3.1.2.

6.2 Collecting any dust particles greater than 1 mm in size in this test method may cause an interference and, therefore, must be avoided.

7. Materials and Equipment

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.[6]

7.2 *Transmission Electron Microscope (TEM)*, an 80 to 120 kV TEM, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM must be equipped with energy dispersive X-ray spectroscopy (EDXA) and it must have a scanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover.

7.3 *Energy Dispersive X-ray System (EDXA).*

7.4 *High Vacuum Carbon Evaporator*, with rotating stage.

7.5 *High Efficiency Particulate Air (HEPA)*, filtered negative flow hood.

7.6 *Exhaust or Fume Hood.*

7.7 *Particle-free Water* (ASTM Type II, see Specification D 1193).

7.8 *Glass Beakers* (50 mL).

7.9 *Glass Sample Containers*, with wide mouth screw cap (200 mL) or equivalent sealable container (height of the glass sample container should be approximately 13 cm high by 6 cm wide).

7.10 *Waterproof Markers.*

7.11 *Forceps* (tweezers).

7.12 *Ultrasonic Bath*, table top model (100 W).

7.13 *Graduated Pipettes* (1, 5, 10 mL sizes), glass or plastic.

7.14 *Filter Funnel*, either 25 mm or 47 mm, glass or disposable. Filter funnel assemblies, either glass or disposable plastic, and using either a 25 mm or 47 mm diameter filter.

7.15 *Side Arm Filter Flask*, 1000 mL.

7.16 *Mixed Cellulose Ester (MCE) Membrane Filters*, 25 or 47 mm diameter, ≤0.22 µm and 5 µm pore size.

7.17 *Polycarbonate (PC) Filters*, 25 or 47 mm diameter, ≤0.2 µm pore size.

7.18 *Storage Containers*, for the 25 or 47 mm filters (for archiving).

7.19 *Glass Slides*, approximately 76 by 25 mm in size.

7.20 *Scalpel Blades*, No. 10, or equivalent.

7.21 *Cabinet-type Desiccator*, or low temperature drying oven.

7.22 *Chloroform*, reagent grade.

7.23 *Acetone*, reagent grade.

7.24 *Dimethylformamide (DMF).*

7.25 *Glacial Acetic Acid.*

7.26 *1-methyl-2-pyrrolidone.*

7.27 *Plasma Asher*, low temperature.

7.28 *pH Paper.*

7.29 *Air Sampling Pump*, low volume personal-type, capable of achieving a flow rate of 1 to 5 L/min.

7.30 *Rotameter.*

7.31 *Air Sampling Cassettes*, 25 mm or 37 mm, containing 0.8µ m or smaller pore size MCE or PC filters.

7.32 *Cork Borer*, 7 mm.

7.33 *Non-Asbestos Mineral*, references as outlined in 6.1.

7.34 *Asbestos Standards*, as outlined in 3.1.2.

7.35 *Tygon*[7] *Tubing*, or equivalent.

7.36 *Small Vacuum Pump*, that can maintain a pressure of 92 kPa.

7.37 *Petri Dishes*, large glass, approximately 90 mm in diameter.

7.38 *Jaffe Washer*, stainless steel or aluminum mesh screen, 30 to 40 mesh, and approximately 75 mm by 50 mm in size.

7.39 *Copper TEM Finder Grids*, 200 mesh.

7.40 *Carbon Evaporator Rods.*

7.41 *Lens Tissue.*

7.42 *Ashless Filter Paper Filters*, 90 mm diameter.

7.43 *Gummed Paper Reinforcement Rings.*

7.44 *Wash Bottles*, plastic.

7.45 *Reagent Alcohol*, HPLC Grade (Fisher A995 or equivalent).

7.46 *Opening Mesh Screen*, plastic, 1.0 by 1.0 mm, (Spectra-Mesh #146410 or equivalent).

7.47 *Diffraction Grating Replica.*

8. Sampling Procedure for Microvacuum Technique

8.1 For sampling asbestos-containing dust in either indoor or outdoor environments, commercially available cassettes must be used. Air monitoring cassettes containing 25 mm or 37 mm diameter mixed cellulose ester (MCE) or polycarbonate (PC) filter membranes with a pore size less than or equal to 0.8 µm are required (7.31). The number of samples collected depends upon the specific circumstances of the study.

8.2 Maintain a log of all pertinent sampling information and sampling locations.

8.3 Sampling pumps and flow indicators shall be calibrated using a certified standard apparatus or assembly (see Practice D 3195 and 7.29).

8.4 Record all calibration information **(5)**.

8.5 Perform a leak check of the sampling system at each sampling site by activating the pump (7.29) with the closed sampling cassette in line. Any air flow shows that a leak is present that must be eliminated before initiating the sampling operation.

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[7] Tygon is a registered trademark of the DuPont Co.

8.6 Attach the sampling cassette to the sampling pump at the outlet side of the cassette with plastic tubing (7.35). The plastic tubing must be long enough in that the sample areas can be reached without interference from the sampling pump. Attach a clean, approximately 25.4 mm long piece of plastic tubing (6.35 mm internal diameter) directly to the inlet orifice. Use this piece of tubing as the sampling nozzle. Cut the sampling end of the tubing at a 45° angle as illustrated in Fig. 1. The exact design of the nozzle is not critical as long as some vacuum break is provided to avoid simply pushing the dust around on the surface with the nozzle rather than vacuuming it into the cassette. The internal diameter of the nozzle and flow rate of the pump may vary as long as the air velocity is 100 (±10) cm/s. This air velocity calculation is based on an internal sampling tube diameter of 6.35 mm at a flow rate of 2 L/min.

8.7 Measure and determine the sample area of interest. A sample area of 100 cm^2 is vacuumed until there is no visible dust or particulates matter remaining. Perform a minimum of two orthogonal passes on the surface within a minimum of 2 min of sampling time. Avoid scraping or abrading the surface being sampled. (Do not sample any debris or dust particles greater than 1 mm in diameter (see 4.2).) Smaller or larger areas can be sampled, if needed. For example, some surfaces of interest may have a smaller area than 100 cm^2. Less dusty surfaces may require vacuuming of larger areas. Unlike air samples, the overloading of the cassettes with dust will not be a problem. As defined in 3.2.5, only dust shall be collected for this analysis.

8.8 At the end of sample collection, invert the cassette so that the nozzle inlet faces up before shutting off the power to the pump. The nozzle is then sealed with a cassette end-plug and the cassette/nozzle taped or appropriately packaged to prevent separation of the nozzle and cassette assembly. A second option is the removal of the nozzle from the cassette, then plugging of the cassette and shipment of the nozzle (also plugged at both ends) sealed in a separate closeable plastic bag. A third option is placing the nozzle inside the cassette for shipment. The nozzle is always saved and rinsed because a significant percentage of the dust drawn from a lightly loaded surface may adhere to the inside walls of the tubing.

8.9 Check that all samples are clearly labeled, that all dust sampling information sheets are completed, and that all pertinent information has been enclosed, in accordance with laboratory quality control practices, before transfer of the samples to the laboratory. Include an unused cassette and nozzle as a field blank.

8.10 Wipe off the exterior surface of the cassettes with disposable wet towels (baby wipes) prior to packaging for shipment.

9. Sample Shipment

9.1 Ship dust samples to an analytical laboratory in a sealed container, but separate from any bulk or air samples. The cassettes must be tightly sealed and packed in a material free of fibers or dust to minimize the potential for contamination. Plastic "bubble pack" is probably the most appropriate material for this purpose.

10. Sample Preparation

10.1 Under a negative flow HEPA hood (7.5), carefully wet-wipe the exterior of the cassettes to remove any possible contamination before taking cassettes into a clean preparation area.

10.2 Perform sample preparation in a clean facility that has a separate work area from both the bulk and air sample preparation areas.

10.3 Initial specimen preparation shall take place in a clean HEPA filtered negative pressure hood to avoid any possible contamination of the laboratory or personnel, or both, by the potentially large number of asbestos structures in an asbestos-containing dust sample. Cleanliness of the preparation area hoods is measured by the cumulative process blank surface loadings (see Section 11).

10.4 All sample preparation steps 10.4.1-10.4.6 shall take place in the dust preparation area inside a HEPA hood.

10.4.1 Remove the upper plug from the sample cassette and carefully introduce approximately 10 mL solution of a 50/50 mixture of particle-free water and reagent alcohol into the cassette using a plastic wash bottle (7.44). If the plugged nozzle was left attached to the cassette, then remove the plug and introduce the water/alcohol solution into the cassette through the tubing, and then remove the tubing, if it is visibly clean.

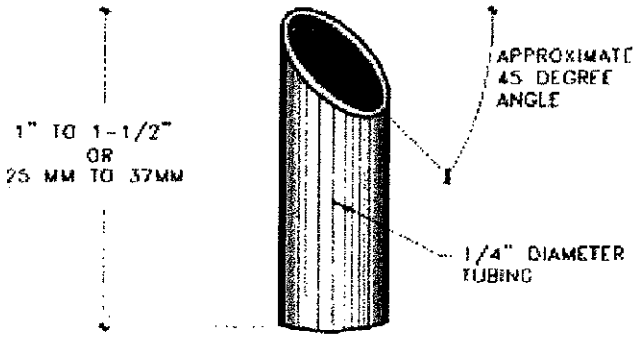

FIG. 1 Example of the Tubing Nozzle

10.4.2 Replace the upper plug or the sample cap and lightly shake the dust solution by hand for 3 s.

10.4.3 Remove the entire cap of the cassette and pour the suspension through a 1.0 by 1.0 mm opening screen (7.46) into a pre-cleaned 200 mL glass specimen bottle (7.9). All visible traces of the sample contained in the cassette shall be rinsed through the screen into the specimen bottle with a plastic wash bottle containing the 50/50 solution of particle-free water and alcohol. Repeat this procedure two additional times for a total of three washings. Next, rinse the nozzle two or three times through the screen into the specimen bottle with the 50/50 mixture of water and alcohol. Typically, the total amount of the 50/50 mixture used in the rinse is 50 to 75 mL. Discard the 1.0 by 1.0 mm screen and bring the volume of suspension in the specimen bottle up to the 100 mL mark on the side of the bottle with particle-free water only.

10.4.4 Adjust the pH of the suspension to 3 to 4 using a 10.0 % solution of acetic acid. Use pH paper for testing. Filter the suspension within 24 h to avoid problems associated with bacterial and fungal growth.

10.4.5 Use either a disposable plastic filtration unit or a glass filtering unit (7.14) for filtration of aliquots of the suspension. The ability of an individual filtration unit to produce a uniform distribution may be tested by the filtration of a colored particulate solution such as diluted India ink (solution of carbon black).

10.4.5.1 If a disposable plastic filtration unit is used, then unwrap a new disposable plastic filter funnel unit (either 25 or 47 mm diameter) and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus, retaining the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000 mL side-arm vacuum flask (7.15). Place a 5.0 μ m pore size MCE (backing filter) on the support pad. Wet it with a few mL of particle-free water and place an MCE (7.16) or PC filter (≤0.22 μm pore size) (7.17) on top of the backing filter. Apply a vacuum (7.36), ensuring that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface requires the discard of that filter. After the filter has been seated properly, replace the funnel and reseal it with the tape. Return the flask to atmospheric pressure.

10.4.5.2 If a glass filtration unit is used, place a 5 μm pore size MCE (backing filter) on the glass frit surface. Wet the filter with particle-free water, and place an MCE or PC filter (≤0.22 μm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface. Before filtration of each set of sample aliquots, prepare a blank filter by filtration of 50 mL of particle-free water. If aliquots of the same sample are filtered in order of increasing surface loading, the glass filtration unit need not be washed between filtration. After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual solution from the filtration assembly by holding it under a flow of water, then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse two times in particle-free water.

10.4.6 With the flask at atmospheric pressure, add 20 mL of particle-free water into the funnel. Cover the filter funnel with its plastic cover if the disposable filtering unit is used.

10.4.7 Briefly hand shake (3 s) the capped bottle with the sample suspension, then place it in a tabletop ultrasonic bath (7.12) and sonicate for 3.0 min. Maintain the water level in the sonicator at the same height as the suspension in sample bottle. The ultrasonic bath shall be calibrated as described in 20.5. The ultrasonic bath must be operated at equilibrium temperature. After sonicating, return the sample bottle to the work surface of the HEPA hood. Preparation steps 10.4.8-10.4.14 shall be carried out in this hood.

10.4.8 Shake the suspension lightly by hand for 3 s, then let it rest for 2.0 min to allow large particles to settle to the bottom of the bottle or float to the surface.

10.4.9 Estimate the amount of liquid to be withdrawn to produce an adequate filter preparation. Experience has shown that a light staining of the filter surface will yield a suitable preparation for analysis. Filter at least 1.0 mL, but no more than half the total volume. If after examination in the TEM, the smallest volume measured (1.0 mL) (7.13) yields an overloaded sample, then perform additional serial dilutions of the suspension. If it is estimated that less than 1.0 mL of suspension has to be filtered because of the density of the suspension, perform a serial dilution.

10.4.9.1 If serial dilutions are required, repeat step 10.4.8 before the serial dilution portion is taken. Do not re-sonicate the original suspension or any serial dilutions. The recommended procedure for a serial dilution is to mix 10 mL of the sample suspension with 90 mL of particle-free water in a clean sample bottle to obtain a 1:10 serial dilution. Follow good laboratory practices when performing dilutions.

10.4.10 Insert a new disposable pipette halfway into the sample suspension and withdraw a portion. Avoid pipetting any of the large floating or settled particles. Uncover the filter funnel and dispense the mixture from the pipette into the water in the funnel.

10.4.11 Apply vacuum to the flask and draw the mixture through the filter.

10.4.12 Discard the pipette.

10.4.13 Disassemble the filtering unit and carefully remove the sample filter with fine tweezers (7.11). Place the completed sample filter particle side up, into a precleaned, labeled, disposable, plastic petri dish (7.48) or other similar container.

10.4.14 In order to ensure that an optimally-loaded filter is obtained, it is recommended that filters be prepared from several different aliquots of the dust suspension. For this series of filters, it is recommended that the volume of each aliquot of the original suspension be a factor of five higher than the previous one. If the filters are prepared in order of increasing aliquot volume, all of the filters for one sample can be prepared using one plastic disposable filtration unit, or without cleaning of glass filtration equipment between individual filtration. Before withdrawal of each aliquot from the sample, shake the suspension without additional sonification and allow to rest for 2 min.

10.4.15 There are many practical methods for drying MCE filters. The following are two examples that can be used: (*1*) dry MCE filters for at least 12 h (over desiccant) in an airtight cabinet-type desiccator (7.21); (*2*) to shorten the drying time (if desired), remove a plug of the damp filter and attach it to a glass slide (7.19) as described in 12.1.2 and 12.1.3. Place the slide with a filter plug or filter plugs (up to eight plugs can be attached to one slide) on a bed of desiccant, in the desiccator for 1 h.

10.4.16 PC filters do not require lengthy drying before preparation, but shall be placed in a desiccator for at least 30 min before preparation.

10.5 Prepare TEM specimens from small sections of each dried filter using the appropriate direct transfer preparation method.

11. Blanks

11.1 Prepare sample blanks that include both a process blank (50 mL of particle-free water) for each set of samples analyzed and one unused filter from each new box of sample filters (MCE or PC) used in the laboratory. If glass filtering units are used, prepare and analyze a process blank each time the filtering unit is cleaned. Blanks will be considered contaminated, if after analysis, they are shown to contain more than 53 asbestos structures per square millimetre. This generally corresponds to three or four asbestos structures found in ten grid openings. The source of the contamination must be found before any further analysis can be performed. Reject samples that were processed along with the contaminated blanks and prepare new samples after the source of the contamination is found.

11.2 Prepare field blanks which are included with sample sets in the same manner as the samples, to test for contamination during the sampling, shipping, handling, and preparation steps of the method.

12. TEM Specimen Preparation of Mixed Cellulose Ester (MCE) Filters

NOTE 1—Use of either the acetone or the diamethylformamide-acetic acid method is acceptable.

12.1 *Acetone Fusing Method*:

12.1.1 Remove a section (a plug) from any quadrant of the sample and blank filters. Sections can be removed from the filters using a 7 mm cork borer (7.32). The cork borer must be wet wiped after each time a section is removed.

12.1.2 Place the filter section (particle side up) on a clean microscope slide. Affix the filter section to the slide with a gummed page reinforcement (7.43), or other suitable means. Label the slide with a glass scribing tool or permanent marker (7.10).

12.1.3 Prepare a fusing dish from a glass petri dish (7.37) and a metal screen bridge (7.38) with a pad of five to six ashless paper filters (7.42) and place in the bottom of the petri dish (**4**). Place the screen bridge on top of the pad and saturate the filter pads with acetone. Place the slide on top of the bridge in the petri dish and cover the dish. Wait approximately 5 min for the sample filter to fuse and clear.

12.2 *Dimethylformamide-Acetic Acid Method*:

12.2.1 Place a drop of clearing solution that consists of 35 % dimethylformamide (DMF), 15 % glacial acetic acid, and 50 % Type II water (v/v) on a clean microscope slide. Gauge the amount used so that the clearing solution just saturates the filter section.

12.2.2 Carefully lay the filter segment, sample surface upward, on top of the solution. Bring the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any excess clearing solution. Place the slide in an oven or on a hot plate, in a fume hood, at 65 to 70°C for 10 min.

12.3 Plasma etching of the collapsed filter is required.

12.3.1 The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher (7.27). Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the exact conditions that must be used. Insufficient etching will result in a failure to expose embedded fibers, and too much etching may result in the loss of particles from the filter surface. To determine the optimum time for ashing, place an unused 25 mm diameter MCE filter in the center of a glass microscope slide. Position the slide approximately in the center of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 to 20 cm^3/min. Adjust the tuning of the system so that the intensity of the plasma is maximized. Determine the time required for complete oxidation of the filter. Adjust the system parameters to achieve complete oxidation of the filter in a period of approximately 15 min. For etching of collapsed filters, use these operating parameters for a period of 8 min. For additional information on calibration, see the *USEPA Asbestos-Containing Materials in Schools* (**4**) or *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* (**6**) documents.

12.3.2 Place the glass slide containing the collapsed filters into the low-temperature plasma asher, and etch the filter.

12.4 Carbon coating of the collapsed and etched filters is required.

12.4.1 Carbon coating must be performed with a high-vacuum coating unit (7.4), capable of less than 10^{-4} torr (13 MPa) pressure. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and shall not be used. Carbon rods (7.40) used for evaporators shall be sharpened with a carbon rod sharpener to a neck of about 4 mm in length and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 100 to 120 mm from the surface of the microscope slide held in the rotating device.

12.4.2 Place the glass slide holding the filters on the rotation device, and evacuate the evaporator chamber to a vacuum of at least 13 MPa. Perform the evaporation in very short bursts, separated by 3 to 4 s to allow the electrodes to cool. An alternate method of evaporation is by using a slow continuous applied current. An experienced analyst can judge the thickness of the carbon film to be applied. Conduct tests on unused filters first. If the carbon film is too thin, large particles will be lost

from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen.

12.4.2.1 If the coating is too thick, it will lead to a TEM image that is lacking in contrast, and the ability to obtain electron diffraction patterns will be compromised. The carbon film shall be as thin as possible and still remain intact on most of the grid openings of the TEM specimen.

12.5 *Preparation of the Jaffe Washer*— The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used (**7, 8**). One such washer consists of a simple stainless steel bridge contained in a glass petri dish.

12.5.1 Place several pieces of lens tissue (7.41) on the stainless steel bridge. The pieces of lens tissue shall be large enough to completely drape over the bridge and into the solvent. In a fume hood, fill the petri dish with acetone (or DMF) until the height of the solvent is brought up to contact the underside of the metal bridge as illustrated in Fig. 2.

12.6 *Placing the Specimens into the Jaffe Washer*:

12.6.1 Place the TEM grids (7.39) shiny side up on a piece of lens tissue or filter paper so that individual grids can be easily picked up with tweezers.

12.6.2 Prepare three grids from each sample.

12.6.2.1 Using a curved scalpel blade (7.20), excise at least two square (3 mm by 3 mm) pieces of the carbon-coated MCE filter from the glass slide.

12.6.2.2 Place the square filter piece carbon-side up on top of a TEM specimen grid.

12.6.2.3 Place the whole assembly (filter/grid) on the saturated lens tissue in the Jaffe washer.

12.6.2.4 Place the three TEM grid sample filter preparations on the same piece of lens tissue in the Jaffe washer.

12.6.2.5 Place the lid on the Jaffe washer and allow the system to stand for several hours.

12.7 Alternately, place the grids on a low level (petri dish filled to the ⅛ mark) DMF Jaffe washer for 60 min. Add enough solution of equal parts DMF/acetone to fill the washer to the screen level. Remove the grids after 30 min if they have cleared, that is, all filter material has been removed from the carbon film, as determined by inspection in the TEM.

12.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean marked grid box.

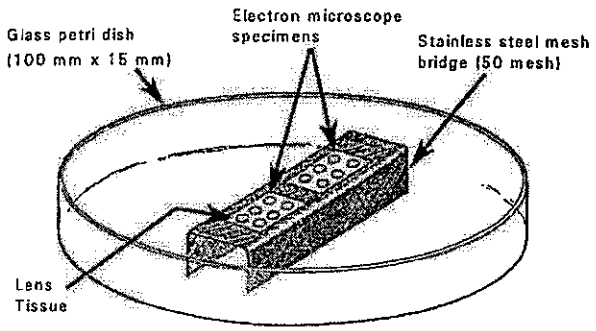

FIG. 2 Example of Design of Solvent Washer (Jaffe Washer)

13. TEM Specimen Preparation of Polycarbonate (PC) Filter

13.1 Cover the surface of a clean microscope slide with two strips of double-sided adhesive tape.

13.2 Cut a strip of filter paper slightly narrower than the width of the slide. Position the filter paper strip on the center of the length of the slide.

13.3 Using a clean, curved scalpel blade, cut a strip of the PC filter approximately 25 by 6 mm. Use a rocking motion of the scalpel blade to avoid tearing the filter. Place the PC strip particle side up on the slide perpendicular to the long axis of the slide. The ends of the PC strip must contact the double sided adhesive tape. Each slide can hold several PC strips. With a glass marker, label each PC strip with the individual sample number.

13.4 Carbon coat the PC filter strips as discussed in 12.4.2. PC filters do not require etching.

NOTE 2—**Caution:** Do not overheat the filter sections while carbon coating.

13.5 Prepare a Jaffe washer as described in 12.5, but fill the washer with chloroform or 1-methyl-2-pyrrolidone to the level of the screen.

13.6 Using a clean curved scalpel blade, excise three, 3-mm square filter pieces from each PC strip. Place the filter squares carbon side up on the shiny side of a TEM grid. Pick up the grid and filter section together and place them on the lens tissue in the Jaffe washer.

13.7 Place the lid on the Jaffe washer and rest the grids in place for at least 4 h. Best results are obtained with longer wicking times, up to 12 h.

13.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean, marked grid box.

14. Grid Opening Measurements

14.1 TEM grids must have a known grid opening area. Determine this area as follows:

14.2 Measure at least 20 grid openings in each of 20 random 75 to 100 μm (200-mesh) copper grids for a total of 400 grid openings for every 1000 grids used, by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

14.3 Grid area measurements can also be made at the TEM at a calibrated screen magnification of between 15 000 and 20 000X. Typically measure one grid opening for each grid examined. Measure grid openings in both the x and y directions and calculate the area.

14.4 Pre-calibrated TEM grids are also acceptable for this test method.

15. TEM Method

15.1 Microscope settings: 80 to 120 kV, 15 000 to 20 000X screen magnification for analysis (7.2).

15.2 Analyze two grids for each sample. Analyze one-half of the sample area on one sample grid preparation and the remaining half on a second sample grid preparation.

15.3 *Determination of Specimen Suitability*:

15.3.1 Carefully load the TEM grid, carbon side facing up (in the TEM column) with the grid bars oriented parallel/perpendicular to the length of the specimen holder. Use a hand lens or loupe, if necessary. This procedure will line up the grid with the x and y translation directions of the microscope. Insert the specimen holder into the microscope.

15.3.2 Scan the entire grid at low magnification (250X to 1000X) to determine its suitability for high magnification analysis as specified in 15.3.3.

15.3.3 Grids are acceptable for analysis if the following conditions are met:

15.3.3.1 The fraction of grid openings covered by the replica section is at least 50 %.

15.3.3.2 Relative to that section of the grid covered by the carbon replica, the fraction of intact grid openings is greater than 50 %.

15.3.3.3 The fractional area of undissolved filter is less than 10 %.

15.3.3.4 The fraction of grid openings with overlapping or folded replica film is less than 50 %.

15.3.3.5 At least 20 grid openings, that have no overlapping or folded replica, are less than 5 % covered with holes and have less than 5 % opaque area due to incomplete filter dissolution.

15.4 *Determination of Grid Opening Suitability*:

15.4.1 If the grid meets acceptance criteria, choose a grid opening for analysis from various areas of the grid so that the entire grid is represented. Determine the suitability of each individual grid opening prior to the analysis.

15.4.2 The individual grid opening must have less than 5 % holes over its area.

15.4.3 Grid openings must be less than 25 % covered with particulate matter.

15.4.4 Grid openings must be uniformly loaded.

15.5 Observe and record the orientation of the grid at 80 to 150X, on a grid map record sheet along with the location of the grid openings that are examined for the analysis. If indexed grids are used, a grid map is not required, but the identifying coordinates of the grid square must be recorded.

16. Recording Data Rules

16.1 Record on the count sheet any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 5.2.

16.2 Use the criteria for fiber, bundle, cluster, and matrix identification, as described in the *USEPA Asbestos-Containing Materials in Schools* document (4). Record, for each AHERA structure identified, the length and width measurements.

16.3 Record NSD (No Structures Detected) when no structures are detected in the grid opening.

16.4 Identify structures classified as chrysotile identified by either electron diffraction or X-ray analysis (7.3) and recorded on a count sheet. Verify at least one out of every ten chrysotile structures by X-ray analysis.

16.5 Structures classified as amphiboles by X-ray analysis and electron diffraction are recorded on the count sheet. For more information on identification, see Yamate, et al, (7) or Chatfield and Dillon (8).

16.6 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.

16.7 *Counting Rules*:

16.7.1 At a screen magnification of between 15 000 and 20 000X evaluate the grids for the most concentrated sample loading; reject the sample if it is estimated to contain more than 50 asbestos structures per grid opening. Proceed to the next lower concentrated sample until a set of grids are obtained that have less than 30 asbestos structures per grid opening.

16.8 *Analytical Sensitivity (AS)*—As determined by the following equation:

$$(EFA \times 100\ mL \times 1)/(GO \times GOA \times V \times SPL) = AS \qquad (1)$$

where:

EFA = effective filter area of the final sampling filter, mm^2
GO = number of grid openings counted
GOA = average grid opening area, mm^2
SPL = surface area sampled, cm^2
V = volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL
AS = analytical sensitivity, expressed as asbestos structures/cm^2

16.8.1 An AS of approximately 1000 asbestos structures per square centimetre (calculated for the detection of a single asbestos structure) has been designed for this analysis. This sensitivity can be achieved by increasing the amount of liquid filtered, increasing the number of grid openings analyzed, increasing the area of the collection, or decreasing the size of the final filter. For example, using a collection area = 500 cm^2, filter area = 1000 mm^2, number of grid openings = 10, and a grid area of 0.01 mm^2, V = 50 mL, the AS is 40 str/cm^2. Occasionally, due to high particle loadings or high asbestos surface loading, this AS cannot be practically achieved and stopping rules apply.

16.8.2 The numerical value of AS required for any specific application of this method may be achieved by selecting an appropriate combination of the sampling and analysis parameters in the above equation. For example, if SPL = 100 cm^2, EFA = 1000 mm^2, GO = 10, GOA = 0.01 mm^2, V = 10 mL, and D = 1, then AS = 1000 str/cm^2. Increasing GO to 50 and V to 50 mL changes the AS to 40 Str/cm^2.

16.8.3 When sample filters are heavily loaded with particulate matter, it may useful to employ serial dilutions during preparation to reduce the loading on grid specimens to acceptable levels and thus facilitate analysis. Under such circumstances, the AS may be calculated by substituting an appropriate value for the dilution factor, D, into the above equation. In general:

$$D = VA/(V + VPFW) \quad (2)$$

VA = the volume of the aliquot from the new, diluted suspension that is filtered to prepare the analytical filter; V = the volume of the aliquot from the initial (100 mL) suspension that is diluted; and VPFW = the volume of particle free water added to V during serial dilution to produce the new, diluted suspension.

Thus, if GO = 10, V = 10 mL, VPFW = 90 mL, and VA = 1.0 mL, D = 0.01 and the AS = 100 000 str/cm^2.

16.9 *Limit of Detection*—The limit of detection for this test method is calculated using the Practice D 6620. All data shall be provided in the laboratory report.

16.10 *Stopping Rules*:

16.10.1 The analysis is stopped upon the completion of the grid square that achieves an AS of less than 1000 asbestos structures per square centimetre.

16.10.2 If an AS of 1000 asbestos structures per square centimetre cannot be achieved after analyzing ten grid openings then stop on grid opening No. 10 or the grid opening which contains the 100th asbestos structure, whichever comes first. A minimum of four grid squares shall be analyzed for each sample.

16.10.2.1 If the analysis is stopped because of the 100th structure rule, the entire grid square containing the 100th structure must be counted.

16.11 After analysis, remove the grids from the TEM, and replace them in the appropriate grid storage holder.

17. Sample Storage

17.1 The washed-out sample cassettes can be discarded after use.

17.2 Sample grids and unused filter sections (7.18) must be stored for a minimum of one year.

18. Reporting

18.1 Report the following information for each dust sample analyzed:

18.1.1 Surface loading in structures/cm^2.

18.1.2 The AS.

18.1.3 Types of asbestos present.

18.1.4 Number of asbestos structures counted.

18.1.5 Effective filtration area.

18.1.6 Average size of the TEM grid openings that were counted.

18.1.7 Number of grid openings examined.

18.1.8 Sample dilution used.

18.1.9 Area of the surface sampled.

18.1.10 Listing of size data for each structure counted.

18.1.11 A copy of the TEM count sheet or a complete listing of the raw data. An example of a typical count sheet is shown in Appendix X1.

18.2 Determine the amount of asbestos in any accepted sample using the following formula:

$$\frac{EFA \times 100\ \text{mL} \times \#STR}{GO \times GOA \times V \times SPL} = \text{asbestos structures/cm}^2 \quad (3)$$

where:

#STR = number of asbestos structures counted,
EFA = effective filter area of the final sampling filter, mm^2,
GO = number of grid openings counted,
GOA = average grid opening area, mm^2,
SPL = surface area sampled, cm^2, and
V = volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL.

19. Quality Control/Quality Assurance

19.1 In general, the laboratory's quality control checks are used to verify that a system is performing according to specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. However, due to the difficulties in preparing known quantitative asbestos samples, routine quality control testing focuses on re-analysis of samples (duplicate recounts).

19.1.1 Re-analyze samples at a rate of 1/10 of the sample sets (one out of every ten samples analyzed not including laboratory blanks). The re-analysis shall consist of a second sample preparation obtained from the final filter.

19.2 In addition, quality assurance programs must follow the criteria shown in the *USEPA Asbestos-Containing Materials in Schools* document **(4)** and in the *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* document **(6)**. These documents describe sample custody, sample preparation, blank checks for contamination, calibration, sample analysis, analyst qualifications, and technical facilities.

20. Calibrations

20.1 Perform calibrations of the instrumentation on a regular basis, and retain these records in the laboratory, in accordance with the laboratory's quality assurance program.

20.2 Record calibrations in a log book along with dates of calibration and the attached backup documentation.

20.3 A calibration list for the instrument is as follows:

20.3.1 *TEM*:

20.3.1.1 Check the alignment and the systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

20.3.1.2 Calibrate the camera length of the TEM in electron diffraction (ED) operating mode before ED patterns of unknown samples are observed. Camera length can be measured by using a carbon coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thick gold films will tend to mask weak diffraction spots from the fibrous particles. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings from thick films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant calculated for that particular instrument and can then be used for ED patterns of unknowns taken during the corresponding period.

20.3.1.3 Perform magnification calibration at the fluorescent screen. This calibration must be performed at the magnification used for structure counting. Calibration is performed with a grating replica (7.47) (for example, one containing at least 2160 lines/mm).

(a) Define a field of view on the fluorescent screen. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

(b) Frequency of calibration will depend on the service history of the particular microscope.

(c) Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high voltage system or the mechanical disassembly of the electron optical column (apart from filament exchange).

(d) The analyst must ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

(e) For instruments that incorporate a eucentric tilting specimen stage, all specimens and the grating replica must be placed at the eucentric position.

20.3.1.4 The smallest spot size of the TEM must be checked.

(a) At the crossover point, photograph the spot size at a screen magnification of 15 000 to 20 000X. An exposure time of 1 s is usually adequate.

(b) The measured spot size must be less than or equal to 250 nm.

20.4 *EDXA*:

20.4.1 The resolution and calibration of the EDXA must be verified.

20.4.1.1 Collect a standard EDXA Cu peak from the Cu grid.

20.4.1.2 Compare the X-ray energy versus channel number for the Cu peak and be certain that readings are within ±10 eV.

20.4.2 Collect a standard EDXA of crocidolite asbestos (NIST SRM 1866).

20.4.2.1 The elemental analysis of the crocidolite must resolve the Na peak.

20.4.3 Collect a standard EDXA of chrysotile asbestos.

20.4.3.1 The elemental analysis of chrysotile must resolve both Si and Mg on a single chrysotile fiber.

20.5 Ultrasonic bath calibration shall be performed as follows:

20.5.1 Fill the bath water to a level equal to the height of suspension in the glass sample container that will be used for the dust analysis. Operate the bath until the water reaches the equilibrium temperature.

20.5.2 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.3 Place the sample container in the water in the ultrasonic bath (with the power turned off). After 60 s, remove the glass container and record its temperature.

20.5.4 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.5 Place the second sample container into the water in the ultrasonic bath (with the power turned on). After 60 s, remove the glass container and record its temperature.

20.5.6 Calculate the rate of energy deposition into the sample container using the following formula:

$$R = 4.185 \times \sigma \times \rho \times \frac{(\theta_2 - \theta_1)}{t} \quad (4)$$

where:

4.185 = Joules/cal,
R = energy deposition, watts/mL,
θ_1 = temperature rise with the ultrasonic bath not operating, °C,
θ_2 = temperature rise with the ultrasonic bath operating, °C,
t = time in seconds, 60 s (20.5.3 and 20.5.5),
σ = specific heat of the liquid in the glass sample container, 1.0 cal/g, and
ρ = density of the liquid in the glass sample container, 1.0 g/cm^3.

20.5.7 Adjust the operating conditions of the bath so that the rate of energy deposition is in the range of 0.08 to 0.12 MW/m^3, as defined by this procedure.

21. Precision and Bias

21.1 *Precision*—The precision of the procedure in this test method is being determined using round robin data from participating laboratories.

21.2 *Bias*—Since there is no accepted reference material suitable for determining the bias of the procedure in this test method, bias has not been determined (see Specification D 3670).

NOTE 3—Round robin data is under development and will be presented as a research report.

22. Keywords

22.1 asbestos; microvacuuming; settled dust; TEM

APPENDIX

(Nonmandatory Information)

X1. DUST SAMPLE ANALYSIS

X1.1 See Figs. X1.1 and X1.2 for the dust analysis worksheet and the TEM count sheet.

DUST SAMPLE ANALYSIS

Client:	_____	Accelerating Voltage:	_____
Sample ID:	_____	Indicated Mag:	KX
Job Number:	_____	Screen Mag:	KX
Date Sample Analyzed:	- -	Microscope:	1 2 3 4 5
Number of Openings/Grids Counted:		Filter Type:	_____
Grid Accepted, 600X:	Yes No	Filter Size:	_____
Percent Loading:	%	Filter Pore Size (μm):	_____
Grid Box #1:	_____	Grid Opening:	1) μm x μm
			2) μm x μm

Analyst: _____

Reviewer: _____

Counting Rules: AHERA LEVEL II

Calculation Data:

Effective Filter Area in mm^2:	(EFA)	_____
Number of Grid Openings Counted:	(GO)	_____
Average Grid Opening Area in mm^2:	(GOA)	_____
Volume of sample Filtered in ml:	(V)	_____
Surface area Sampled in cm^2:	(SPL)	_____
Number of Asbestos Structures Counted:*	(#STR)	_____

* If the number of asbestos structures counted is less than or equal to 4, enter 4 structures as the limit of detection here.

FORMULA FOR CALCULATION OF ASBESTOS STRUCTURES "DUST" PER CM^2:

$$\frac{EFA \times 100 \times \#STR}{GO \times GOA \times V \times SPL} = \text{(Asbestos Structures per } cm^2\text{)}$$

Results for Total Asbestos Structures: _____
(Structures per cm^2)

Results for Structures ≥ microns: _____
(Structures per cm^2)

FIG. X1.1 Dust Sample Analysis Work Sheet

Job Number: _____

Structure #	Grid # Square #	Type	Structure	Length Microns	Width Microns	Confirmation		
						Morph.	SAED	EDS

Note: Keys to Abbreviations Used in Figure:

Type:

C = Chrysotile
AM = Amosite
CR = Crocidolite
AC = Actinolite
TR = Tremolite
AN = Anthophyllite
N = Non Asbestos

Structure:

F = Fiber
B = Bundle
C = Cluster
M = Matrix

Others:

NSD = No Structures Detected
Morph = Morphology
SAED = Selected Area Electron Diffraction
EDS = Energy Dispersive X-Ray Spectroscopy
ER = Inter-Row Spacing
NP = No Pattern

FIG. X1.2 TEM Count Sheet

REFERENCES

(1) Steel, E. and Wylie, A., "Mineralogical Characteristics of Asbestos," in *Geology of Asbestos Deposits*, Riordon, P. H., Ed., SME-AIME, 1981, pp. 93–101.

(2) Zussman, J., "The Mineralogy of Asbestos," in *Asbestos: Properties, Applications and Hazards*, John Wiley and Sons, 1979, pp. 45–67.

(3) Leake, B. E., "Nomenclature of Amphiboles," *American Mineralogist*, Vol 63, 1978, pp. 1023–1052.

(4) " *USEPA Asbestos-Containing Materials in Schools: Final Rule and Notice," Federal Register*, 40 CFR Part 763, Appendix A to Sub-part E., Oct. 30, 1987.

(5) OSHA, *OSHA Technical Manual, OSHA Instruction CPL 2-20B*, Directorate of Technical Support, U.S. Department of Labor, Washington, DC 20210, Feb. 5, 1990, pp. 1–8 to 1–11.

(6) *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis*, NISTIR, August 1989, pp. 89–4137.

(7) Yamate, G., Agarwall, S. C., and Gibbons, R. D., "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy," EPA Draft Report, Contract No. 68-02-3266, 1984.

(8) Chatfield, E. J., and Dillon, M. J., "Analytical Method for the Determination of Asbestos in Water," EPA No. 600/4-83-043, 1983.

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Test Method

Method for the Determination of Asbestos in Bulk Building Materials

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TEST METHOD

METHOD FOR THE DETERMINATION OF ASBESTOS IN BULK BUILDING MATERIALS

by

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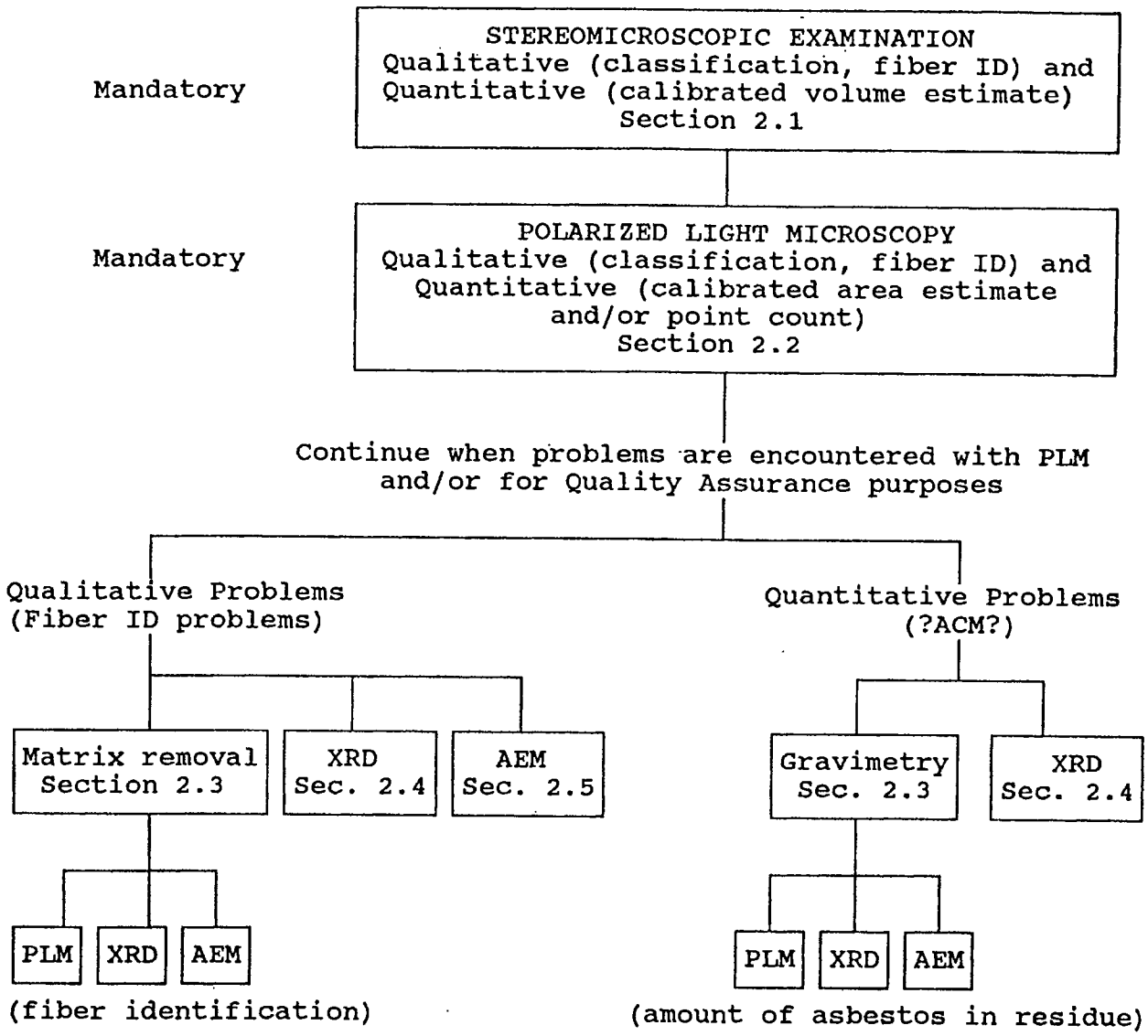
1.0 INTRODUCTION

Laboratories are now called upon to identify asbestos in a variety of bulk building materials, including loose-fill insulations, acoustic and thermal sprays, pipe and boiler wraps, plasters, paints, flooring products, roofing materials and cementitious products.

The diversity of bulk materials necessitates the use of several different methods of sample preparation and analysis. An analysis with a simple stereomicroscope is always followed by a polarized light microscopic (PLM) analysis. The results of these analyses are generally sufficient for identification and quantitation of major concentrations of asbestos. However, during these stereomicroscopic and PLM analyses, it may be found that additional techniques are needed to: 1) attain a positive identification of asbestos; 2) attain a reasonable accuracy for the quantity of asbestos in the sample; or 3) perform quality assurance activities to characterize a laboratory's performance. The additional techniques include x-ray diffraction (XRD), analytical electron microscopy (AEM), and gravimetry, for which there are sections included in the method. Other techniques will be considered by the Environmental Protection Agency (EPA) and may be added at some future time. Table 1-1 presents a simplified flowchart for analysis of bulk materials.

This Method for the Determination of Asbestos in Bulk Building Materials outlines the applicability of the various preparation and analysis methods to the broad spectrum of bulk building materials now being analyzed. This method has been evaluated by the EPA Atmospheric Research and Exposure Assessment Laboratory (EPA/AREAL) to determine if it offers improvements to current analytical techniques for building materials. This method demonstrated a capability for improving the precision and accuracy of analytical results. It contains significant revisions to procedures outlined in the Interim Method,[1] along with the addition of several new procedures. Each technique may reduce or introduce bias, or have some effect on the precision of the measurement, therefore results need to be interpreted judiciously. Data on each technique, especially those new to asbestos analysis, will be collected over time and carefully evaluated, with resulting recommendations for changes to the Method to be passed on to the appropriate program office within EPA.

TABLE 1-1. SIMPLIFIED FLOWCHART FOR ANALYSIS OF BULK MATERIALS



This is an analytical method. It is not intended to cover bulk material sampling, an area addressed previously[2,3,4,5] by the EPA. However, subsampling or sample splitting as it pertains to laboratory analysis procedures in this method, is discussed throughout.

1.1 References

1. **Interim Method for the Determination of Asbestos in Bulk Insulation Samples,** U.S. E.P.A. 600/M4-82-020, 1982.
2. **Asbestos-Containing Materials in School Buildings: A Guidance Document,** Part 1 and 2, U.S. E.P.A./O.T.S NO. C00090, 1979.
3. **Asbestos in Buildings: Simplified Sampling Scheme for Friable Surfacing Materials,** U.S. E.P.A. 560/5-85-030a, 1985.
4. **Guidance for Controlling Asbestos-Containing Materials in Buildings,** U.S. E.P.A. 560/5-85-024, 1985.
5. **Asbestos-Containing Materials in Schools: Final Rule and Notice,** 40 CFR Part 763, October, 1987.

2.0 METHODS

2.1 Stereomicroscopic Examination

A preliminary visual examination using a simple stereomicroscope is mandatory for all samples. A sample should be of sufficient size to provide for an adequate examination. For many samples, observations on homogeneity, preliminary fiber identification and semi-quantitation of constituents can be made at this point. Another method of identification and semi-quantitation of asbestos must be used in conjunction with the stereomicroscopic examination. A description of the suggested apparatus needed for stereomicroscopic examination is given in Appendix B.

The laboratory should note any samples of insufficient volume. A sufficient sample volume is sample-type dependent. For samples such as floor tiles, roofing felts, paper insulation, etc., three to four square inches of the layered material would be a preferred sample size. For materials such as ceiling tiles, loose-fill insulation, pipe insulation, etc., a sample size of approximately one cubic inch (~15cc) would be preferred. For samples of thin-coating materials such as paints, mastics, spray plasters, tapes, etc., a smaller sample

size may be suitable for analysis. Generally, samples of insufficient volume **should be rejected**, and further analysis curtailed until the client is contacted. The quantity of sample affects the sensitivity of the analysis and reliability of the quantitation steps. If there is a question whether the sample is representative due to inhomogeneity, the sample should be rejected, at least until contacting the client to see if: 1) the client can provide more material or 2) the client wishes the laboratory to go ahead with the analysis, but with the laboratory including a statement on the limited sensitivity and reliability of quantitation. If the latter is the case, the report of analysis should state that the client was contacted, that the client decided that the lab should use less material than recommended by the method, and that the client acknowledges that this may have limited the sensitivity and quantitation of the method. At the time the client is contacted about the material, he or she should be informed that a statement reflecting these facts will be placed in the report.

2.1.1 Applicability

Stereomicroscopic analysis is applicable to all samples, although its use with vinyl floor tile, asphaltic products, etc., may be limited because of small asbestos fiber size and/or the presence of interfering components. It does not provide positive identification of asbestos.

2.1.2 Range

Asbestos may be detected at concentrations less than one percent by volume, but this detection is highly material dependent.

2.1.3 Interferences

Detection of possible asbestos fibers may be made more difficult by the presence of other nonasbestos fibrous components such as cellulose, fiber glass, etc., by binder/matrix materials which may mask or obscure fibrous components, and/or by exposure to conditions (acid environment, high temperature, etc.) capable of altering or transforming asbestos.

2.1.4 Precision and Accuracy

The precision and accuracy of these estimations are material dependent and must be determined by the individual laboratory for the percent range involved. These values may be

determined for an individual analyst by the in-house preparation and analysis of standards and the use of error bars, control charts, etc.

The labs should also compare to National Voluntary Laboratory Accreditation Program (NVLAP) proficiency testing samples, if the lab participates in the Bulk Asbestos NVLAP, or to external quality assurance system consensus results such as from proficiency testing programs using characterized materials. However, at this time, consensus values for the quantity of asbestos have been shown to be unreliable. Only proficiency testing materials characterized by multiple techniques should be used to determine accuracy and precision.

2.1.5 Procedures

NOTE: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are oftentimes friable and may release fibers during handling or matrix reduction steps. All sample handling and examination must be carried out in a HEPA-filtered hood, a class 1 biohazard hood or a glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst to and contamination of samples by airborne fibers.

2.1.5.1 Sample Preparation

No sample preparation should be undertaken before initial stereomicroscopic examination. Distinct changes in texture or color on a stereomicroscopic scale that might denote an uneven distribution of components should be noted. When a sample consists of two or more distinct layers or building materials, each should be treated as a separate sample, when possible. Thin coatings of paint, rust, mastic, etc., that cannot be separated from the sample without compromising the layer are an exception to this case and may be included with the layer to which they are attached. Drying (by heat lamp, warm plate, etc.) of wet or damp samples is recommended before further stereomicroscopic examination and is mandatory before PLM examination. **Drying must be done in a safety hood.**

For nonlayered materials that are heterogeneous, homogenization by some means (mill, blender, mortar and pestle) may provide a more even distribution of sample components. It

may also facilitate disaggregation of clumps and removal of binder from fibers (rarely however, it may mask fibers that were originally discernable).

For materials such as cementitious products and floor tiles, breaking, pulverizing, or grinding may improve the likelihood of exposing fibrous components.

It may be appropriate to treat some materials by dissolution with hydrochloric acid to remove binder/matrix materials. Components such as calcite, gypsum, magnesite, etc., may be removed by this method. For materials found to possess a high organic content (cellulose, organic binders), ashing by means of a muffle furnace or plasma asher (for small, cellulosic samples), or dissolution by solvents may be used to remove interfering material. In either case, it is recommended that matrix removal be tracked gravimetrically.

Additional information concerning homogenization, ashing and acid dissolution may be found in Sections 2.2.5.1 and 2.3.

2.1.5.2 Analysis

Samples should be examined with a simple stereomicroscope by viewing multiple fields of view over the entire sample. The whole sample should be observed after placement in a suitable container (watchglass, weigh boat, etc.) substrate. Samples that are very large should be subsampled. The sample should be probed, by turning pieces over and breaking open large clumps. The purpose of the stereomicroscopic analysis is to determine homogeneity, texture, friability, color, and the extent of fibrous components of the sample. This information should then be used as a guide to the selection of further, more definitive qualitative and quantitative asbestos analysis methods. Homogeneity refers to whether each subsample made for other analytical techniques (e.g. the "pinch" mount used for the PLM analysis), is likely to be similar or dissimilar. Color can be used to help determine homogeneity, whether the sample has become wet (rust color), and to help identify or clarify sample labelling confusion between the building material sampler and the laboratory. Texture refers to size, shape and arrangement of sample components. Friability may be indicated by the ease with which the sample is disaggregated (see definitions in Appendix A) as received by the analyst. This does not necessarily represent the friability of the material as determined by the assessor at the collection site. The relative proportion of fibrous

components to binder/matrix material may be determined by comparison to similar materials of known fibrous content. For materials composed of distinct layers or two or more distinct building materials, each layer or distinct building material should be treated as a discrete sample. The relative proportion of each in the sample should be recorded. The layers or materials should then be separated and analyzed individually. Analysis results for each layer or distinct building material should be reported. If monitoring requirements call for one reported value, the results for the individual layers or materials should always be reported along with the combined value. Each layer or material should be checked for homogeneity during the stereomicroscopic analysis to determine the extent of sample preparation and homogenization necessary for successful PLM or other analysis. Fibers and other components should be removed for further qualitative PLM examination.

Using the information from the stereomicroscopic examination, selection of additional preparation and analytical procedures should be made. Stereomicroscopic examination should typically be performed again after any change or major preparation (ashing, acid dissolution, milling, etc.) to the sample. Stereomicroscopic examination for estimation of asbestos content may also be performed again after the qualitative techniques have clarified the identities of the various fibrous components to assist in resolving differences between the initial quantitative estimates made during the stereomicroscopic analysis and those of subsequent techniques. Calibration of analysts by use of materials of known asbestos content is essential.

The stereomicroscopic examination is often an iterative process. Initial examination and estimates of asbestos concentration should be made. The sample should then be analyzed by PLM and possibly other techniques. These results should be compared to the initial stereomicroscopic results. Where necessary, disagreements between results of the techniques should be resolved by reanalyzing the sample stereomicroscopically.

2.1.6 Calibration Materials

Calibration materials fall into several categories, including internal laboratory standards and other materials that have known asbestos weight percent content. These calibration materials could include:

- Actual bulk samples: asbestos-containing materials that have been characterized by other analytical methods such as XRD, AEM and/or gravimetry. (e.g. NVLAP test samples).

- Generated samples: in-house standards that can be prepared by mixing known quantities of asbestos and known quantities of asbestos-free matrix materials (by weight), and mixing (using blender, mill, etc.) thoroughly to achieve homogeneity; matrix materials such as vermiculite, perlite, sand, fiberglass, calcium carbonate, etc. may be used. A range of asbestos concentrations should be prepared (e.g. 1, 3, 5, 10, 20%, etc.). The relationship between specific gravities of the components used in standards should be considered so that weight/volume relationships may be determined.

- Photographs, drawings: photomicrographs of standards, computer-generated drawings, etc.

Suggested techniques for the preparation and use of in-house calibration standards are presented in Appendix C, and at greater length by Harvey et al.[1] The use of synthesized standards for analyst calibration and internal laboratory quality control is not new however, having been outlined by Webber et al.[2] in 1982.

2.1.7 References

1. Harvey, B. W., R. L. Perkins, J. G. Nickerson, A. J. Newland and M. E. Beard, "Formulating Bulk Asbestos Standards", **Asbestos Issues**, April 1991, pp. 22-29.

2. Webber, J. S., A. Pupons and J. M. Fleser, "Quality-Control Testing for Asbestos Analysis with Synthetic Bulk Materials". **American Industrial Hygiene Associations Journal**, 43, 1982, pp. 427-431.

2.2 Polarized Light Microscopy

2.2.1 Principle and Applicability

Samples of bulk building materials taken for asbestos identification should first be examined with the simple stereomicroscope to determine homogeneity and preliminary fiber identification. Subsamples should then be examined using PLM to determine optical properties of constituents and to provide positive identification of suspect fibers.

The principles of optical mineralogy are well-established.[1,2,3,4] A light microscope equipped with two polarizing filters is used to observe specific optical characteristics of a sample. The use of plane polarized light allows for the determination of refractive indices relative to specific crystallographic orientations. Morphology and color are also observed while viewing under plane polarized light. Observation of particles or fibers while oriented between polarizing filters whose privileged vibration directions are perpendicular (crossed polars) allows for determination of isotropism/anisotropism, extinction characteristics of anisotropic particles, and calculation of birefringence. A retardation plate may be placed in the polarized light path for verification of the sign of elongation. If subsamples are prepared in such a way as to represent all sample components and not just suspect fibers, semi-quantitative analysis may also be performed. Semi-quantitative analysis involves the use of calibrated visual area estimation and/or point counting. Visual area estimation is a semi-quantitative method that must relate back to calibration materials. Point counting, also semi-quantitative, is a standard technique used in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting[3] and the interpretation of point count data[5] is available.

Although PLM analysis is the primary technique used for asbestos determination, it can show significant bias leading to false negatives and false positives for certain types of materials. PLM is limited by the visibility of the asbestos fibers. In some samples the fibers may be reduced to a diameter so small or masked by coatings to such an extent that they cannot be reliably observed or identified using PLM.

2.2.2 Range

The detection limit for visual estimation is a function of the quantity of sample analyzed, the nature of matrix interference, sample preparation, and fiber size and distribution. Asbestos may be detected in concentrations of less than one percent by area if sufficient material is analyzed. Since floor tiles may contain fibers too small to be resolved by PLM ($< 0.25\ \mu m$ in diameter), detection of those fibers by this method may not be possible. When point counting is used, the detection limit is directly proportional to the amount of sample analyzed, but is also limited by fiber visibility. Quantitation by area estimation, both visual and by point counting, should yield similar results if based on calibration standards.

2.2.3 Interferences

Fibrous and nonfibrous, organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the asbestos mineral content. Binder/matrix materials may coat fibers, affect color, or obscure optical characteristics to the extent of masking fiber identity. Many organic mastics are soluble in refractive index liquids and, unless removed prior to PLM examination, may affect the refractive index measurement of constituent materials. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in identification. Gravimetric procedures for the removal of interfering materials are presented in Section 2.3.

2.2.4 Precision and Accuracy

Data obtained for samples containing a single asbestos type in a sample matrix have been reported previously by Brantley et al.[6] Data for establishing the accuracy and precision of the method for samples with various matrices have recently become available. Perkins,[7] Webber et al.[8] and Harvey et al.[9] have each documented the tendency for visual estimates to be high when compared to point-count data. Precision and accuracy must be determined by the individual laboratory for the percent range involved. If point counting and/or visual estimates are used, a table of reasonably expanded errors, such as those shown in Table 2-1, should be generated for different concentrations of asbestos.

If the laboratory cannot demonstrate adequate precision and accuracy (documented by control charts, etc), quantitation by additional methods, such as gravimetry, may be required. Refer to the Handbook for SRM Users[10] for additional information concerning the concepts of precision and accuracy.

TABLE 2-1. SUGGESTED ACCEPTABLE ERRORS FOR PLM ANALYSIS
(Based on 400 point counts of a reasonably homogeneous sample or 100 fields of view for visual estimate)

% Area Asbestos	Acceptable Mean Result	% Area Asbestos	Acceptable Mean Result
1	>0-3%	50	40-60%
5	>1-9%	60	50-70%
10	5-15%	70	60-80%
20	10-30%	80	70-90%
30	20-40%	90	80-100%
40	30-50%	100	90-100%

2.2.5 Procedures

NOTE: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are oftentimes friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations must be carried out in a HEPA-filtered, a class 1 biohazard hood, or a glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst to and contamination of samples by airborne fibers.

2.2.5.1 Sample Preparation

Slide mounts are prepared for the identification and quantitation of asbestos in the sample.

2.2.5.1.1 Qualitative Analysis Preparation

The qualitative preparation must allow the PLM analysis to classify the fibrous components of the sample as asbestos or nonasbestos. The major goal of the qualitative

preparation is to mount easily visible fibers in appropriate refractive index liquids for complete optical characterization. Often this can be accomplished by making immersion grain mounts of random subsamples of the homogeneous material. Immersion liquids with refractive indices close to the suspected (see stereomicroscopic analysis) asbestos mineral should be used for the qualitative analysis so that n_D can be determined. Problem samples include those with inhomogeneities, coatings, small fibers, and interfering compounds. Additional qualitative preparations are often necessary for these types of samples. All samples, but especially those lacking homogeneity, may require picking of fibers from specific sample areas during the stereomicroscopic examination. Coatings on the fibers often need to be removed by mechanical or chemical means. Teasing the particles apart or use of a mortar and pestle or similar mechanical method often is sufficient to free fibers from coatings. Chemical means of removing some coatings and interfering compounds are discussed in Section 2.3, Gravimetry.

2.2.5.1.2 Quantitative Analysis Preparation

The major purpose of the quantitative preparation is to provide the analyst with a representative grain mount of the sample in which the asbestos can be observed and distinguished from the nonasbestos matrix. This is typically performed by using randomly selected subsamples from a homogeneous sample (see stereomicroscopic analysis). Particles should be mounted in a refractive index (RI) liquid that allows the asbestos to be visible and distinguished from nonasbestos components. Care should be taken to ensure proper loading and even distribution of particles. Both the qualitative and quantitative sample preparations are often iterative processes. Initial samples are prepared and analyzed. The PLM analysis may disclose problems or raise questions that can only be resolved by further preparations (e.g. through the use of different RI immersion liquids, elimination of interfering compounds, sample homogenization, etc.)

For layered materials, subsamples should be taken from each individual or discrete layer. Each of these subsamples should be treated as a discrete sample, but as stated in Section 2.1.5.2, the results for the individual layers or materials may be combined if called for by monitoring requirements.

Homogenization involves the use of any of a variety of devices, such as a mortar and pestle, mill, or blender to pulverize, disaggregate and mix heterogeneous, friable bulk materials. Selection of the appropriate device is dependent upon personal preference and the nature of the materials encountered. A blender or mortar and pestle may be adequate for homogenizing materials that lack appreciable amounts of tacky matrix/binder, and for separating interfering components from the fibers. For materials which are unusually sticky or tacky, or contain unusually long asbestos fibers, milling (especially freezer milling) may be more efficient. However, milling should be discontinued as soon as the material being milled appears homogeneous, in order to reduce the potential for mechanically reducing fiber size below the resolving power of the polarizing microscope. Hammer mills or cutting mills may also be used on these materials; however, the same precaution regarding reduction of fiber size should be taken. Blending /milling devices should be disassembled (to the extent possible) and thoroughly cleaned after each use to minimize contamination.

2.2.5.2 Analysis

Analysis of bulk building materials consists of the identification and semi-quantitation of the asbestos type(s) present, along with the identification, where possible, of fibrous nonasbestos materials, mineral components and matrix materials. If the sample is heterogeneous due to the presence of discrete layers or two or more distinct building materials, each layer or distinct material should be analyzed, and results reported. Total asbestos content may also be stated in terms of a relative percentage of the total sample.

2.2.5.2.1 Identification

Positive identification of asbestos requires the determination of the following optical properties:

- Morphology
- Color and, if present, pleochroism
- Refractive indices ($\pm$ .005)
- Birefringence
- Extinction characteristics
- Sign of elongation

Descriptions of the optical properties listed above for asbestos fibers may be found in Appendix A, Glossary of Terms. Table 2-2 lists the above properties for the six types of asbestos and Table 2-3 presents the central stop dispersion staining colors for the asbestos minerals with selected high-dispersion index liquids. Tables 2-4 and 2-5 list selected optical properties of several mineral and man-made fibers. All fibrous materials in amounts greater than trace should be identified as asbestos or nonasbestos, with all optical properties measured for asbestos and at least one optical property measured for each nonasbestos fibrous component that will distinguish each from asbestos. Small fiber size and/or binder may necessitate viewing the sample at higher magnification (400-500x) than routinely used (100x).

Although it is not the purpose of this section to explain the principles of optical mineralogy, some discussion of the determination of refractive indices is warranted due to its importance to the proper identification of the asbestos minerals. Following is a brief discussion of refractive index determination for the asbestos minerals.

All asbestos minerals are anisotropic, meaning that they exhibit different optical properties (including indices of refraction) in different directions. All asbestos minerals are biaxial, meaning that they have one principal refractive index parallel (or nearly parallel) to the length of the fiber and two principal refractive indices (plus all intermediate indices between these two) in the plane perpendicular (or nearly so) to the length of the fiber. Although chrysotile (serpentine) is classified as a biaxial mineral, it behaves as a uniaxial mineral (two principal refractive indices) due to its scrolled structure. Amosite and crocidolite, although also biaxial, exhibit uniaxial properties due to twinning of the crystal structure and/or random orientation of fibrils in a bundle around the long axis of the bundle. For all of the asbestos minerals except crocidolite, the highest refractive index (γ) is aligned with the fiber length (positive sign of elongation). For crocidolite, the lowest refractive index (α) is aligned with the fiber length (negative sign of elongation). A more complete explanation of the relationship of refractive indices to the crystallographic directions of the asbestos minerals may be found in References 1, 2, 4, 11 and 12. It should be noted that for the measurement of refractive indices in an anisotropic particle (e.g. asbestos fibers), the orientation of the particle is quite critical. Orientation with respect to rotation about the axis

of the microscope (and thus with respect to the vibration directions of the polarizer and analyzer) and also to the horizontal plane (plane of the microscope stage) will affect the determination of the correct values for refractive indices. The refractive index that is measured will always correspond to a direction perpendicular to the axis of the microscope (i.e., lying in the plane of the stage) and is the direction in that horizontal plane parallel to the vibration direction of the polarizer, by convention E-W.

To determine $\gamma(n \parallel)$ for chrysotile, anthophyllite and amosite, the index is measured when the length of the fiber is aligned parallel to the vibration direction of the polarizer (E-W). Under crossed polars, the fiber should be at extinction in this orientation. To determine the lowest refractive index, α $(n \perp)$, for chrysotile and amosite, the fiber should be oriented N-S (extinction position under crossed polars). The determination of $n \parallel$ and $n \perp$ with crocidolite is accomplished in the same manner as with amosite and chrysotile with the exception that the α and γ directions are reversed. For crocidolite, α is measured at the E-W position (parallel to the polarizer) and γ is measured at the N-S orientation (perpendicular to the polarizer). For anthophyllite, the fiber should be oriented N-S and the lowest and highest indices for this orientation should be measured. These correspond to α and β respectively.

The extinction behavior of tremolite-actinolite is anomalous compared to that of most monoclinic minerals due to the orientation of the optic axes relative to the crystallographic axes. This relationship is such that the refractive indices of the principal axes α and γ <u>are not measured</u> when the fiber is exhibiting the maximum extinction angle. The values measured at these positions are α' and γ'. The fiber exhibits an extinction angle within a few degrees of the maximum throughout most of its rotation. A wide range of refractive indices from α' to α, and from γ' to γ, are observed. For tremolite-actinolite, β is measured on those fibers displaying parallel extinction when oriented in the N-S position. The refractive index for α is also measured when the fiber is oriented generally in the N-S position and exhibits <u>the true extinction</u> angle; true α will be the <u>minimum</u> index. To determine the refractive index for γ, the fibers should be oriented E-W and exhibit the true extinction angle; true γ will be the <u>maximum</u> value for this orientation.

When viewing single fibers, the analyst may often be able to manipulate the microscope slide cover slip and "roll" the fibers to positions that facilitate measuring the true values of refractive indices. When viewing a large population of fibers with the microscope in the dispersion staining mode, the analyst can easily detect fibers that exhibit the highest and lowest indices (β and α) in the N-S position and the highest indices (γ) in the E-W position. Since individual asbestos fibrils cannot generally be resolved using polarized light microscopy, refractive indices are most commonly measured on fiber bundles. Such measurements would not result in true values for the indices and therefore by convention should be reported as α' and γ'.

Asbestos types chrysotile, amosite and crocidolite are currently available as SRM 1866 and actinolite, tremolite and anthophyllite as SRM 1867 from the Office of Standard Reference Materials, National Institute of Standards and Technology.

2.2.5.2.2 Quantitation of Asbestos Content

As described in Sections 2.1.5 and 2.1.6, a calibrated visual volume estimation of the relative concentrations of asbestos and nonasbestos components should be made during the stereomicroscopic examination. In addition, quantitation of asbestos content should be performed on subsample slide mounts using calibrated visual area estimates and/or a point counting procedure. Section 2.1.6 and Appendix C discuss the procedures for preparation and use of calibration standards. After thorough PLM analysis in which the asbestos and other components of the bulk material are identified, several slides should be carefully prepared from randomly selected subsamples. If the sample is not homogeneous, some homogenization procedure should be performed to ensure that slide preparations made from small pinch samples are representative of the total sample. Homogenization may range from gentle mixing using a mortar and pestle to a brief period of mixing using a blender equipped with a mini-sample container. The homogenization should be of short duration ($\sim$ 15 seconds) if using the blender technique so as to preclude a significant reduction in fiber size. The use of large cover slips (22x30mm) allows for large subsamples to be analyzed. Each slide should be checked to ensure that the subsample is representative, uniformly dispersed, and loaded in a way so as not to be dominated by superimposed (overlapping) particles.

During the qualitative analysis of the sample, the analyst should decide on the appropriate optical system (including magnification) to maximize the visibility of the asbestos in the sample while still allowing the asbestos to be uniquely distinguished from the matrix materials. The analyst may choose to alter the mounting medium or the optical system to enhance contrast. During the quantitative analysis, slides should be scanned using an optical setup that yields the best visibility of the asbestos. Upon finding asbestos, the parameters that were selected in the qualitative analysis for uniquely distinguishing it from the matrix should be used for identification. These properties will vary with the sample but include any or all of the parameters required for the qualitative analysis. For instance, low magnification allows for concurrent use of dispersion staining (focal screening), but compromises resolution of extremely small diameter fibers; use of a compensator plate and crossed polarizers frequently enhances the contrast between asbestos fibers and matrix material.

Visual area estimates should be made by comparison of the sample to calibration materials that have similar textures and fiber abundance (see Section 2.1.6 and Appendix C). A minimum of three slide mounts should be examined to determine the asbestos content by visual area estimation. Each slide should be scanned in its entirety and the relative proportions of asbestos and nonasbestos noted. It is suggested that the ratio of asbestos to nonasbestos material be recorded for several fields for each slide and the results be compared to data derived from the analysis of calibration materials having similar textures and asbestos content.

For point counting, an ocular reticle (cross-line or point array) should be used to visually superimpose a point or points on the microscope field of view. The cross-line reticle is preferred. Its use requires the scanning of most, if not all, of the slide area, thereby minimizing bias that might result from lack of homogeneity in the slide preparation. In conjunction with this reticle, a click-stop counting stage can be used to preclude introducing bias during slide advancement. Magnification used will be dictated by fiber visibility. The slide should be examined along multiple parallel traverses that adequately cover the sample area. The analyst should score (count) only points <u>directly over</u> occupied (nonempty) areas. Empty points should <u>not</u> be scored on the basis of the closest particle. If an asbestos fiber and a nonasbestos particle overlap so that a point is superimposed on their visual intersection,

a point should be scored for both categories. If the point(s) is/are superimposed on an area which has several overlapping particles, the slide should be moved to another field. While not including them in the total asbestos points counted, the analyst should record the presence of any asbestos detected but not lying under the reticle cross-line or array points. A minimum of 400 counts (maximum of eight slides with 50 counts each to minimum of two slides with 200 counts each) per sample is suggested, but it should be noted that accuracy and precision improve with number of counts. Point counting provides a determination of the projected area percent asbestos. Conversion of area percent to dry weight percent is not feasible unless the specific gravities and relative volumes of the different materials are known. It should be noted that the total amount of material to be analyzed is dependent on the asbestos concentration, i.e. the lower the concentration of asbestos, the larger the amount of sample that should be analyzed, in both the visual estimation and point counting methods. Quantitation by either method is made more difficult by low asbestos concentration, small fiber size, and presence of interfering materials.

It is suggested that asbestos concentration be reported as volume percent, weight percent or area percent depending on the method of quantitation used. A weight concentration cannot be determined without knowing the relative specific gravities and volumes of the sample components.

TABLE 2-2. OPTICAL PROPERTIES OF ASBESTOS FIBERS

Mineral	Morphology and Color[1]	Refractive Indices[2] α	Refractive Indices[2] γ[5]	Birefringence[6]	Extinction	Sign of Elongation
Chrysotile (asbestiform serpentine)	Wavy fibers. Fiber bundles have splayed ends and "kinks". Aspect ratio typically >10:1. Colorless[3]	1.493-1.546 1.532-1.549 1.529-1.559 1.544-1.553	1.517-1.557 1.545-1.556 1.537-1.567 1.552-1.561	0.004-0.017	Parallel	+ (length slow)
Amosite (asbestiform grunerite)	Straight to curved, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so.[4] Opaque inclusions may be present	1.657-1.663 1.663-1.686 1.663-1.686 1.676-1.683	1.699-1.717 1.696-1.729 1.696-1.729 1.697-1.704	0.021-0.054	Usually parallel	+ (length slow)
Crocidolite (asbestiform riebeckite)	Straight to curved, rigid fibers. Aspect ratio typically > 10:1. Thick fibers and bundles common, blue to dark-blue in color. Pleochroic.	1.693 1.654-1.701 1.680-1.698	1.697 1.668-1.717 1.685-1.706	0.003-0.022	Usually parallel	- (length fast)
Anthophyllite-asbestos	Straight to curved fibers and bundles. Aspect ratio typically > 10:1. Anthophyllite cleavage fragments may be present with aspect ratios <10:1. Colorless to light brown.	1.598-1.652 1.596-1.694 1.598-1.674 1.6148[7]	1.623-1.676 1.615-1.722 1.615-1.697 1.6362[7]	0.013-0.028	Parallel	+ (length slow)
Tremolite-Actinolite-asbestos	Straight to curved fibers and bundles. Aspect ratio typically > 10:1. Cleavage fragments may be present with aspect ratios <10:1. Colorless to pale green	Tremolite 1.600-1.628 1.604-1.612 1.599-1.612 1.6063[7] Actinolite 1.600-1.628 1.612-1.668 1.613-1.628 1.6126[7]	Tremolite 1.625-1.655 1.627-1.635 1.625-1.637 1.6343[7] Actinolite 1.625-1.655 1.635-1.688 1.638-1.655 1.6393[7]	0.017-0.028 0.017-0.028	Parallel and oblique (up to 21°); Composite fibers show parallel extinction.	+ (length slow)

[1]Colors cited are seen by observation with plane polarized light.

[2]From references 2, 11, 12, and 18, respectively. Refractive indices for n_d at 589.3nm.

[3]Fibers subjected to heating may be brownish. (references 13, 14, and 15)

[4]Fibers subjected to heating may be dark brown and pleochroic. (references 13, 14, and 15)

[5]∥ to fiber length, except ⊥ to fiber length for crocidolite only.

[6]Maximum and minimum values from references 2, 11, 12, and 18 given.

[7]± 0.0007

TABLE 2-3. TYPICAL CENTRAL STOP DISPERSION STAINING COLORS[1]

Mineral	Cargille® RI Liquid	n∥	n⊥
Chrysotile	1.550HD	Magenta to light blue-green λ_0's ca. 520-620nm	Blue-green to pale blue λ_0's ca. 600-700nm
Amosite	1.680	Yellow to magenta λ_0's ca. 420-520nm	Blue magenta to light blue λ_0's ca. 560-660nm
Crocidolite	1.680	Yellow to magenta λ_0's ca. 420-520nm	Pale yellow to golden yellow λ_0's ca. 360-460nm
Anthophyllite-asbestos	1.605HD	Pale yellow to yellow λ_0's ca. 330-430nm	Golden yellow to light blue green λ_0's ca. 460-700nm
Tremolite-asbestos	1.605HD	Pale yellow to yellow λ_0's ca. 330-430nm	Golden yellow to light blue green λ_0's ca. 460-700nm
Actinolite-asbestos	1.605HD	Pale yellow λ_0's ca. 260-360nm	Pale yellow to golden yellow λ_0's ca. 360-460nm
	1.630HD	Yellow to magenta λ_0's ca. 420-520nm	Golden yellow to blue λ_0's ca. 450-600nm

[1]Modified from reference 16

TABLE 2-4. OPTICAL PROPERTIES OF MAN-MADE TEXTILE FIBERS[1,2]

Fiber Type	n∥	n⊥	n∥ - n⊥	Sign of Elongation
Polyester (Dacron®)	1.710	1.535	0.175	+
Polyamide (Nylon®)	1.582	1.514	0.063	+
Aramid (Kevlar®)	≈2.37	≈1.641	0.729	+
Olefin (Polyethylene)	1.556	1.512	0.044	+
Olefin (Polypropylene)	1.520	1.495	0.025	+
Viscose Rayon	1.535-1.555	1.515-1.535	0.020	+
Acetate	1.478-1.480	1.473-1.476	0.004-0.005	+
Acrylic (Orlon®)	1.505-1.515	1.507-1.517	0.004-0.002	-
Modacrylic (Dynel®)	1.535	1.532	0.002	+

[1]Modified from reference 17

[2]Refractive indices for specific fibers; other fibers may vary

TABLE 2-5. OPTICAL PROPERTIES OF SELECTED FIBERS[1]

FIBER TYPE	MORPHOLOGY	REFRACTIVE INDICES	BIREFRINGENCE ($n\parallel - n\perp$)	EXTINCTION ANGLE	SIGN OF ELONGATION	DISPERSION STAINING COLORS
Paper (Cellulose)	Tapered, flat ribbons	$n\parallel \sim 1.580$ $n\perp \sim 1.530$	High (0.05)	Parallel and incomplete	+	in 1.550HD $n\parallel$: yellow (λ_0's < 400nm) $n\perp$: pale blue (λ_0's > 700nm)
Olefin (polyethylene)	Filaments or shredded like chrysotile	$n\parallel \sim 1.556$ $n\perp \sim 1.512$	Moderate (0.044)	Parallel	+	in 1.550HD $n\parallel$: yellow to magenta (λ_0's = 440-540nm) $n\perp$: pale blue (λ_0's > 700nm)
Brucite (nemalite)	Straight fibers	$n\parallel \sim 1.560$-1.590 $n\perp \sim 1.580$-1.600	Moderate (0.012-0.020)	Usually parallel	- occasionally +	in 1.550HD $n\parallel$: golden yellow (λ_0's 440-460nm) $n\perp$: yellow (λ_0's 400-440nm)
Heated amosite	Similar to unheated, (brittle and shorter) pleochroic: $n\parallel$-dark brown $n\perp$ yellow	$n\parallel$ and $n\perp$ >1.700[2]	High (> 0.05)	Usually parallel	+	in 1.680HD $n\parallel$ & $n\perp$: both pale yellow to white (λ_0's < 400nm)
Glass fibers, Mineral wool	Exotic shapes, tear drops, single filaments	1.515-1.700	Isotropic	------	-	in 1.550HD usually pale blue to blue (λ_0's 580 to > 700nm)
Wollastonite	Straight needles and blades	$n\parallel \sim 1.630$ $n\perp \sim 1.632$ $n\perp$ also 1.610	Moderate to low (0.018 to 0.002)	Parallel and oblique	+ and -	in 1.605HD $n\parallel$ & $n\perp$: yellow to pale yellow (λ_0's < 460nm)
Fibrous talc	Thin cleavage ribbons and wavy fibers	$n\parallel \sim 1.60$ $n\perp \sim 1.54$	High (0.06)	Parallel and oblique	+	in 1.550HD $n\parallel$: pale yellow (λ_0's <400nm) $n\perp$: pale blue (λ_0's >660nm)

[1]From reference 19

[2]From references 13, 14, and 15

2.2.5.2.3 Microscope Alignment

In order to accurately measure the required optical properties, a properly aligned polarized light microscope must be utilized. The microscope is aligned when:

1) the privileged directions of the substage polarizer and the analyzer are at 90° to one another and are represented by the ocular cross-lines;

2) the compensator plate's privileged vibration directions are 45° to the privileged directions of the polarizer and analyzer;

3) the objectives are centered with respect to stage rotation; and,

4) the substage condenser and iris diaphragm are centered in the optic axis.

Additionally, the accurate measurement of the refractive index of a substance requires the use of calibrated refractive index liquids. These liquids should be calibrated regularly to an accuracy of 0.004, with a temperature accuracy of 2°C using a refractometer or R.I. glass beads.

2.2.6 References

1. Bloss, F. Donald, **An Introduction to the Methods of Optical Crystallography,** Philadelphia: Saunders College Publishing, 1989.

2. Kerr, Paul F., **Optical Mineralogy**, 4th Edition, New York: McGraw Hill, 1977.

3. Chamot, E. M. and C. W. Mason, **Handbook of Chemical Microscopy, Volume One**, 3rd edition, New York: John Wiley & Sons, 1958.

4. Ehlers, Ernest G., **Optical Mineralogy, Vols. 1 and 2,** Palo Alto, CA: Blackwell Scientific Publications, 1987.

5. Chayes, F., **Petrographic Modal Analysis: An Elementary Statistical Appraisal,** New York: John Wiley & Sons, 1956.

6. Brantley, E. P., Jr., K. W. Gold, L. E. Myers, and D. E. Lentzen, **Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method,** EPA-600/S4-82-021, 1982.

7. Perkins, R.L., "Point-Counting Technique for Friable Asbestos-Containing Materials", **The Microscope**, 38, 1990, pp.29-39.

8. Webber, J.S., R. J. Janulis, L. J. Carhart and M.B. Gillespie, "Quantitating Asbestos Content in Friable Bulk Samples: Development of a Stratified Point-Counting Method", **American Industrial Hygiene Association Journal**, 51, 1990, pp. 447-452.

9. Harvey, B. W., R. L. Perkins, J. G. Nickerson, A. J. Newland and M. E. Beard, "Formulating Bulk Asbestos Standards", **Asbestos Issues**, April 1991, pp. 22-29.

10. **Handbook for SRM Users**, NIST (formerly NBS) Special Publication 260-100, U.S. Department of Commerce, 1985.

11. Deer, W.A., R. A. Howie, and J. Zussman, **An Introduction to the Rock Forming Minerals**, Longman, 1966.

12. Heinrich, E. W., **Microscopic Identification of Minerals**, McGraw Hill, 1965.

13. Kressler, J. R., "Changes in Optical Properties of Chrysotile During Acid Leaching", **The Microscope**, 31, 1983, pp. 165-172.

14. Prentice, J. and M. Keech, "Alteration of Asbestos with Heat", **Microscopy and Analysis**, 10, 1989, pp. 7-12.

15. Laughlin, G. and W. C. McCrone, "The Effect of Heat on the Microscopical Properties of Asbestos", **The Microscope**, 37, 1989, pp. 8-15.

16. **Interim Method for the Determination of Asbestos in Bulk Insulation Samples**, U.S. E.P.A. 600/M4-82-020, 1982.

17. McCrone, Walter C., "Routine Detection and Identification of Asbestos", **The Microscope**, 33, 1985, pp. 273-284

18. Reports of Analysis, SRM 1866 and 1867, National Institute of Standards & Technology.

19. McCrone, Walter C., **Asbestos Identification**, McCrone Research Institute, 1987.

2.3 Gravimetry

2.3.1 Principle and Applicability

Many components of bulk building materials, specifically binder components, can be selectively removed using appropriate solvents or, in the case of some organics, by ashing. The removal of these components serves the following purposes:

1) to isolate asbestos from the sample, allowing its weight to be determined;

2) to concentrate asbestos and therefore lower the detection limit in the total sample;

3) to aid in the detection and identification of fibrous components; and,

4) to remove organic (ashable) fibers which are optically similar to asbestos.

Common binder materials which are removed easily using the techniques described include: 1) calcite, gypsum, magnesite, brucite, bassanite, portlandite, and dolomite, using hydrochloric acid, and 2) vinyl, cellulose, and other organic components, by ashing. The removal of the binder components results in a residue containing asbestos, if initially present, and any other non-soluble or non-ashable components which were present in the original sample. Unless the procedures employed result in the loss of some asbestos, the weight percent of the residue is the upper limit for the weight percent of asbestos in the sample.

This section describes the procedure for removing acid-soluble and ashable components, and for determining the weight percent of the residue. However, the acid dissolution and ashing techniques can be used without the accompanying weight measurements to either liberate or clean fibers to aid in qualitative PLM or AEM analyses.

This technique is not an identification technique. Other methods, such as PLM, XRD, or AEM must be used to determine the identity of the components. A description of the suggested apparatus, reagents, etc. needed for the techniques described is included in Appendix B.

2.3.2 Interferences

Any components which cannot by removed from the sample by selective dissolution or ashing interfere with asbestos quantitation. These components include, but are not limited to, many silicates (micas, glass fibers, etc.) and oxides (TiO_2, magnetite, etc.). When interfering phases are present (the residue contains other phases in addition to asbestos), other techniques such as PLM, AEM, or XRD must be used to determine the percent of asbestos in the residue.

Care must be taken to prevent loss of or chemical/structural changes in the critical components (asbestos). Prolonged exposure to acids or excessive heating (above 500°C) can cause changes in the asbestos components in the sample and affect the optical properties.[1,2,3]

2.3.3 Quantitation

The weight of the residue remaining after solvent dissolution/ashing should be compared with the original weight of the material. Presuming no insoluble material is lost, the weight percent of the residue is the upper limit for the amount of asbestos in the sample. If the residue is comprised only of asbestos, then the weight percent of residue equals the weight percent of asbestos in the sample. If the residue contains other phases, then techniques such as PLM, XRD, or AEM must be employed to determine the relative abundance of asbestos in the residue.

The precision and accuracy of the technique are dependent upon the homogeneity of the material, the accuracy of the weight measurements, and the effectiveness of the sample reduction and filtering procedures. In practice, the precision can be equal to $\pm 1\%$, and the accuracy at 1 wt% asbestos can be less than or equal to $\pm 10\%$ relative.

The incomplete solution of components and the presence of other nonasbestos components in the residue contribute to producing a positive bias for the technique (falsely high percentages of asbestos).

2.3.4 Preliminary Examination and Evaluation

Stereomicroscopic and PLM examinations of the sample should already have been conducted prior to initiating this procedure. These examinations should have provided information about: 1) whether the sample contains components which can be removed by acid-washing, solvent dissolution, or ashing, and 2) whether the sample contains asbestos, or fibers that might be asbestos, or whether no asbestos was detected.

If the sample is friable and contains organic (ashable) components, the ashing procedure should be followed. If the sample is friable and contains HCl-soluble components, the acid dissolution procedure should be followed. If the sample is friable and contains both types of

components, the two procedures can be applied, preferably with acid dissolution following ashing.

If the sample is nonfriable (e.g. floor tiles), it is also recommended that the ashing procedure be used first, followed by the acid dissolution procedure. The ashing procedure reduces floor tiles to a material which is easily powdered, simplifying the sample preparation for acid dissolution.

2.3.5 Sample Preparation

2.3.5.1 Drying

Any moisture in the sample will affect the weight measurements, producing falsely low percentages of residue. If the sample is obviously wet, it should be dried at low temperature (using a heat lamp, or simply by exposure at ambient conditions, prior to starting the weighing procedure). If an oven is used, the drying temperature should not exceed 60°C. Drying by means of heat lamp or ambient air must be performed within a safety-filtered hood. Even if the sample appears dry, it can contain enough moisture to affect the precision and accuracy of the technique. The test for sample moisture involves placing the amount of sample to be used on the weighing pan; if the weight remains stable with time, then the sample is dry enough. If the weight decreases as the sample sits on the weighing pan, then the sample should be dried. Where conditions of moderate to high humidity are known to exist, all materials to be weighed should be allowed time to stabilize to these ambient conditions.

2.3.5.2 Homogenization/Grain Size Reduction

To increase the accuracy and precision of the acid dissolution technique, the sample should be homogenized prior to analysis. This reduces the grain size of the binder material and releases it from fiber bundles so that it may be dissolved in a shorter time period. Leaving the sample in the acid for a longer period of time to complete the dissolution process can adversely affect the asbestos components, and is not recommended. Homogenization of the sample also ensures that any material removed for analysis will more likely be representative of the entire sample.

Homogenization of friable samples prior to ashing may also accelerate the ashing process; however, the ashing time can simply be increased without affecting the asbestos in the sample. Nonfriable samples, such as vinyl floor tiles, can be broken or shaved into pieces to increase surface area and accelerate the ashing process.

Homogenization and grain size reduction can be accomplished in a variety of ways: 1) hand grinding in a mortar and pestle; 2) crushing with pliers or similar instrument; 3) mixing in a blender; 4) milling (i.e. Wylie mill, cryomill, etc.); or 5) any other technique which seems suitable. If the fibers are extremely long, a pair of scissors or similar implement can be used to reduce the fiber length.

2.3.6 Procedure for Ashing

1) **Weigh appropriate amount of material.**

   There is no restriction on the maximum weight of material used; however, a large amount of material may take longer to ash. Enough material should be used to avoid a significant contribution of weighing errors to the total accuracy and precision.

2) **Place material in crucible, weigh, and cover with lid.**

   Placing a lid on the crucible both minimizes the amount of oxygen available, slowing the rate of combustion of the sample, and prevents any foreign material from falling into the crucible during ashing.

3) **Place crucible into furnace, and ash for at least 6 hours.**

   The furnace temperature at the sample position should be at least 300°C but should not exceed 500°C. If the sample combusts (burns), the temperature of the sample may exceed 500°C. Chrysotile will decompose above approximately 500°C.

   The furnace area should be well-ventilated and the fumes produced by ashing should be exhausted outside the building.

   The ashing time is dependent on the furnace temperature, the amount of sample, and the surface area (grain size). Six hours at 450°C is usually sufficient.

4) **Remove crucible from furnace, allow contents to adjust to room temperature and humidity, and weigh.**

5) **Divide residue weight by starting weight and multiply by 100 to determine weight% residue.**

6) **Analyze residue and/or proceed to acid dissolution procedure.**

   If the objective was to remove organic fibers that may be confused optically with asbestos, examine residue with PLM to determine whether any fibers remain.

   If the sample is a floor tile, the acid dissolution procedure must now be performed. The residue does not have to be analyzed at this stage.

2.3.7 Use of Solvents for Removal of Organics

Solvent dissolution may be used as a substitute for low temperature ashing for the purpose of removing organic interferences from bulk building materials. However, solvent dissolution, because of the involvement of potentially hazardous reagents such as tetrahydrofuran, amyl acetate, 1-1-1, trichlorethane, etc., requires **that all work be performed with extreme caution inside a biohazard hood.** Material Safety Data Sheets should be reviewed before using any solvent. Solvent dissolution involves more apparatus than does ashing, and requires more time, mainly due to set-up and slow filtration resulting from viscous solvent/residue mixtures.

The following is a brief description of the solvent dissolution process.

1) **Weigh starting material.**

   Place approximately 15-25ml of solvent in a 100ml beaker. Add 2.5-3.0 grams (carefully weighed for continued gravimetric tracking) of powdered sample.

2) **Untrasonicate sample.**

   Place the beaker in an ultrasonic bath (or ultrasonic stirrer) for approximately 0.5 hours. The sample containers should be covered to preclude escape of an aerosol spray.

3) **Centrifuge sample.**

   Weigh centrifuge vial before adding beaker ingredients. Wash beaker with an additional 10-15ml of solvent to remove any remaining concentrate. Then centrifuge

at approximately 2000-2500 rpm for 0.5 hour. Use solvent-resistant centrifuge tubes.

4) **Decant sample, reweigh.**

After separation by centrifuging, decant solvent by pipetting. Leave a small amount of solvent in the centrifuge vial to minimize the risk of decanting solid concentrate. Allow solid concentrate to dry in vial, then reweigh.

2.3.8 Procedure for Acid Dissolution

1) **Weigh starting material, transfer to acid resistant container.**

Small, dry sample weights between 0.1g and 0.5g are recommended (determined for 47mm filters - adjust amount if different diameter filters are used). If too much material is left after acid dissolution the filter can get clogged and prevent complete filtration. Very small samples are also to be avoided, as the weighing errors will have a large effect on the total accuracy and precision of the technique.

2) **Weigh filter.**

3) **Add HCl to sample in container, stir, allow to sit for 2-10 minutes.**

Either concentrated or dilute HCl can be used. If concentrated HCl is used, add enough acid to completely soak the material, allow the reaction to proceed to completion, and then dilute with distilled water. Alternatively, a dilute solution, made by adding concentrated HCl to distilled water, can be used in the place of concentrated HCl. A solution of 1 part concentrated HCl to 3 parts distilled water (approximately 3N solution) has been found to be quite effective in removing components within 5 minutes. For a sample size less than 0.5g, 20-30 ml of a 3N HCl solution is appropriate. In either case (using concentrated or dilute HCl), the reaction will be more effective if the sample has been homogenized first. All obvious signs of reaction (bubbling) should cease before the sample is filtered. Add fresh acid, a ml or two at a time, to ensure complete reaction. It should be noted that if dolomite is present, a 15-20 minute exposure to concentrated HCl may be required to completely dissolve the carbonate materials.

NOTE: Other solvents may be useful for selective dissolution of nonasbestos components. For example, acetic acid will dissolve calcite, and will not dissolve asbestos minerals. If any solvent other than hydrochloric acid is used for the dissolution of inorganic components, the laboratory must be able to demonstrate that the solvent does not remove asbestos from the sample.

4) **Filter solution.**

Use the pre-weighed filter. Pour the solution into the vacuum filter assembly, then rinse all material from container into filter assembly. Rinse down the inside walls of the glass filter basin and check for particles clinging to the basin after removal.

5) **Weigh dried filter + residue, subtract weight of filter from total.**

6) **Divide residue weight by starting weight and multiply by 100 to determine weight% residue.**

7) **Analyze residue.**

Perform stereomicroscopic examination of residue (can be performed without removing the residue from the filter). Note in particular whether any binder material is still present.

Perform PLM, AEM, or XRD analysis of residue to identify fibers and determine concentration as described in the appropriate sections of this method.

8) **Modify procedure if necessary.**

If removal of the acid soluble components was not complete, start with a new subsample of material and try any of the following:

a) Decrease grain size of material (by grinding, milling, etc.)
b) Put solutions on hot plate - warm slightly
c) Increase soak time (exercise caution)

9) **Calculate relative weight% asbestos in sample.**

wt% asbestos in sample = % asbestos in residue x wt% residue ÷ 100

For floor tiles, if the ashing procedure was used first, multiply the weight % of asbestos in the sample, as determined above, by the weight percent of the residue from the ashing procedure, then divide by 100.

Example:
A = wt% residue from ashing = 70%
B = wt% residue from HCl = 20%
C = wt% of asbestos in HCl residue = 50%

wt% asbestos after HCl dissolution = B x C ÷ 100 = 20 x 50 ÷ 100 = 10%

wt% asbestos in floor tile = (B x C ÷ 100) x A ÷ 100 = 10 x 70 ÷ 100 = 7%

If weights are expressed in decimal form, multiply the weight % of asbestos in the sample by the weight % of the residue from the ashing procedure, then multiply by 100.

wt% asbestos after HCl dissolution = B x C = 0.2 x 0.5 = 0.1 (x 100 = 10%)

wt% asbestos in floor tile = (B x C) x A = 0.1 x 0.7 = 0.07 (x 100 = 7%)

2.3.9 Determination of Optimal Precision and Accuracy

The precision of the technique can be determined by extracting multiple subsamples from the original sample and applying the same procedure to each. The optimal accuracy of the technique can be determined by applying gravimetric standards. Mixtures of calcite and asbestos (chrysotile, amosite, etc.) in the following proportions are recommended for testing the accuracy of the acid dissolution technique: 0.1 wt% asbestos/99.9 wt% calcite, 1.0 wt% asbestos/99.0 wt% calcite, and 10 wt% asbestos/90 wt% calcite. Mixtures of cellulose and asbestos are useful for testing the accuracy of the ashing technique.

Mixtures of only two components, as described above, are simplifications of "real-world" samples. The accuracy determined by analyzing these mixtures is considered optimal and may not apply directly to the measurement of each unknown sample. However, analyzing replicates and standards using the full laboratory procedure, including homogenization, ashing, acid dissolution, filtration, and weighing, may uncover steps that introduce significant bias or variation that the laboratory may then correct.

2.3.10 References

1. Kressler, J. R., "Changes in Optical Properties of Chrysotile During Acid Leaching", **The Microscope**, 31, 1983, pp. 165-172.

2. Prentice, J. and M. Keech, "Alteration of Asbestos with Heat", **Microscopy and Analysis,** March 1989.

3. Laughlin, G. and W. C. McCrone, "The Effect of Heat on the Microscopical Properties of Asbestos", **The Microscope,** 37, 1989, pp. 8-15.

2.4 X-Ray Powder Diffraction

2.4.1 Principle and Applicability

The principle of x-ray powder diffraction (XRD) analysis is well established.[1,2] Any solid crystalline material will diffract an incident beam of parallel, monochromatic x-rays whenever Bragg's Law,

$$\lambda = 2d \sin \theta,$$

is satisfied for a particular set of planes in the crystal lattice, where

λ = the x-ray wavelength, Å;
d = the interplanar spacings of the set of reflecting lattice planes, Å and
θ = the angle of incidence between the x-ray beam and the reflecting lattice planes.

By appropriate orientation of a sample relative to the incident x-ray beam, a diffraction pattern can be generated that will be uniquely characteristic of the structure of the crystalline phases present.

Unlike optical methods of analysis, however, XRD cannot determine crystal morphology. Therefore, in asbestos analysis, XRD does not distinguish between fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-6). However, when used in conjunction with methods such as PLM or AEM, XRD techniques can provide a reliable analytical method for the identification and characterization of asbestiform minerals in bulk materials.

For **qualitative** analysis by XRD methods, samples should initially be scanned over limited diagnostic peak regions for the serpentine (~7.4 Å) and amphibole (8.2-8.5 Å) minerals (Table 2-7). Standard slow-scanning methods for bulk sample analysis may be used for materials shown by PLM to contain significant amounts of asbestos (>5 percent). Detection of minor or trace amounts of asbestos may require special sample preparation and step-scanning analysis. All samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals should be submitted to a full (5°-60° 2θ; 1° 2θ/min) qualitative XRD scan, and their diffraction patterns should be compared with standard reference powder

diffraction patterns[3] to verify initial peak assignments and to identify possible matrix interferences when subsequent quantitative analysis will be performed.

Accurate **quantitative** analysis of asbestos in bulk samples by XRD is critically dependent on particle size distribution, crystallite size, preferred orientation and matrix absorption effects, and comparability of standard reference and sample materials. The most intense diffraction peak that has been shown to be free from interference by prior qualitative XRD analysis should be selected for quantitation of each asbestiform mineral. A "thin-layer" method of analysis[5,6] can be used in which, subsequent to comminution of the bulk material to $\sim 10\ \mu m$ by suitable cryogenic milling techniques, an accurately known amount of the sample is deposited on a silver membrane filter. The mass of asbestiform material is determined by measuring the integrated area of the selected diffraction peak using a step-scanning mode, correcting for matrix absorption effects, and comparing with suitable calibration standards. Alternative "thick-layer" or bulk methods[7,8] are commonly used for **semi-quantitative analysis.**

TABLE 2-6. THE ASBESTOS MINERALS AND THEIR NONASBESTIFORM ANALOGS

Asbestiform	Nonasbestiform	Chemical Abstract Service No.
Serpentine		
Chrysotile	Antigorite, lizardite	12001-29-5
Amphibole		
Anthophyllite asbestos	Anthophyllite	77536-67-5
Cummingtonite-grunerite asbestos (Amosite)	Cummingtonite-grunerite	12172-73-5
Crocidolite	Riebeckite	12001-28-4
Tremolite asbestos	Tremolite	77536-68-6
Actinolite asbestos	Actinolite	77536-66-4

TABLE 2-7. PRINCIPAL LATTICE SPACINGS OF ASBESTIFORM MINERALS[1]

Minerals	Principal d-spacings (Å) and relative intensities			JCPDS Powder diffraction file[2] number
Chrysotile (Serpentine)	7.31_{100} 7.36_{100} 7.10_{100}	3.65_{70} 3.66_{80} 2.33_{80}	4.57_{50} 2.45_{65} 3.55_{70}	21-543[3] 25-645 22-1162 (theoretical)
Amosite (Grunerite)	8.33_{100} 8.22_{100}	3.06_{70} 3.060_{85}	2.756_{70} 3.25_{70}	17-745 (nonfibrous) 27-1170 (UICC)
Anthophyllite	3.05_{100} 3.06_{100}	3.24_{60} 8.33_{70}	8.26_{55} 3.23_{50}	9-455 16-401 (synthetic)
Crocidolite (Riebeckite)	8.35_{100} 8.40_{100}	3.10_{55} 3.12_{55}	2.720_{35} 2.726_{40}	27-1415 (UICC) 19-1061
Actinolite	2.72_{100}	2.54_{100}	3.40_{80}	25-157
Tremolite	8.38_{100} 2.706_{100} 3.13_{100}	3.12_{100} 3.14_{95} 2.706_{60}	2.705_{90} 8.43_{40} 8.44_{40}	13-437[3] 20-1310[3] (synthetic) 23-666 (synthetic mixture w/richterite)

1. This information is intended as a guide only. Complete powder diffraction data, including mineral type and source, should be referred to ensure comparability of sample and reference materials where possible. Additional precision XRD data on amosite, crocidolite, tremolite and chrysotile are available from the U.S. Bureau of Mines, Reference 4.

2. From Reference 3

3. Fibrosity questionable

This XRD method is applicable as a confirmatory method for identification and quantitation of asbestos in bulk material samples that have undergone prior analysis by PLM or other optical methods.

2.4.2 Range and Sensitivity

The range and sensitivity of the method have not been determined. They will be variable and dependent upon many factors, including matrix effects (absorption and interferences), diagnostic reflections selected and their relative intensities, preferred orientation, and instrumental limitations. A detection limit of one percent is feasible given certain sample characteristics.

2.4.3 Limitations

2.4.3.1 Interferences

Since the asbestiform and nonasbestiform analogs of the serpentine and amphibole minerals (Table 2-7) are indistinguishable by XRD techniques unless special sample preparation techniques and instrumentation are used,[9] the presence of nonasbestiform serpentines and amphiboles in a sample will pose severe interference problems in the identification and quantitative analysis of their asbestiform analogs.

The use of XRD for identification and quantitation of asbestiform minerals in bulk samples may also be limited by the presence of other interfering materials in the sample. For naturally-occurring materials, the commonly associated asbestos-related mineral interferences can usually be anticipated. However, for fabricated materials, the nature of the interferences may vary greatly (Table 2-8) and present more serious problems in identification and quantitation.[10] Potential interferences are summarized in Table 2-9 and include the following:

- **Chlorite** has major peaks at 7.19 Å and 3.58 Å that interfere with both the primary (7.31 Å) and secondary (3.65 Å) peaks for serpentine (chrysotile). Resolution of the primary peak to give good quantitative results may be possible when a step-scanning mode of operation is employed.

- **Vermiculite** has secondary peaks at 7.14 Å and 3.56 Å that could interfere with the primary peak (7.31 Å) and a secondary peak (3.65 Å) of serpentine (chrysotile).

TABLE 2-8. COMMON CONSTITUENTS IN BUILDING MATERIAL
(From Ref. 10)

A. Insulation Materials

Chrysotile
Amosite
Crocidolite
*Rock wool
*Slag wool
*Fiber glass
Gypsum ($CaSO_4 \cdot 2H_2O$)
Vermiculite (micas)
*Perlite
Clays (kaolin)
*Wood pulp
*Paper fibers (talc, clay carbonate filters)
Calcium silicates (synthetic)
Opaques (chromite, magnetite inclusions in serpentine)
Hematite (inclusions in "amosite")
Magnesite
*Diatomaceous earth

B. Flooring Materials

Calcite
Dolomite
Titanium Oxide
Quartz
Antigorite
Chrysotile
Anthophyllite
Tremolite
*Organic binders
Talc
Wollastonite

C. Spray Finishes or Paints

Bassanite
Carbonate minerals (calcite, dolomite, vaterite)
Talc
Tremolite
Anthophyllite
Serpentine (including chrysotile)
Amosite
Crocidolite
*Mineral wool
*Rock wool
*Slag wool
*Fiber glass
Clays (kaolin)
Micas
Chlorite
Gypsum
Quartz
*Organic binders and thickeners
Hydromagnesite
Wollastonite
Opaques (chromite, magnetite inclusion in serpentine)
Hematite (inclusions in "amosite")

D. Cementitious Materials

Chrysotile
Amosite
Crocidolite
Micas
Fiber glass
Cellulose
Animal hair
Quartz
Gypsum
Calcite
Dolomite
Calcium silicates

E. Roofing Materials

Chrysotile
Cellulose
Fiber glass
Mineral Wool
Asphalt
Quartz
Talc
Micas

* Amorphous materials--contribute only to overall scattered radiation and increased background radiation.

TABLE 2-9 INTERFERENCES IN XRD ANALYSIS OF ASBESTIFORM MINERALS

Asbestiform Mineral	Primary diagnostic peaks (approximate d spacings in Å)	Interference
Serpentine Chrysotile	7.3	Nonasbestiform serpentines, (antigorite, lizardite), chlorite, vermiculite, sepiolite, kaolinite, gypsum
	3.7	Nonasbestiform serpentines (antigorite, lizardite), chlorite, vermiculite, halloysite, cellulose
Amphibole Amosite (Grunerite) Anthophyllite Crocidolite (Riebeckite) Tremolite Actinolite	3.1	Nonasbestiform amphiboles (grunerite-cummingtonite, anthophyllite, riebeckite, tremolite), mutual interferences, talc, carbonates
	8.3	Nonasbestiform amphiboles (grunerite-cummingtonite, anthophyllite, riebeckite, tremolite), mutual interferences

- **Sepiolite** produces a peak at 7.47 Å which could interfere with the primary peak (7.31 Å) of serpentine (chrysotile).

- **Halloysite** has a peak at 3.63 Å that interferes with the secondary (3.65 Å) peak for serpentine (chrysotile).

- **Kaolinite** has a major peak at 7.15 Å that may interfere with the primary peak of serpentine (chrysotile) at 7.31 Å when present at concentrations of > 10 percent. However, the secondary serpentine (chrysotile) peak at 3.65 Å may be used for quantitation.

- **Gypsum** has a major peak at 7.5 Å that overlaps the 7.31 Å peak of serpentine (chrysotile) when present as a major sample constituent. This may be removed by careful washing with distilled water, or by heating to 300°C to convert gypsum to plaster of paris (bassanite).

- **Cellulose** has a broad peak that partially overlaps the secondary (3.65 Å) serpentine (chrysotile) peak.[8]

- Overlap of major diagnostic peaks of the amphibole minerals, grunerite (amosite), anthophyllite, riebeckite (crocidolite), and tremolite, at approximately 8.3 Å and 3.1 Å causes mutual interference when these minerals occur in the presence of one another. In some instances adequate resolution may be attained by using step-scanning methods and/or by decreasing the collimator slit width at the x-ray port.

- **Carbonates** may also interfere with quantitative analysis of the amphibole minerals grunerite (amosite), anthophyllite, riebeckite (crocidolite), and tremolite-actinolite. Calcium carbonate ($CaCO_3$) has a peak at 3.035 Å that overlaps major amphibole peaks at approximately 3.1 Å when present in concentrations of >5 percent. Removal of carbonates with a dilute acid wash is possible; however, the time in acid should be no more than 20 minutes to preclude any loss of chrysotile.[11]

- A major **talc** peak at 3.12 Å interferes with the primary tremolite peak at this same position and with secondary peaks of actinolite (3.14 Å), riebeckite (crocidolite) (3.10 Å), grunerite (amosite) (3.06 Å), and anthophyllite (3.05 Å). In the presence of talc, the major diagnostic peak at approximately 8.3 Å should be used for quantitation of these asbestiform minerals.

The problem of intraspecies and matrix interference is further aggravated by the variability of the silicate mineral powder diffraction patterns themselves, which often makes definitive identification of the asbestos minerals by comparison with standard reference diffraction patterns difficult. This variability results from alterations in the crystal lattice associated with differences in isomorphous substitution and degree of crystallinity. This is especially true for the amphiboles. These minerals exhibit a wide variety of very similar chemical compositions, resulting in diffraction patterns characterized by having major (110) reflections of the monoclinic amphiboles and (210) reflections of orthorhombic anthophyllite separated by less than 0.2 Å.[12]

2.4.3.2 Matrix Effects

If a copper x-ray source is used, the presence of iron at high concentrations in a sample will result in significant x-ray fluorescence, leading to loss of peak intensity, increased background intensity, and an overall decrease in sensitivity. This situation may be corrected by use of an x-ray source other than copper; however, this is often accompanied both by loss of intensity and by decreased resolution of closely spaced reflections. Alternatively, use of a

diffracted beam monochromator will reduce background fluorescent radiation, enabling weaker diffraction peaks to be detected.

X-ray absorption by the sample matrix will result in overall attenuation of the diffracted beam and may seriously interfere with quantitative analysis. Absorption effects may be minimized by using sufficiently "thin" samples for analysis.[5,13,14] However, unless absorption effects are known to be the same for both samples and standards, appropriate corrections should be made by referencing diagnostic peak areas to an internal standard[7,8] or filter substrate (Ag) peak.[5,6]

2.4.3.3 Particle Size Dependence

Because the intensity of diffracted x-radiation is particle-size dependent, it is essential for accurate quantitative analysis that both sample and standard reference materials have similar particle size distributions. The optimum particle size (i.e., fiber length) range for quantitative analysis of asbestos by XRD has been reported to be 1 to 10 μm.[15] Comparability of sample and standard reference material particle size distributions should be verified by optical microscopy (or another suitable method) prior to analysis.

2.4.3.4 Preferred Orientation Effects

Preferred orientation of asbestiform minerals during sample preparation often poses a serious problem in quantitative analysis by XRD. A number of techniques have been developed for reducing preferred orientation effects in "thick layer" samples.[7,8,15] For "thin" samples on membrane filters, the preferred orientation effects seem to be both reproducible and favorable to enhancement of the principal diagnostic reflections of asbestos minerals, actually increasing the overall sensitivity of the method.[12,14] However, further investigation into preferred orientation effects in both thin layer and bulk samples is required.

2.4.3.5 Lack of Suitably Characterized Standard Materials

The problem of obtaining and characterizing suitable reference materials for asbestos analysis is clearly recognized. The National Institute of Standards and Technology can

provide standard reference materials for chrysotile, amosite and crocidolite (SRM 1866) and anthophyllite, tremolite and actinolite (SRM 1867).

In addition, the problem of ensuring the comparability of standard reference and sample materials, particularly regarding crystallite size, particle size distribution, and degree of crystallinity, has yet to be adequately addressed. For example, Langer et al.[18] have observed that in insulating matrices, chrysotile tends to break open into bundles more frequently than amphiboles. This results in a line-broadening effect with a resultant decrease in sensitivity. Unless this effect is the same for both standard and sample materials, the amount of chrysotile in the sample will be under-estimated by XRD analysis. To minimize this problem, it is recommended that standardized matrix reduction procedures be used for both sample and standard materials.

2.4.4 Precision and Accuracy

Neither the precision nor accuracy of this method has been determined. The individual laboratory should obtain or prepare a set of calibration materials containing a range of asbestos weight percent concentrations in combination with a variety of matrix/binder materials. Calibration curves may be constructed for use in semi-quantitative analysis of bulk materials.

2.4.5 Procedure

2.4.5.1 Sampling

Samples taken for analysis of asbestos content should be collected as specified by EPA[19]

2.4.5.2 Analysis

All samples must be analyzed initially for asbestos content by PLM. XRD may be used as an additional technique, both for identification and quantitation of sample components. **Note: Asbestos is a toxic substance. All handling of dry materials should be performed in a safety-hood.**

2.4.5.2.1 Sample Preparation

The method of sample preparation required for XRD analysis will depend on: (1) the condition of the sample received (sample size, homogeneity, particle size distribution, and overall composition as determined by PLM); and (2) the type of XRD analysis to be performed (qualitative or quantitative; thin-layer or bulk).

Bulk materials are usually received as heterogeneous mixtures of complex composition with very wide particle size distributions. Preparation of a homogeneous, representative sample from asbestos-containing materials is particularly difficult because the fibrous nature of the asbestos minerals inhibits mechanical mixing and stirring, and because milling procedures may cause adverse lattice alterations.

A discussion of specific matrix reduction procedures is given below. Complete methods of sample preparation are detailed in Sections 2.4.5.3 and 2.4.5.4. **Note: All samples should be examined microscopically before and after each matrix reduction step to monitor changes in sample particle size distribution, composition, and crystallinity, and to ensure sample representativeness and homogeneity for analysis.**

2.4.5.2.2 Milling

Mechanical milling of asbestos materials has been shown to decrease fiber crystallinity, with a resultant decrease in diffraction intensity of the specimen; the degree of lattice alteration is related to the duration and type of milling process.[20-23] Therefore, all milling times should be kept to a minimum.

For **qualitative analysis**, particle size is not usually of critical importance and initial characterization of the material with a minimum of matrix reduction is often desirable to document the composition of the sample as received. Bulk samples of very large particle size (>2-3 mm) should be comminuted to ~ 100 μm. A mortar and pestle can sometimes be used in size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced by cutting with a razor blade in a mortar, or by grinding in a suitable mill (e.g., a microhammer mill or equivalent). When using a mortar for grinding or cutting, the sample should be moistened with ethanol, or some other

suitable wetting agent, to minimize exposure, and the procedure should be performed in a HEPA-filtered hood.

For accurate, reproducible **quantitative analysis**, the particle size of both sample and standard materials should be reduced to $\sim 10\ \mu m$. Dry ball milling at liquid nitrogen temperatures (e.g., Spex Freezer Mill*, or equivalent) for a maximum time of 10 minutes (some samples may require much shorter milling time) is recommended to obtain satisfactory particle size distributions while protecting the integrity of the crystal lattice.[5] Bulk samples of very large particle size may require grinding in two stages for full matrix reduction to $< 10\ \mu m$.[8,16]

Final particle size distributions should always be verified by optical microscopy or another suitable method.

2.4.5.2.3 Ashing

For materials shown by PLM to contain large amounts of cellulose or other organic materials, it may be desirable to ash prior to analysis to reduce background radiation or matrix interference. Since chrysotile undergoes dehydroxylation at temperatures between 550°C and 650°C, with subsequent transformation to forsterite,[24,25] ashing temperatures should be kept below 500°C. Use of a muffle furnace is recommended. In all cases, calibration of the furnace is essential to ensure that a maximum ashing temperature of 500°C is not exceeded (see Section 2.3).

2.4.5.2.4 Acid Washing

Because of the interference caused by gypsum and some carbonates in the detection of asbestiform minerals by XRD (see Section 2.4.3.1), it may be necessary to remove these interferences by a simple acid washing procedure prior to analysis (see Section 2.3).

2.4.5.3 Qualitative Analysis

2.4.5.3.1 Initial Screening of Bulk Material

Qualitative analysis should be performed on a representative, homogeneous portion of the sample, with a minimum of sample treatment, using the following procedure:

1. Grind and mix the sample with a mortar and pestle (or equivalent method, see Section 2.4.5.2.2) to a final particle size sufficiently small ($\sim 100\ \mu m$) to allow adequate packing into a sample holder.

2. Pack sample into a standard bulk sample holder. Care should be taken to ensure that a representative portion of the milled sample is selected for analysis. Particular care should be taken to avoid possible size segregation of the sample. **(Note: Use of back-packing method[26] for bulk sample preparation may reduce preferred orientation effects.)**

3. Mount the sample on the diffractometer and scan over the diagnostic peak regions for the serpentine (~ 7.4 Å) and amphibole (8.2-8.5 Å) minerals (see Table 2-7). The x-ray diffraction equipment should be optimized for intensity. A slow scanning speed of $1°\ 2\theta$/min is recommended for adequate resolution. Use of a sample spinner is recommended.

4. Submit all samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals to a full qualitative XRD scan ($5°$-$60°\ 2\theta$; $1°\ 2\theta$/min) to verify initial peak assignments and to identify potential matrix interferences when subsequent quantitative analysis is to be performed.

5. Compare the sample XRD pattern with standard reference powder diffraction patterns (i.e., JCPDS powder diffraction data[3] or those of other well-characterized reference materials). Principal lattice spacings of asbestiform minerals are given in Table 2-7; common constituents of bulk insulation and wall materials are listed in Table 2-8.

2.4.5.3.2 Detection of Minor or Trace Constituents

Routine screening of bulk materials by XRD may fail to detect small concentrations ($<1\%$) of asbestos. The limits of detection will, in general, be improved if matrix absorption effects are minimized, and if the sample particle size is reduced to the optimal 1 to 10 μm range, provided that the crystal lattice is not degraded in the milling process. Therefore, in those instances when confirmation of the presence of an asbestiform mineral at very low levels is required, or where a negative result from initial screening of the bulk material by XRD (see Section 2.4.5.3.1) is in conflict with previous PLM results, it may be desirable to prepare the sample as described for quantitative analysis (see Section 2.4.5.4) and step-scan over appropriate 2θ ranges of selected diagnostic peaks (Table 2-7). Accurate

transfer of the sample to the silver membrane filter is not necessary unless subsequent quantitative analysis is to be performed.

2.4.5.4 Quantitative Analysis

The proposed method for quantitation of asbestos in bulk samples is a modification of the NIOSH-recommended thin-layer method for chrysotile in air.[6] A thick-layer bulk method involving pelletizing the sample may be used for semi-quantitative analysis;[7,8] however, this method requires the addition of an internal standard, use of a specially fabricated sample press, and relatively large amounts of standard reference materials. Additional research is required to evaluate the comparability of thin- and thick-layer methods for quantitative asbestos analysis.

For quantitative analysis by thin-layer methods, the following procedure is recommended:

1. Mill and size all or a substantial representative portion of the sample as outlined in Section 2.4.5.2.2.

2. Dry at 60°C for 2 hours; cool in a desiccator.

3. Weigh accurately to the nearest 0.01 mg.

4. Samples shown by PLM to contain large amounts of cellulosic or other organic materials, gypsum, or carbonates, should be submitted to appropriate matrix reduction procedures described in Sections 2.4.5.2.3 and 2.4.5.2.4. After ashing and/or acid treatment, repeat the drying and weighing procedures described above, and determine the percent weight loss, L.

5. Quantitatively transfer an accurately weighed amount (50-100 mg) of the sample to a 1-L volumetric flask containing approximately 200 mL isopropanol to which 3 to 4 drops of surfactant have been added.

6. Ultrasonicate for 10 minutes at a power density of approximately 0.1 W/mL, to disperse the sample material.

7. Dilute to volume with isopropanol.

8. Place flask on a magnetic-stirring plate. Stir.

9. Place silver membrane filter on the filtration apparatus, apply a vacuum, and attach the reservoir. Release the vacuum and add several milliliters of isopropanol to the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw

an aliquot from the center of the suspension so that total sample weight, W_T, on the filter will be approximately 1 mg. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and repeat the procedure with a clean pipet. Transfer the aliquot to the reservoir. Filter rapidly under vacuum. Do not wash the reservoir walls. Leave the filter apparatus under vacuum until dry. Remove the reservoir, release the vacuum, and remove the filter with forceps. (Note: Water-soluble matrix interferences such as gypsum may be removed at this time by careful washing of the filtrate with distilled water. Extreme care should be taken not to disturb the sample.)

10. Attach the filter to a flat holder with a suitable adhesive and place on the diffractometer. Use of a sample spinner is recommended.

11. For each asbestos mineral to be quantitated, select a reflection (or reflections) that has (have) been shown to be free from interferences by prior PLM or qualitative XRD analysis and that can be used unambiguously as an index of the amount of material present in the sample (see Table 2-7).

12. Analyze the selected diagnostic reflection(s) by step-scanning in increments of 0.02° 2θ for an appropriate fixed time and integrating the counts. (A fixed count scan may be used alternatively; however, the method chosen should be used consistently for all samples and standards.) An appropriate scanning interval should be selected for each peak, and background corrections made. For a fixed time scan, measure the background on each side of the peak for one-half the peak-scanning time. The net intensity, I_a, is the difference between the peak integrated count and the total background count.

13. Determine the net count, I_{Ag}, of the filter 2.36 Å silver peak following the procedure in step 12. Remove the filter from the holder, reverse it, and reattach it to the holder. Determine the net count for the unattenuated silver peak, I°_{Ag} Scan times may be less for measurement of silver peaks than for sample peaks; however, they should be constant throughout the analysis.

14. Normalize all raw, net intensities (to correct for instrument instabilities) by referencing them to an external standard (e.g., the 3.34 Å peak of an α-quartz reference crystal). After each unknown is scanned, determine the net count, I°_r, of the reference specimen following the procedure in step 12. Determine the normalized intensities by dividing the peak intensities by I°_r:

$$\hat{I}_a = \frac{I_a}{I_r^\circ}, \quad \hat{I}_{Ag} = \frac{I_{Ag}}{I_r^\circ}, \text{ and } \hat{I}_{Ag}^\circ = \frac{I_{Ag}^\circ}{I_r^\circ}$$

2.4.6 Calibration

2.4.6.1 Preparation of Calibration Standards

1. Mill and size standard asbestos materials according to the procedure outlined in Section 2.4.5.2.2. **Equivalent standardized matrix reduction and sizing techniques should be used for both standard and sample materials.**

2. Dry at 100°C for 2 hours; cool in a desiccator.

3. Prepare two suspensions of each standard in isopropanol by weighing approximately 10 and 50 mg of the dry material to the nearest 0.01 mg. Transfer each to a 1-L volumetric flask containing approximately 200 mL isopropanol to which a few drops of surfactant have been added.

4. Ultrasonicate for 10 minutes at a power density of approximately 0.1 W/mL, to disperse the asbestos material.

5. Dilute to volume with isopropanol.

6. Place the flask on a magnetic stirring plate. Stir.

7. Prepare, in triplicate, a series of at least five standard filters to cover the desired analytical range, using appropriate aliquots of the 10 and 50 mg/L suspensions. For each standard, mount a silver membrane filter on the filtration apparatus. Place a few mL of isopropanol in the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Keep the tip of the pipet near the surface of the isopropanol. Filter rapidly under vacuum. Do not wash the sides of the reservoir. Leave the vacuum on for a time sufficient to dry the filter. Release the vacuum and remove the filter with forceps.

2.4.6.2 Analysis of Calibration Standards

1. Mount each filter on a flat holder. Perform step scans on selected diagnostic reflections of the standards and reference specimen using the procedure outlined in Section 2.4.5.4, step 12, and the same conditions as those used for the samples.

2. Determine the normalized intensity for each peak measured, $\hat{I}^{\circ}_{std}$, as outlined in Section 2.4.5.4, step 14.

2.4.7 Calculations

For each asbestos reference material, calculate the exact weight deposited on each standard filter from the concentrations of the standard suspensions and aliquot volumes. Record the weight, w, of each standard. Prepare a calibration curve by regressing $\hat{I}^{\circ}_{std}$, on w. Poor reproducibility ($\pm$15 percent RSD) at any given level indicates problems in the sample preparation technique, and a need for new standards. The data should fit a straight-line equation.

Determine the slope, m, of the calibration curve in counts/microgram. The intercept, b, of the line with the $\hat{I}^{\circ}_{std}$ axis should be approximately zero. A large negative intercept indicates an error in determining the background. This may arise from incorrectly measuring the baseline or from interference by another phase at the angle of background measurement. A large positive intercept indicates an error in determining the baseline or that an impurity is included in the measured peak.

Using the normalized intensity, $\hat{I}_{Ag}$ for the attenuated silver peak of a sample, and the corresponding normalized intensity from the unattenuated silver peak $\hat{I}^{\circ}_{Ag}$, of the sample filter, calculate the transmittance, T, for each sample as follows:[27,28]

$$T = \frac{\hat{I}_{Ag}}{\hat{I}^{\circ}_{Ag}}$$

Determine the correction factor, f(T), for each sample according to the formula:

$$f(T) = \frac{-R(\ln T)}{1 - T^R}$$

where

$$R = \frac{\sin \theta_{Ag}}{\sin \theta_a}$$

θ_{Ag} = angular position of the measured silver peak (from Bragg's Law), and

θ_a = angular position of the diagnostic asbestos peak.

Calculate the weight, W_a, in micrograms, of the asbestos material analyzed for in each sample, using the absorption corrections:

$$W_a = \frac{\hat{I}_a f(t) - b}{m}$$

Calculate the percent composition, P_a, of each asbestos mineral analyzed for in the parent material, from the total sample weight, W_T, on the filter:

$$P_a = \frac{W_a(1 - .01L)}{W_T} \times 100$$

where

P_a = percent asbestos mineral in parent material;

W_a = mass of asbestos mineral on filter, in μg;

W_T = total sample weight on filter, in μg;

L = percent weight loss of parent material on ashing and/or acid treatment (see Section 2.4.5.4).

2.4.8 References

1. Klug, H. P. and L. E. Alexander, **X-Ray Diffraction Procedures for Polycrystalline and Amorphous Materials**, 2nd ed., New York: John Wiley and Sons, 1979.

2. Azaroff, L. V. and M. J. Buerger, **The Powder Method of X-Ray Crystallography**, New York: McGraw-Hill, 1958.

3. **JCPDS-International Center for Diffraction Data Powder Diffraction Studies**, 1601 Park Lane, Swarthmore, PA.

4. Campbell, W. J., C. W. Huggins, and A. G. Wylie, **Chemical and Physical Characterization of Amosite, Chrysotile, Crocidolite, and Nonfibrous Tremolite for National Institute of Environmental Health Sciences Oral Ingestion Studies**, U.S. Bureau of Mines Report of Investigation R18452, 1980.

5. Lange, B. A. and J. C. Haartz, Determination of microgram quantities of asbestos by x-ray diffraction: Chrysotile in thin dust layers of matrix material, **Anal. Chem.**, 51(4):529-525, 1979.

6. **NIOSH Manual of Analytical Methods**, Volume 5, U.S. Dept. HEW, August 1979, pp. 309-1 to 309-9.

7. Dunn, H.W. and J. H. Stewart, Jr., Determination of chrysotile in building materials by x-ray diffractometry, **Analytical Chemistry**, 54 (7); 1122-1125, 1982.

8. Taylor, A., Methods for the quantitative determination of asbestos and quartz in bulk samples using x-ray diffraction, **The Analyst**, 103(1231):1009-1020, 1978.

9. Birks, L., M. Fatemi, J. V. Gilfrich, and E. T. Johnson, **Quantitative Analysis of Airborne Asbestos by X-Ray Diffraction**, Naval Research Laboratory Report 7879, Naval Research Laboratory, Washington, DC, 1975.

10. **Asbestos-Containing Materials in School Buildings: A Guidance Document**, U. S. Environmental Protection Agency. EPA/OTS No. C00090, March 1979.

11. Krause, J. B. and W. H. Ashton, Misidentification of asbestos in talc, pp. 339-353, In: **Proceedings of Workshops on Asbestos: Definitions and Measurement Methods** (NBS Special Publication 506), C. C. Gravatt, P. D. LaFleur, and K. F. Heinrich (eds.), Washington, DC: National Measurement Laboratory, National Bureau of Standards, 1977 (issued 1978).

12. Stanley, H. D., The detection and identification of asbestos and asbestiform minerals in talc, pp. 325-337, In: **Proceedings of Workshop on Asbestos: Definitions and Measurement Methods** (NBS Special Publication 506), C. C. Gravatt, P. D. LaFleur, and K. F. Heinrich (eds.), Washington, DC: National Measurement Laboratory, National Bureau of Standards, 1977 (issued 1978).

13. Rickards, A. L., Estimation of trace amounts of chrysotile asbestos by x-ray diffraction, **Anal. Chem.**, 44(11):1872-3, 1972.

14. Cook, P. M., P. L. Smith, and D. G. Wilson, Amphibole fiber concentration and determination for a series of community air samples: use of x-ray diffraction to supplement electron microscope analysis, In: **Electron Microscopy and X-Ray Applications to Environmental and Occupation Health Analysis**, P. A. Russell and A. E. Hutchings (eds.), Ann Arbor: Ann Arbor Science Publications, 1977.

15. Rohl, A. N. and A. M. Langer, Identification and quantitation of asbestos in talc, **Environ. Health Perspectives**, 9:95-109, 1974.

16. Graf, J. L., P. K. Ase, and R. G. Draftz, **Preparation and Characterization of Analytical Reference Materials**, DHEW (NIOSH) Publication No. 79-139, June 1979.

17. Haartz, J. C., B. A. Lange, R. G. Draftz, and R. F. Scholl, Selection and characterization of fibrous and nonfibrous amphiboles for analytical methods development, pp. 295-312, In: **Proceedings of Workshop on Asbestos: Definitions and Measurement Methods** (NBS special Publication 506), C. C. Gravatt, P. D. LaFleur, and K. F. Heinrich (eds.), Washington, DC: National Measurement Laboratory, National Bureau of Standards, 1977 (issued 1978).

18. Personal Communication, A. M. Langer, formerly of Environmental Sciences Laboratory, Mount Sinai School of Medicine of the City University of New York, New York, NY, now of Brooklyn College, Brooklyn, N.Y.

19. **Asbestos in Buildings: Simplified Sampling Scheme for Friable Surfacing Materials**, U.S. Environmental Protection Agency. EPA 560/5-85-030a, October 1985.

20. Langer, A. M., M. S. Wolff, A. N. Rohl, and I. J. Selikoff, Variation of properties of chrysotile asbestos subjected to milling, **J. Toxicol. and Environ. Health**, 4:173-188, 1978.

21. Langer, A. M., A. D. Mackler, and F. D. Pooley, Electron microscopical investigation of asbestos fibers, **Environ. Health Perspect.**, 9:63-80, 1974.

22. Occella, E. and G. Maddalon, X-ray diffraction characteristics of some types of asbestos in relation to different techniques of comminution, **Med. Lavoro,** 54(10):628-636, 1963.

23. Spurny, K. R., W. Stober, H. Opiela, and G. Weiss, On the problem of milling and ultrasonic treatment of asbestos and glass fibers in biological and analytical applications, **Am. Ind. Hyg. Assoc. J.**, 41:198-203, 1980.

24. Berry, L. G. and B. Mason, **Mineralogy**, San Francisco: W. H. Greeman & Co., 1959.

25. Schelz, J. P., The detection of chrysotile asbestos at low levels in talc by differential thermal analysis, **Thermochimica Acta**, 8:197-204, 1974.

26. Reference 1, pp. 372-374.

27. Leroux, J., Staub-Reinhalt Luft, 29:26 (English), 1969.

28. Leroux, J. A., B. C. Davey, and A. Paillard, Proposed standard methodology for the evaluation of silicosis hazards, **Am. Ind. Hyg. Assoc. J.**, 34:409, 1973.

2.5 Analytical Electron Microscopy

2.5.1 Applicability

Analytical electron microscopy (AEM) can often be a reliable method for the detection and positive identification of asbestos in some bulk building materials, both friable and nonfriable. The method is particularly applicable to bulk materials that contain a large amount of interfering materials that can be removed by ashing and/or dissolution and contain asbestos fibers that are not resolved by PLM techniques. Many floor tiles and plasters would be included in this type of sample. In combination with suitable specimen preparation techniques, the AEM method can also be used to quantify asbestos concentrations.

2.5.2 Range

The range is dependent on the type of bulk material being analyzed. The upper detection limit is 100%, and the lower detection limit can be as low as 0.0001% depending on the extent to which interfering materials can be separated during the preparation of AEM

specimens, the sophistication of the AEM preparation, and the amount of labor expended on AEM examination.

2.5.3 Interferences

The presence of a large amount of binder/matrix materials associated with fibers can make it difficult to positively identify fibers as asbestos. The portion of the fiber examined by either electron diffraction or energy dispersive x-ray analysis (EDXA) must be free of binder/matrix materials.

2.5.4 Precision and Accuracy

The precision and accuracy of the method have not been determined.

2.5.5 Procedures

The procedures for AEM specimen preparation depend on the data required. In analysis of floor tiles, the weighed residue after removal of the matrix components (see Section 2.3, Gravimetry) is often mostly asbestos, and the task is primarily to identify the fibers. In this situation the proportion of asbestos in the residue can be estimated by AEM and this estimate can be used to refine the gravimetric result. For many floor tiles, the final result is not very sensitive to errors in this estimation because the proportion of asbestos in the residue is very high. For samples in which this is not the case, precise measurements can be made using a quantitative AEM preparation, in which each grid opening of the specimen grid corresponds to a known weight of the original sample or of a concentrate derived from the original sample. Asbestos fibers on these grids are then identified and measured, using a fiber counting protocol which is directed towards a precise determination of mass concentration. This latter procedure is suitable for samples of low asbestos concentration, or for those in which it is not possible to remove a large proportion of the matrix material.

2.5.5.1 AEM Specimen Preparation for Semi-Quantitative Evaluation

The residual material from any ashing or dissolution procedures (see Section 2.3) used (usually trapped on a membrane filter) should be placed in a small volume of ethanol or another solvent such as acetone or isopropyl alcohol, in a disposable beaker, and dispersed

by treatment in an ultrasonic bath. A small volume of this suspension (approximately 3μl) should be pipetted onto the top of a carbon-coated TEM grid. The suspension should be allowed to dry under a heat lamp. The grid is then ready for examination.

Samples that are not conducive to ashing or dissolution may also be prepared in this way for AEM analysis. A few milligrams of the sample may be ground in a mortar and pestle or milled, dispersed in ethanol or another solvent using an ultrasonic bath, and pipetted onto a grid as described previously.

2.5.5.2 AEM Specimen Preparation for Quantitative Evaluation

The objective of this preparation is to obtain a TEM grid on which a known weight of the bulk sample is represented by a known area of the TEM grid. A known weight of the bulk sample, or of the residue after extraction, should be dispersed in a known volume of distilled water. Aliquots of this dispersion should then be filtered through 0.22 μm pore-size MCE or 0.2 μm pore-size PC filters, using filtration techniques as described for analysis of water samples.[1] In order to obtain filters of appropriate particulate loading for AEM analysis, it may be necessary to perform serial dilutions of the initial dispersion. TEM grids should then be prepared from appropriately-loaded filters, using the standard methods.[2]

Determination of the mass concentration of asbestos on the TEM grids requires a different fiber counting protocol than that usually used for determination of numerical fiber concentrations. Initially, the grids should be scanned to determine the dimensions of the largest asbestos fiber or fiber bundle on the specimens. The volume of this fiber or bundle should be calculated. The magnification of the AEM should be set at a value for which the length of this fiber or bundle just fills the fluorescent screen. Asbestos fiber counting should then be continued at this magnification. The count should be terminated when the volume of the initial large fiber or bundle represents less than about 5% of the integrated volume of all asbestos fibers detected. This counting strategy ensures that the fiber counting effort is directed toward those fibers which contribute most to the mass, and permits a precise mass concentration value to be obtained.

2.5.5.2.1 Identification

To document the positive identification of asbestos in a sample, the analyst should record the following physical properties: morphology data, electron diffraction data, EDXA data, and any other distinguishing characteristics observed. For fibrous structures identified as nonasbestos, the unique physical property or properties that differentiate the material from asbestos should be recorded.

The purpose of the identification data collected is to prevent or limit false negatives and false positives. This can be accomplished by having a system for measuring and recording the d-spacings and symmetry of the diffraction patterns, determining the relative abundance of the elements detected by EDXA, and comparing these results to reference data. The laboratory should have a set of reference asbestos materials from which a set of reference diffraction patterns and x-ray spectra have been developed. Also, the laboratory should have available reference data on the crystallography and chemical composition of minerals that might analytically interfere with asbestos.

2.5.6 References

1. Chatfield, E.J., and M. J. Dillon, **Analytical Method for Determination of Asbestos Fibers in Water,** EPA-600/4-83-043. U.S. Environmental Protection Agency Environmental Research Laboratory, 1983.

2. Environmental Protection Agency's **Interim Transmission Electron Microscopy Analytical Methods--Mandatory and Nonmandatory--and Mandatory Section to Determine Completion of Response Actions**, Appendix A to subpart E, 40 CFR part 763.

2.6 Other Methodologies

Additional analytical methods (e.g. Scanning Electron Microscopy) may be applicable for some bulk materials. However, the analyst should take care to recognize the limitations of any analytical method chosen. Conventional SEM, for example, cannot detect small diameter fibers ($\sim < 0.2\mu m$), and cannot determine crystal structure. It is, however, very useful for observing surface features in complex particle matrices, and for determining elemental compositions.

3.0 QUALITY CONTROL/QUALITY ASSURANCE OPERATIONS- PLM

A program to routinely assess the quality of the results produced by the PLM laboratory must be developed and implemented. **Quality Control (QC)** is a system of activities whose purpose is to control the quality of the product or service so that it meets the need of the users. This also includes **Quality Assessment**, whose purpose is to provide assurance that the overall quality control is being done effectively. While the essential elements of a quality control system are described in detail elsewhere;[1,2,3,4,5,6] only several of the elements will be discussed here. **Quality Assurance (QA)** is comprised of Quality Control and Quality Assessment and is a system of activities designed to provide assurance that a product or service meets defined standards of quality.

The purpose of the Quality Assurance program is to minimize failures in the analysis of materials prior to submitting the results to the client. Failures in the analysis of asbestos materials include false positives, false negatives, and misidentification of asbestos types. False positives result from identification or quantitation errors. False negatives result from identification, detection, or quantitation errors.

For the stereomicroscopic and PLM techniques, the quality control procedures should characterize the accuracy and precision of both individual analysts and the techniques. Analysts should demonstrate their abilities on calibration materials, and also be checked routinely on the analysis of unknowns by comparison with results of a second analyst. The limitations of the stereomicroscopic and PLM techniques can be determined by using a second analytical technique, such as gravimetry, XRD, or AEM. For example, stereomicroscopic and PLM techniques can fail in the analysis of floor tiles because the asbestos fibers in the sample may be too small to be resolved by light microscopy. An XRD or AEM analysis is not subject to the same limitations, and may indicate the presence of asbestos in the sample.

The accuracy, precision, and detection limits of all analytical techniques described in this method are dependent on the type of sample (matrix components, texture, etc.), on the preparation of the sample (homogeneity, grain size, etc.), and the specifics of the method (number of point counts for PLM, mass of sample for gravimetry, counting time for XRD,

etc.). These should be kept in mind when designing quality control procedures and characterizing performance, and are variables that must be tracked in the quality assurance system.

3.1 General Considerations

3.1.1 Training

Of paramount importance in the successful use of this or any other analytical method is the well-trained analyst. It is highly recommended that the analyst have completed course work in optical mineralogy on the collegiate level. That is not to say that others cannot successfully use this method, but the classification error rate[7] may, in some cases, be directly attributable to level of training. In addition to completed course work in optical mineralogy, specialized course work in PLM and asbestos identification by PLM is desirable. Experience is as important as education. A good laboratory training program can be used in place of course work. Analysts that are in training and not yet fully qualified should have all analyses checked by a qualified analyst before results are released. A QC Plan for asbestos identification would be considered incomplete without a detailed description of the analyst training program, together with detailed records of training for each analyst.

3.1.2 Instrument Calibration and Maintenance

Microscope alignment checks (alignment of the polarizer at 90° with respect to the analyzer, and coincident with the cross-lines, proper orientation of the slow vibration direction of the Red I compensator plate, image of the field diaphragm focussed in the plane of the specimen, centering of the central dispersion staining stop, etc.) should be performed with sufficient frequency to ensure proper operations. Liquids used for refractive index determination and those optionally used for dispersion staining should have periodic refractive index checks using a refractometer or known refractive index solids. These calibrations must be documented.

Microscopes and ancillary equipment should be maintained daily. It is recommended that at least once per year each microscope be thoroughly cleaned and re-aligned by a professional microscope service technician. Adequate inventories of replaceable parts

(illumination lamps, etc.) should be established and maintained. All maintenance must be documented.

3.2 Quality Control of Asbestos Analysis

3.2.1 Qualitative Analysis

All analysts must be able to correctly identify the six regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite) using combined stereomicroscopic and PLM techniques. Standards for the six asbestos types listed are available from NIST, and should be used to train analysts in the measurement of optical properties and identification of asbestos. These materials can also be used as identification standards for XRD and AEM.

Identification errors between asbestos types (e.g. reporting amosite when tremolite is present) implies that the analyst cannot properly determine optical properties and is relying on morphology as the identification criteria. This is not acceptable. Each analyst in the lab should prove his or her proficiency in identifying the asbestos types; this can be checked through use of calibration materials (NVLAP proficiency testing materials, materials characterized by an independent technique, and synthesized materials) and by comparing results with another analyst. The identification of all parameters (e.g. refractive indices, birefringence, sign of elongation, etc.) leading to the identification should fall within control limits determined by the laboratory. In addition, a subset of materials should be analyzed using another technique to confirm the analysis.

As discussed earlier, the qualitative analysis is dependent upon matrix and asbestos type and texture. Therefore, the quality assurance system should monitor for samples that are difficult to analyze and develop additional or special steps to ensure accurate characterization of these materials. When an analyst is found to be out of the control limits defined by the laboratory, he or she should undergo additional training and have confirmatory analyses performed on all samples until the problem has been corrected.

3.2.2 Quantitative Analysis

The determination of the amount of asbestos in a sample can be accomplished using the various techniques outlined in this method. The mandatory stereomicroscopic and PLM examinations provide concentrations in terms of volume, area, or weight, depending upon the calibration procedure. Gravimetric and quantitative XRD techniques result in concentrations in units of weight percent. Specific guidelines for determining accuracy and precision using these techniques are provided in the appropriate sections of this method. In general, however, the accuracy of any technique is determined through analysis of calibration materials which are characterized by multiple independent techniques in order to provide an unbiased value for the analyte (asbestos) in question. The precision of any technique is determined by multiple analyses of the sample. The analyst is the detector for stereomicroscopic and PLM techniques, as opposed to gravimetric and XRD techniques, and therefore must be calibrated as an integral part of the procedure.

As in the qualitative analysis, the laboratory should determine its accuracy and precision for quantitative asbestos analysis according to the type of material analyzed and the technique used for analysis. For example, the laboratory may determine that its analysts have a problem with calibrated area estimates of samples containing cellulose and chrysotile and therefore needs to make or find special calibration materials for this class of sample.

Calibration materials for quantitative analysis of asbestos are available through the Bulk Asbestos NVLAP as proficiency testing materials for those laboratories enrolled in NVLAP. In a report provided following a test round, the concentration of asbestos in each sample is given in weight percent with 95%/95% tolerance limits, along with a description of the major matrix components. Materials from other round robin and quality assurance programs for asbestos analysis may not have been analyzed by independent techniques; the concentrations may represent consensus PLM results that could be significantly biased. Therefore, values from these programs should <u>not</u> be used as calibration materials for quantitative analysis.

Calibration materials for quantitative analysis can also be synthesized by mixing asbestos and appropriate matrix materials, as described in Appendix C of this method. These

materials are usually simplifications of "real world" samples; therefore the accuracy and precision determined from analysis of these materials are probably ideal.

Limits on permissible analytical variability must be established by the laboratory prior to QC implementation. It is recommended that a laboratory initially be at 100% quality control (all samples reanalyzed.) The proportion of quality control samples can later be lowered gradually, as control indicates, to a minimum of 10%. Quantitative results for standards including the mean and error estimate (typically 95% confidence or tolerance intervals) should be recorded. Over time these data can be used to help determine control limits for quality control charts.

The establishment and use of control charts is extensively discussed elsewhere in the literature. [1,2,3,4,5] Several cautions are in order:

- Control charts are based on the assumption that the data are distributed normally. Using rational subgrouping, the means of the subgroups are approximately normally distributed, irrespective of the distribution of the individual values in the subgroups. Control charts for asbestos analysis are probably going to be based on individual measurements, not rational subgroups. Check the data for normality before proceeding with the use of control charts. Ryan[8] suggests a minimum of 50 analyses before an attempt is made to establish control limits. However, for this analysis, consider setting "temporary" limits after accumulating 20-30 analyses of the sample.

- Include <u>both</u> prepared slides as well as bulk samples in your reference inventory.

- Make certain that sample quantities are sufficient to last, and that the act of sampling will not alter the composition of the reference sample.

Data on analytical variability can be obtained by having analysts repeat their analyses of samples and also by having different analysts analyze the same samples.

3.3 Interlaboratory Quality Control

The establishment and maintenance of an interlaboratory QC program is fundamental to continued assurance that the data produced within the laboratory are of consistent high quality. Intralaboratory programs may not be as sensitive to accuracy and precision error, especially if the control charts (see Section 3.2.2) for all analysts in the laboratory indicate small percent differences. A routine interlaboratory testing program will assist in the detection of internal bias and analyses may be performed more frequently than proficiency

testing. Arrangements should be made with at least two (preferably more) other laboratories that conduct asbestos identification by PLM. Samples (the number of which is left to the participating laboratories, but at least 4-10) representing the types of samples and matrices routinely submitted to the lab for analysis should be exchanged with sufficient frequency to determine intralaboratory bias. Both reference slides and bulk samples should be used. Results of the interlaboratory testing program should be evaluated by each of the participating laboratories and corrective actions, if needed, identified and implemented. Since quantitation problems are more pronounced at low concentrations ($\leq$ 5%), it would be prudent to include approximately 30-50% from this concentration range in the sample selection process.

3.4 Performance Audits

Performance audits are independent quantitative assessments of laboratory performance. These audits are similar to the interlaboratory QC programs established between several laboratories, but with a much larger cohort (the EPA Asbestos Bulk Sample Analysis Quality Assurance Program had as many as 1100 participating laboratories). Participation in this type of program permitted assessment of performance through the use of "consensus" test materials, and served to assist in assessing the bias relative to individual interlaboratory, as well as intralaboratory programs. Caution should be exercised in the use of "consensus" quantitation results, as they are likely to be significantly responsible for the propagation of high bias in visual estimates. The current NIST/NVLAP[9] for bulk asbestos laboratories (PLM) does not use concensus quantitation results. Results are reported in weight percent with a 95% tolerance interval. The American Industrial Hygiene Association (AIHA)[10] also conducts a proficiency testing program for bulk asbestos laboratories. Quantitation results for this program are derived from analyses by two reference laboratories and PLM, XRD and gravimetric analysis performed by Research Triangle Institute.

3.5 Systems Audits

Where performance audits are quantitative in nature, systems audits are qualitative. Systems audits are assessments of the laboratory quality system as specified in the Laboratory

Quality Assurance Manual. Such an audit might consist of an evaluation of some facet of the QA Manual, or the audit may be larger in scope. For example, the auditor might request specific laboratory data sheets which will be evaluated against written procedures for data recording in the laboratory. Or, the auditor might request air monitoring or contamination control data to review for frequency of sampling, analysis methodology, and/or corrective actions taken when problems were discovered. The audit report should reflect the nature of the audit as well as the audit results. Any recommendations for improvement should also be reflected in such a report.

3.6 References

1. **Quality Assurance for Air Pollution Measurement Systems. Volume I, Principles.** EPA-600/9-76-005, March, 1976.

2. Juran, J. and F. Gryna, **Quality Planning Analysis,** 2nd edition, McGraw-Hill, Inc., 1980.

3. Taylor, J.R., **Quality Control Systems,** McGraw Hill, Inc., 1989.

4. Ratliff, T.A., **The Laboratory Quality Assurance System,** Van Nostrand Reinhold, 1990.

5. Taylor, J.K., **Quality Assurance of Chemical Measurements,** Lewis Publishers, 1987.

6. Bulk Asbestos Handbook, National Institute of Standards and Technology, National Voluntary Laboratory Accreditation Program, NISTIR 88-3879, October 1988.

7. Harvey, B.W., "Classification and Identification Error Tendencies in Bulk Insulation Proficiency Testing Materials," **American Environmental Laboratory, 2**(2), 4/90, pp. 8-14.

8. Ryan, T.P., **Statistical Techniques for Quality Improvement,** John Wiley & Sons, Inc., New York, 1989.

9. National Institute of Standards & Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP), Building 411, Room A124, Gaithersburg, MD 20899, telephone (301) 975-4016.

10. American Industrial Hygiene Association (AIHA), 2700 Prosperity Avenue, Suite 250, Fairfax, VA 22031, (703) 849-8888.

APPENDIX A

Glossary Of Terms

APPENDIX A. GLOSSARY OF TERMS

Accuracy - The degree of agreement of a measured value with the true or expected value.

Anisotropic - Refers to substances that have more than one refractive index (e.g. are birefringent), such as nonisometric crystals, oriented polymers, or strained isotropic substances.

Asbestiform (morphology) - Said of a mineral that is like asbestos, i.e., crystallized with the habit of asbestos. Some asbestiform minerals may lack the properties which make asbestos commercially valuable, such as long fiber length and high tensile strength. With the light microscope, the asbestiform habit is generally recognized by the following characteristics:

- Mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5μm. Aspect ratios should be determined for fibers, not bundles.
- Very thin fibrils, usually less than 0.5 micrometers in width, and
- Two or more of the following:
  - Parallel fibers occurring in bundles,
  - Fiber bundles displaying splayed ends,
  - Matted masses of individual fibers, and/or
  - Fibers showing curvature

These characteristics refer to the population of fibers as observed in a bulk sample. It is not unusual to observe occasional particles having aspect ratios of 10:1 or less, but it is unlikely that the asbestos component(s) would be dominated by particles (individual fibers) having aspect ratios of $<$20:1 for fibers longer than 5μm. If a sample contains a fibrous component of which most of the fibers have aspect ratios of $<$20:1 and that do not display the additional asbestiform characteristics, by definition the component should not be considered asbestos.

Asbestos - A commercial term applied to the asbestiform varieties of six different minerals. The asbestos types are chrysotile (asbestiform serpentine), amosite (asbestiform grunerite), crocidolite (asbestiform riebeckite), and asbestiform anthophyllite, asbestiform tremolite, and asbestiform actinolite. The properties of asbestos that caused it to be widely used commercially are: 1) its ability to be separated into long, thin, flexible fibers; 2) high tensile strength; 3) low thermal and electrical conductivity; 4) high mechanical and chemical durability, and 5) high heat resistance.

Becke Line - A band of light seen at the periphery of a specimen when the refractive indices of the specimen and the mounting medium are different; it is used to determine refractive index.

Bias - A systematic error characterized by a consistent (non-random) measurement error.

Binder - With reference to a bulk sample, a component added for cohesiveness (e.g. plaster, cement, glue, etc.).

Birefringence - The numerical difference between the maximum and minimum refractive indices of an anisotropic substance. Birefringence may be estimated, using a Michel-Levy chart, from the interference colors observed under crossed polarizers. Interference colors are also dependent on the orientation and thickness of the grain, and therefore are used qualitatively to determine placement in one of the four categories listed below.

Qualitative	Quantitative(N-n)
none	0.00 or isotropic
low	$\leq$0.010
moderate	0.011-0.050
high	>0.050

Bulk Sample - A sample of building material taken for identification and quantitation of asbestos. Bulk building materials may include a wide variety of friable and nonfriable materials.

Bundle - Asbestos structure consisting of several fibers having a common axis of elongation.

Calibration Materials - Materials, such as known weight % standards, that assist in the calibration of microscopists in terms of ability to quantitate the asbestos content of bulk materials.

Color - The color of a particle or fiber when observed in plane polarized light.

Compensator - A device with known, fixed or variable retardation and vibration direction used for determining the degree of retardation (hence the thickness or value of birefringence) in an anisotropic specimen. It is also used to determine the sign of elongation of elongated materials. The most common compensator is the first-order red plate (530-550nm retardation).

Control Chart - A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.

Detection Limit - The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

Dispersion Staining (focal masking) - An optical means of imparting apparent or virtual color to transparent substances by the use of stops in the objective back focal plane; ir it is used to determine refractive indices.

Error - Difference between the true or expected value and the measured value of a quantity or parameter.

Extinction - The condition in which an anisotropic substance appears dark when observed between crossed polars. This occurs when the vibration directions in the specimen are parallel to the vibration directions in the polarizer and analyzer. Extinction may be complete or incomplete; common types include parallel, oblique, symmetrical and undulose.

Extinction Angle - For fibers, the angle between the extinction position and the position at which the fiber is parallel to the polarizer or analyzer privileged directions.

Fiber - With reference to asbestiform morphology, a structure consisting of one or more fibrils.

Fibril - The individual unit structure of fibers.

Friable - Refers to the cohesiveness of a bulk material, indicating that it may be crumbled or disaggregated by hand pressure.

Gravimetry - Any technique in which the concentration of a component is determined by weighing. As used in this document, it refers to measurement of asbestos-containing residues after sample treatment by ashing, dissolution, etc.

Homogeneous - Uniform in composition and distribution of all components of a material, such that multiple subsamples taken for analysis will contain the same components in approximately the same relative concentrations.

Heterogeneous - Lacking uniformity in composition and/or distribution of material; components not uniform. Does not satisfy the conditions stated for homogenous; e.g., layered or in clumps, very coarse grained, etc.

Isotropic - Refers to substances that have a single refractive index such as unstrained glass, un-oriented polymers and unstrained substances in the isometric crystal system.

Lamda Zero (λ_0) - The wavelength (λ_0) of the dispersion staining color shown by a specimen in a medium; both the specimen and medium have the same refractive index at that wavelength.

Matrix - Nonasbestos, nonbinder components of a bulk material. Includes such components as cellulose, fiberglass, mineral wool, mica, etc.

Michel-Levy Scale of Retardation colors - A chart plotting the relationship between birefringence, retardation and thickness of anisotropic substances. Any one of the three variables can be determined if the other two are known.

Morphology - The structure and shape of a particle. Characterization may be descriptive (platy, rod-like, acicular, etc) or in terms of dimensions such as length and diameter (see asbestiform).

Pleochroism - The change in color or hue of colored anisotropic substance when rotated relative to the vibration direction of plane polarized light.

Point Counting - A technique used to determine the relative projected areas occupied by separate components in a microscope slide preparation of a sample. For asbestos analysis, this technique is used to determine the relative concentrations of asbestos minerals to nonasbestos sample components.

Polarization Colors - Interference colors displayed by anisotropic substances between two polarizers. Birefringence, thickness and orientation of the material affect the colors and their intensity.

Precision - The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions. It is concerned with the variability of results.

Reference Materials - Bulk materials, both asbestos-containing and nonasbestos-containing, for which the components are well-documented as to identification and quantitation.

Refractive Index (index of refraction) - The ratio of the velocity of light in a vacuum relative to the velocity of light in a medium. It is expressed as n and varies with wavelength and temperature.

Sign of Elongation - Referring to the location of the high and low refractive indices in an elongated anisotropic substance, a specimen is described as positive when the higher refractive index is lengthwise (length slow), and as negative when the lower refractive index is lengthwise (length fast).

Standard Reference Material (SRM) - A reference material certified and distributed by the National Institute of Standards and Technology.

Visual Estimate - An estimation of concentration of asbestos in a sample as compared to the other sample components. This may be a volume estimate made during stereomicroscopic examination and/or a projected area estimation made during microscopic (PLM) examination.

APPENDIX B

Apparatus For Sample Preparation And Analysis

B1.0 INTRODUCTION

The following lists the apparatus and materials required and suggested for the methods of sample preparation and analysis described in the test method.[1,2,3]

B2.0 STEREOMICROSCOPIC EXAMINATION

The following are suggested for routine stereomicroscopic examination.

- HEPA-filtered hood or class 1 biohazard hood, negative pressure
- Microscope: binocular microscope, preferably stereoscopic, 5-60X magnification (approximate)
- Light source: incandescent or fluorescent
- Tweezers, dissecting needles, scalpels, probes, etc. (for sample manipulation)
- Glassine paper, glass plates, weigh boats, petri dishes, watchglasses, etc. (sample containers)

The following are suggested for sample preparation.

- Mortar and pestle, silica or porcelain-glazed
- Analytical balance (readability less than or equal to one milligram) (optional)
- Mill or blender (optional)

B3.0 POLARIZED LIGHT MICROSCOPY

The laboratory should be equipped with a polarized light microscope (preferably capable of Köhler or Köhler-type illumination if possible) and accessories as described below.

- Ocular(s) binocular or monocular with cross hair reticle, or functional equivalent, and a magnification of at least 8X
- 10X, 20X, and 40X objectives, (or similar magnification)

- Light source (with optional blue "day-light" filter)
- 360-degree rotatable stage
- Substage condenser with iris diaphragm
- Polarizer and analyzer which can be placed at 90 degrees to one another, and can be calibrated relative to the cross-line reticle in the ocular.
- Accessory slot for wave plates and compensators (or demonstrated equivalent).
- Wave retardation plate (Red I compensator) with approximately 550 nanometer retardation, and with known slow and fast vibration directions.
- Dispersion staining objective or a demonstrated equivalent. (optional)
- Monochromatic filter (n_D), or functional equivalent. (optional)

In addition, the following equipment, materials and reagents are required or recommended.[1]

- NIST traceable standards for the major asbestos types (NIST SRM 1866 and 1867)
- Class I biohazard hood or better (see "Note", Section 2.2.5)
- Sampling utensils (razor knives, forceps, probe needles, etc.)
- Microscope slides and cover slips
- Mechanical Stage
- Point Counting Stage (optional)
- Refractive index liquids: 1.490-1.570, 1.590-1.720 in increments of less than or equal to 0.005; high dispersion, (HD) liquids are optional; however, if using dispersion staining, HD liquids are recommended.
- Mortar and pestle
- Distilled water
- HCl, ACS reagent grade concentrated

- Muffle furnace (optional)
- Mill or blender (optional)
- Beakers and assorted glassware (optional)
- Other reagents (tetrahydrofuran, amyl acetate, acetone, sodium hexametaphosphate, etc.) (optional)

B4.0 GRAVIMETRY

The following equipment, materials, and reagents are suggested.

- Scalpels
- Crucibles, silica or porcelain-glazed, with lids
- Muffle furnace - temperature range at least to 500°C, temperature stable to $\pm$ 10°C, temperature at sample position calibrated to $\pm$ 10°C
- Filters, 0.4 μm pore size polycarbonate
- Petri dishes
- Glass filtration assembly, including vacuum flask, water aspirator, and/or air pump
- Analytical balance, readable to 0.001 gram
- Mortar and pestle, silica or porcelain-glazed
- Heat lamp or slide warmer
- Beakers and assorted glassware
- Centrifuge, bench-top
- Class I biohazard hood or better
- Bulb pipettes
- Distilled water
- HCl, reagent-grade concentrated

- Organic solvents (tetrahydrofuran, amyl acetate,etc)
- Ultrasonic bath

B5.0 X-RAY DIFFRACTION

Sample Preparation

Sample preparation apparatus requirements will depend upon the sample type under consideration and the kind of XRD analysis to be performed.

- Mortar and pestle: agate or porcelain
- Razor blades
- Sample mill: SPEX, Inc., freezer mill or equivalent
- Bulk sample holders
- Silver membrane filters: 25-mm diameter, 0.45-μm pore size. Selas Corp. of America, Flotronics Div., 1957 Pioneer Road, Huntington Valley, PA 19006
- Microscope slides
- Vacuum filtration apparatus: Gelman No. 1107 or equivalent, the side-arm vacuum flask
- Microbalance
- Ultrasonic bath or probe: Model W140, Ultrasonics, Inc., operated at a power density of approximately 0.1 W/mL, or equivalent
- Volumetric flasks: 1-L volume
- Assorted pipets
- Pipet bulb
- Nonserrated forceps
- Polyethylene wash bottle
- Pyrex beakers: 50-mL volume

- Desiccator
- Filter storage cassettes
- Magnetic stirring plate and bars
- Porcelain crucibles
- Muffle furnace or low temperature asher
- Class 1 biohazard hood or better

Sample Analysis

Sample analysis requirements include an x-ray diffraction unit, equipped with:

- Constant potential generator; voltage and mA stabilizers
- Automated diffractometer with step-scanning mode
- Copper target x-ray tube: high intensity; fine focus, preferably
- X-ray pulse height selector
- X-ray detector (with high voltage power supply): scintillation or proportional counter
- Focusing graphite crystal monochromator; or nickel filter (if copper source is used, and iron fluorescence is not a serious problem)
- Data output accessories:
  - Strip chart recorder
  - Decade scaler/timer
  - Digital printer

    or

  - PC, appropriate software and Laser Jet Printer
- Sample spinner (optional)
- Instrument calibration reference specimen: α-quartz reference crystal (Arkansas quartz standard, #180-147-00, Philips Electronics Instruments, Inc., 85 McKee Drive, Mahwah, NJ 07430) or equivalent.

Reagents, etc.

Reference Materials - The list of reference materials below is intended to serve as a guide. Every attempt should be made to acquire pure reference materials that are comparable to sample materials being analyzed.

- Chrysotile: UICC Canadian, NIST SRM 1866 (UICC reference material available from: UICC, MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan, CF61XW, UK); (NIST Standard Reference Materials available from the National Institute of Standards and Technology, Office of Reference Standards, Gaithersburg, MD 20899)
- Crocidolite: UICC, NIST SRM 1866.
- "Amosite": UICC, NIST SRM 1866.
- Anthophyllite-Asbestos: UICC, NIST SRM 1867
- Tremolite Asbestos: Wards Natural Science Establishment, Rochester, NY; Cyprus Research Standard, Cyprus Research, 2435 Military Ave., Los Angeles, CA 900064 (washed with dilute HCl to remove small amount of calcite impurity); Indian tremolite, Rajasthan State, India; NIST SRM 1867.
- Actinolite Asbestos: NIST SRM 1867

Adhesive - Tape, petroleum jelly, etc. (for attaching silver membrane filters to sample holders).

Surfactant - 1 Percent aerosol OT aqueous solution or equivalent.

Isopropanol - ACS Reagent Grade.

B6.0 ANALYTICAL ELECTRON MICROSCOPY

AEM equipment requirements will not be discussed in this document; it is suggested that equipment requirements stated in the AHERA regulations be followed. Additional information may be found in the NVLAP Program Handbook for Airborne Asbestos Analysis.[3]

The following additional materials and equipment are suggested:

- Analytical balance, readable to 0.001 gram
- Ultrasonic bath
- Glass filtration assembly (25mm), including vacuum flask and water aspirator
- Mixed cellulose ester (MCE) filters (0.22μm pore size) or 0.2μm pore size polycarbonate filters
- MCE backing filters (5μm pore size)
- Silica mortar and pestle
- Beakers - glass and disposable
- Pipettes, disposable, 1,5, and 10 ml

B7.0 REFERENCES

1. National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) Bulk Asbestos Handbook, NISTIR 88-3879, 1988.
2. **Interim Method for the Determination of Asbestos in Bulk Insulation Samples,** U.S. E.P.A. 600/M4-82-020, 1982.
3. National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) Program Handbook for Airborne Asbestos Analysis, NISTIR 89-4137, 1989.

APPENDIX C

Preparation and Use of Bulk Asbestos
Calibration Standards

C1.0 INTRODUCTION

Evaluation of the results from national proficiency testing programs for laboratories analyzing for asbestos in bulk materials indicates that laboratories have had, and continue to have, problems with quantitation of asbestos content, especially with samples having a low asbestos concentration.[1] For such samples, the mean value of asbestos content reported by laboratories may be four to ten times the true weight percent value. It is assumed that the majority of the laboratories quantify asbestos content by visual estimation, either stereomicroscopically or microscopically; therefore, the problem of quantitation must be attributed to lack of or inadequate calibration of microscopists.

As calibration standards for asbestos-containing bulk materials are not currently commercially available, laboratories should consider generating their own calibration materials. This may be done rather easily and inexpensively.

C2.0 MATERIALS AND APPARATUS

Relatively pure samples of asbestos minerals should be obtained. Chrysotile, amosite and crocidolite (SRM 1866) and anthophyllite, tremolite and actinolite (SRM 1867) are available from NIST. A variety of matrix materials are commercially available; included are calcium carbonate, perlite, vermiculite, mineral wool/fiberglass, and cellulose. Equipment, and materials needed to prepare calibration bulk materials are listed below.

- Analytical balance, readable to 0.001 gram
- Blender/mixer; multi-speed, ~ one quart capacity
- Filtration assembly, including vacuum flask, water aspirator and/or air pump (optional)
- HEPA-filtered hood with negative pressure
- Filters, 0.4μm pore size polycarbonate (optional)
- Beakers and assorted glassware, weigh boats, petri dishes, etc.
- Hot/warm plate

- Asbestos minerals
- Matrix materials
- Distilled water.

C3.0 MATERIAL FORMULATION PROCEDURES

The formulation procedure involves first weighing appropriate quantities of asbestos and matrix material to give the desired asbestos weight percent. The following formula may be used to determine the weights of asbestos and matrix materials needed to give a desired weight percent asbestos.

$$\frac{WTa}{Wa} = \frac{WTm}{Wm}$$

Where:

WTa = weight of asbestos in grams (to 0.001 gram)
WTm = weight of matrix materials in grams (to 0.001 gram)
Wa = weight percent asbestos
Wm = weight percent matrix

Example: The desired total weight for the calibration sample is ~ 10 grams containing 5% asbestos by weight. If 0.532 grams of asbestos are first weighed out, what corresponding weight of matrix material is required?

WTa = 0.532 grams
Wa = 5%
Wm = 95%

$$\frac{0.532}{5} = \frac{WTm}{95}$$

Then: WTm = 10.108 grams

The matrix is then placed into the pitcher of a standard over-the-counter blender, the pitcher being previously filled to approximately one-fourth capacity (8-10 ounces) with distilled water. Blending is performed at the lowest speed setting for approximately ten seconds which serves to disaggregate the matrix material. The asbestos is then added, with additional blending of approximately 30 seconds, again at the lowest speed setting. Caution should be taken not to overblend the asbestos-matrix mixture. This could result in a significant reduction in the size of the asbestos fibers causing a problem with detection at normal magnification during stereomicroscopic and microscopic analyses. Ingredients of the

pitcher are then poured into a filtering apparatus, with thorough rinsing of the pitcher to ensure complete material removal. After filtering, the material is transferred to a foil dish which is placed on a hot plate. The material is covered and allowed to sit over low heat until drying is complete; intermittent stirring will speed the drying process. For fine-grained matrix materials such as gypsum, calcium carbonate, clays, etc., the sample is not filtered after the blending process. Instead, the ingredients in the pitcher are transferred into a series of shallow, glass (petri) dishes. The ingredients should be stirred well between each pouring to minimize the possible settling (and over-representation) of some components. The dishes are covered and placed on a hot plate until the contents are thoroughly dried. For small quantities of any matrix materials (15 grams or less), air-drying without prior filtering is generally very suitable for removing water from the prepared sample. For each material, the final step involves placing all formulated, dried subsamples into a plastic bag (or into one petri dish, for small quantities), where brief hand-mixing will provide additional blending and help to break up any clumps produced during drying. **All operations should be performed in a safety-hood with negative pressure.**

C4.0 ANALYSIS OF MATERIALS

All formulations should be examined with the stereomicroscope to determine homogeneity. Gravimetric analysis (ashing and/or acid dissolution) should be performed on those materials containing organic and/or acid-soluble components. Matrix materials to which no asbestos has been added should be analyzed by gravimetric analysis to determine the amount of nonashable or insoluble materials that are present. Several subsamples of each material should be analyzed by the gravimetric technique to provide information concerning the uniformity of the prepared materials. Experience has shown that the previously described formulation procedure results in relatively homogeneous materials.[2]

C4.1 Stereomicroscopic Analysis

Visual estimation of sample components using the stereomicroscope is in reality a comparison of the relative volumes of the components.[3] Therefore, differences in specific gravity between asbestos and matrix material must be considered and the relationship

between weight percent and volume percent must be determined.[4] Materials such as expanded vermiculite, perlite, and cellulose have specific gravities significantly lower than asbestos minerals. Table C1 lists the specific gravities for the three most commonly encountered asbestos varieties and several common matrix materials.

TABLE C1. SPECIFIC GRAVITIES OF ASBESTOS VARIETIES AND MATRIX MATERIALS

Asbestos Type	Specific Gravity	Matrix Type	Specific Gravity
Chrysotile	2.6	Calcium Carbonate	2.7
		Gypsum	2.3
Amosite	3.2	Perlite	~0.4
		Vermiculite (expanded)	~0.3
		Mineral Wool	~2.5
Crocidolite	3.3	Fiberglass	~2.5
		Cellulose	~0.9

The conversion of weight percent asbestos to equivalent volume percent asbestos is given by the following formula:

$$\frac{\frac{Wa}{Ga}}{\frac{Wa}{Ga} + \frac{Wm}{Gm}} \times 100 = Va$$

where:

Wa = weight percent asbestos
Ga = specific gravity of asbestos
Wm = weight percent matrix
Gm = specific gravity of matrix
Va = volume percent asbestos

Example: Chrysotile and perlite have been combined to form a 5% asbestos calibration standard, by weight. What is the equivalent volume percent asbestos?

$$Wa = 5\% \quad Ga = 2.6 \quad Wm = 95\% \quad Gm = 0.4$$

$$Va = \frac{\frac{5}{2.6}}{\frac{5}{2.6} + \frac{95}{0.4}} \times 100 = 0.8\%$$

Conversely, to convert volume percent asbestos to equivalent weight percent, the following formula may be used.

$$\frac{(Va)(Ga)}{(Va)(Ga)+(Vm)(Gm)} \times 100 = Wa$$

Vm = volume percent matrix

Example: A calibration standard consisting of amosite and cellulose is estimated to contain 2% asbestos, by volume. What is the equivalent weight percent asbestos?

$$Va = 2\% \quad Ga = 3.2 \quad Vm = 98\% \quad Gm = 0.9$$

$$Wa = \frac{(2)(3.2)}{(2)(3.2)+(98)(0.9)} \times 100 = 6.77\%$$

Volume percentages should be calculated for all calibration materials prepared so that visual estimates determined by examination with the stereomicroscope may be compared to true volume concentrations.

Figure C1 illustrates the relationship between volume percent and weight percent of chrysotile mixed with vermiculite and cellulose respectively. It should be noted that when asbestos in a low weight percentage is mixed with matrix materials having low specific gravities (vermiculite, perlite), the resulting volume concentration of asbestos is very low. For example, a mixture containing three percent chrysotile by weight in a cellulose matrix would result in a volume percent asbestos of approximately 1.1%; in a vermiculite matrix, the resulting volume percent asbestos would be approximately 0.4%. In the latter case especially, an analyst might possibly fail to detect the asbestos or consider it to be present in only trace amounts.

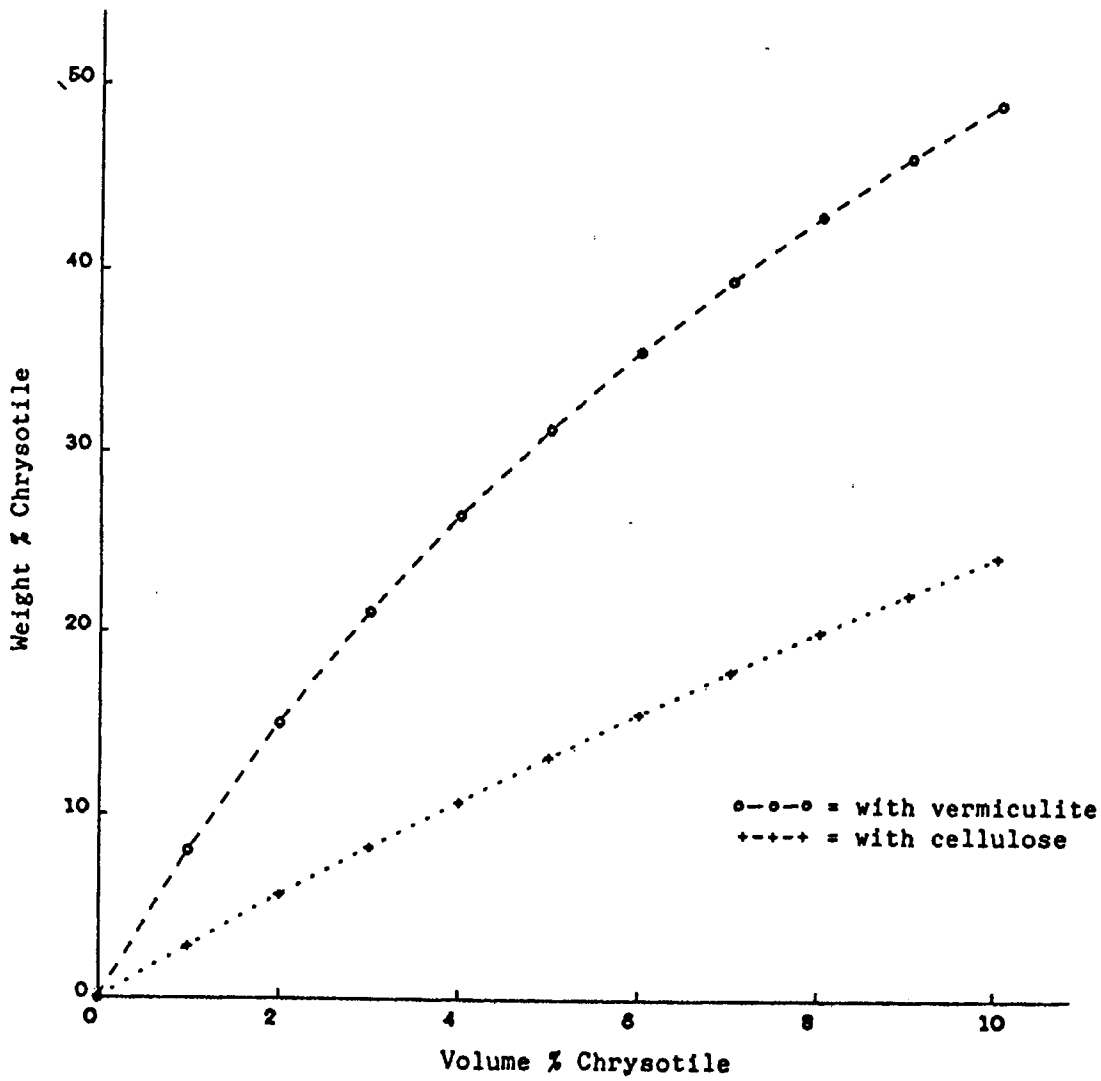


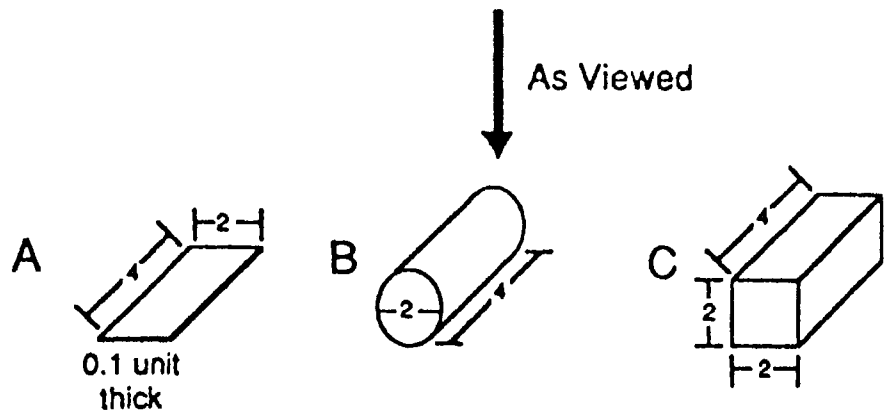
Figure C1. Relationship between volume % and weight % of chrysotile mixed with a)vermiculite and b) cellulose.

C4.2 Microscopical Analysis (PLM)

The polarized light microscope may be used to quantify asbestos and other components of a sample. Slide mounts are prepared from "pinch" samples of the calibration material and asbestos content is determined by visual area estimate and/or point counting. Both of these quantitation techniques are in fact estimates or measurements of the relative projected areas of particles as viewed in two dimensions on a microscope slide. For quantitation results to be meaningful, the following conditions should be met:

- The sample should be homogeneous for slide preparations, which are made from small pinches of the sample, to be representative of the total sample.
- Slide preparation should have an even distribution of particles and approach a one particle thickness (seldom achieved) to avoid particle overlap.
- All materials used should be identified and specific gravities determined in order to relate area percent to volume and/or weight percent.
- The size (thickness) relationship between matrix particles and asbestos fibers should be determined if the results based on projected area are to be related to volume and/or weight percent.

Particle characteristics can greatly affect the quantitation results obtained by visual area estimation or point counting. Figure C2 illustrates three hypothetical particle shapes of identical length and width (as viewed from above). Although the three-dimensional shape is different, the projected area is equal for all particles. The table accompanying Figure C2 presents data for each particle in terms of thickness, volume and projected area. It should be noted that although the projected areas may be equal, the volumes represented by the particles may vary by a factor of 20(0.8 vs 16 cubic units). It is obvious that quantitation of a sample consisting of a mixture of particles with widely ranging particle thicknesses could result in different results. For example, if a sample contained relatively thick bundles of asbestos and a fine-grained matrix such as clay or calcium carbonate, the true asbestos content (by volume) would likely be underestimated. Conversely, if a sample contained thick "books" of mica and thin bundles of asbestos, the asbestos content (by volume) would likely be overestimated.



Particle	Thickness	Volume	Projected Area
A	0.1 units	0.8 cubic units	8 sq. units
B	2 units	12.6 cubic units	8 sq. units
C	2 units	16 cubic units	8 sq. units

Note that although all particles have the same projected area, particle C volume is 20x that of particle A.

Figure C2. Relationship of projected area to volume and thickness for three different particles as viewed on a slide mount.

Table C2 illustrates several examples of expected results from area estimates or point counting of samples in which the asbestos fibers and matrix particles differ in thickness.

TABLE C2. RELATIONSHIP OF WEIGHT PERCENT, VOLUME PERCENT AND PARTICLE THICKNESS TO QUANTITATION RESULTS

Composition of Sample In Wt. %	Theoretical Vol. % Asbestos	Thickness Factor* (Matrix/Asbestos)	Expected Area %
1% Amosite 99% Calcium Carbonate	0.9	0.5	0.4
1% Amosite 99% Calcium Carbonate	0.9	1	0.9
1% Amosite 99% Calcium Carbonate	0.9	2	1.8
1% Amosite 99% Vermiculite	0.1	1	0.1
1% Amosite 99% Vermiculite	0.1	10	1.0
1% Amosite 99% Vermiculite	0.1	20	2.0
1% Amosite 99% Vermiculite	0.1	30	2.9

* Value represents the relationship between the mean thickness of the matrix particles compared to the mean thickness of the asbestos particles.

It should be noted that it is not uncommon for matrix particle thickness to differ greatly from asbestos fiber thickness, especially with matrix materials such as vermiculite and perlite; vermiculite and perlite particles may be 20 - 30 times as thick as the asbestos fibers.

The general size relationships between matrix particles and asbestos fibers may be determined by scanning slide mounts of a sample. A micrometer ocular enables the microscopist to actually measure particle sizes.

If a thickness factor can be determined for a calibration sample of known volume proportions of asbestos and matrix materials, an expected equivalent projected area asbestos can be calculated using the following formula:

$$\frac{Va}{\frac{Vm}{T} + Va} \times 100 = Aa$$

where:

Va	=	true volume percent asbestos
Vm	=	true volume percent matrix
T	=	thickness factor (mean size matrix particle/mean size asbestos fiber)
Aa	=	expected projected area percent asbestos

Example: A calibration standard of known weight percent asbestos is determined, by factoring in component specific gravities, to be 5.0% asbestos by volume. The matrix particles are estimated to be ten times thicker than the asbestos fibers. What would be the expected projected area percentage of asbestos?

Va = 5%
Vm = 95%
T = 10

$$Aa = \frac{5}{\frac{95}{10} + 5} \times 100 = 34.5\%$$

Conversely, to convert projected area percent asbestos to equivalent volume percent, the following formula may be used:

$$\frac{Aa}{T(Am) + Aa} \times 100 = Va$$

Where: Am = projected area matrix

Example: A slide containing a subsample of an amosite/mineral wool calibration standard is determined by point counting to have a projected area asbestos of 18.6%. If the mineral wool fibers are estimated to be six times the asbestos fibers, in diameter, what is the equivalent volume percent asbestos?

$$Am = 81.4\%$$
$$Aa = 18.6\%$$
$$T = 6$$

$$Va = \frac{(18.6)}{6(81.4) + 18.6} \times 100 = 3.67\%$$

Based on specific gravity values listed in Table 1C and on the above volume asbestos determination, what is the equivalent weight percent asbestos in the sample?

$$Va = 3.67\%$$
$$Ga = 3.2$$
$$Vm = 96.33\%$$
$$Gm = 2.5$$

$$Wa = \frac{(3.67)(3.2)}{(3.67)(3.2) + (96.33)(2.5)} \times 100 = 4.7\%$$

C5.0 USE OF CALIBRATION STANDARDS FOR QA/QC

Once the materials have been formulated and thoroughly characterized by all techniques to determine their suitability as calibration standards, a system for incorporating them into the QA/QC program should be established. Someone should be designated (QA officer, lab supervisor, etc.) to control the distribution of standards and to monitor the analysis results of the microscopists. Both precision and accuracy may be monitored with the use of suitable standard sets.

Records such as range charts, control charts, etc. may be maintained for volume (stereomicroscopic estimates), area (PLM) estimates and point counts. For point counts and area estimates, relatively permanent slides may be made using epoxy or Melt Mount *. Such slides may be very accurately quantified over time as to point count values, and due to their very long shelf life, may be used for QA/QC purposes almost indefinitely.

C6.0 REFERENCES

1. "Analysis Summaries for Samples used in NIST Proficiency Testing", National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) for Bulk Asbestos, January 1989 to present.

2. Harvey, B. W., R. L. Perkins, J. G. Nickerson, A. J. Newland and M. E. Beard, "Formulating Bulk Asbestos Standards", **Asbestos Issues**, April 1991.

3. Perkins, R. L. and M. E. Beard, "Estimating Asbestos Content of Bulk Materials", **National Asbestos Council Journal**, Vol. 9, No. 1, 1991, pp. 27-31.

4. **Asbestos Content in Bulk Insulation Samples: Visual Estimates and Weight Composition**, U.S. Environmental Protection Agency 560/5-88-011, 1988.

APPENDIX D

Special-Case Building Materials

Asbestos laboratories are now called upon to analyze many types of bulk building materials that are very difficult to characterize by routine PLM analysis. These materials are dominantly nonfriable and can be grouped into the following categories:

- Cementitious Products (pipe, sheeting, etc.)
- Viscous Matrix Products (adhesives, cements, coatings, etc.)
- Vinyl Materials (vinyl floor tile, sheeting)
- Asphaltic Roofing Materials (shingles, roll roofing)
- Miscellaneous Products (paints, coatings, friction plates, gaskets, etc.)

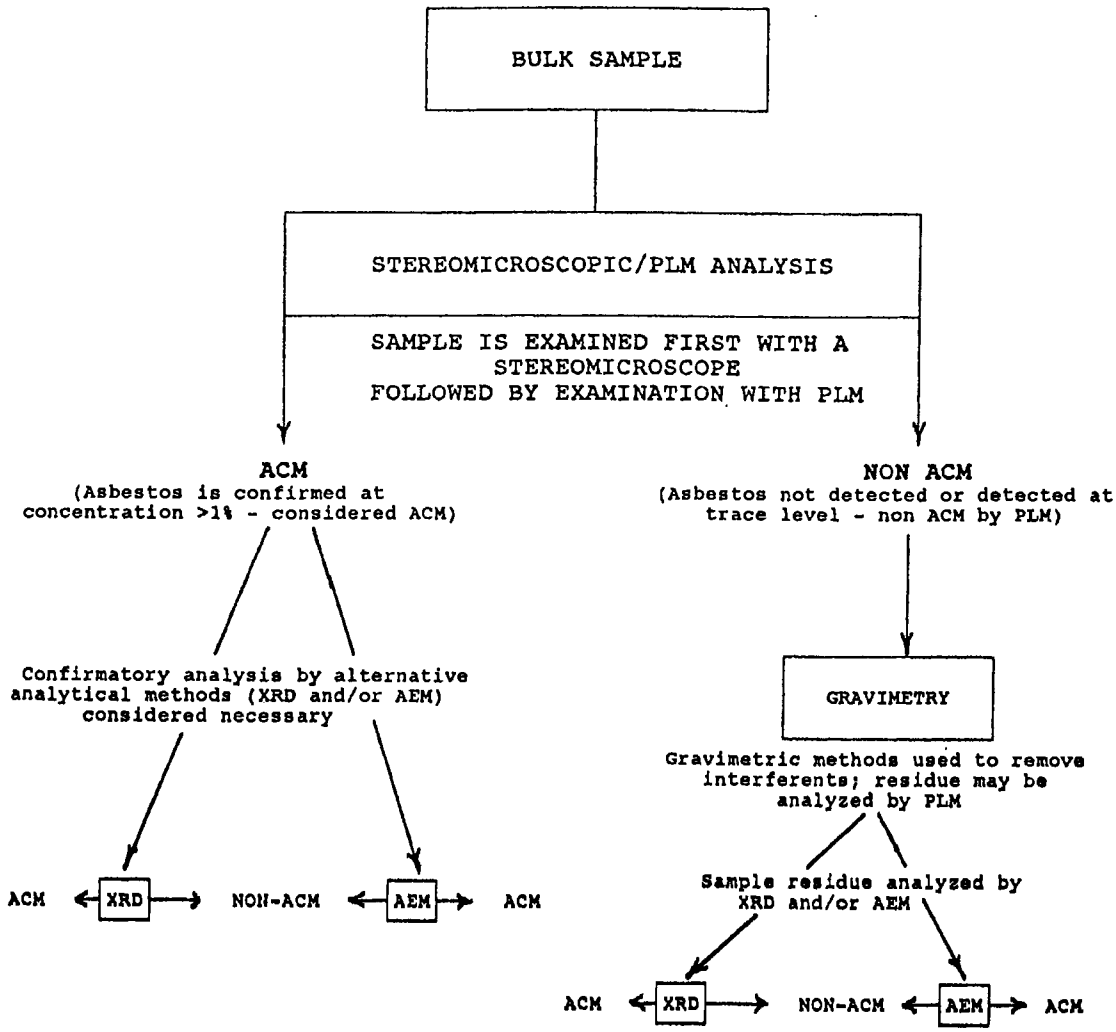
Materials characterized by interfering binder/matrix, low asbestos content, and/or small fiber size may require that additional sample treatment(s) and analysis be performed beyond routine PLM analysis. The sample treatment(s) required is(are) determined by the dominant nonasbestos sample components (see Section 2.3, Gravimetry). Materials containing an appreciable amount of calcareous material may be treated by dissolution with hydrochloric acid. Samples containing organic binders such as vinyl, plasticizers, esters, asphalts, etc. can be treated with organic solvents or ashed in a muffle furnace (preferred method) or low temperature plasma asher to remove unwanted components. Materials containing cellulose, synthetic organic fibers, textiles, etc. may also be ashed in a muffle furnace or low temperature plasma asher.

The method chosen for analysis of a sample after treatment is dependent on asbestos concentration and/or fiber size. An examination of the sample residue by PLM may disclose asbestos if the fibers are large enough to be resolved by the microscope, but additional analytical methods are required if the sample appears negative. Analysis by XRD is not fiber-size dependent, but may be limited by low concentration of asbestos and the presence of interfering mineral phases. In addition, the XRD method does not differentiate between fibrous and nonfibrous varieties of a mineral. Analysis by AEM is capable of providing positive identification of asbestos type(s) and semi-quantitation of asbestos content.

The following flowchart illustrates a possible scheme for the analysis of special-case building materials.

NOTE: Preliminary studies indicate that the XRD method is capable of detecting serpentine (chrysotile) in floor tile samples without extensive sample preparation prior to XRD analysis. XRD analysis of small, intact sections of floor tile yielded diffraction patterns that confirmed the presence of serpentine, even at concentrations of ~one percent by weight. TEM analysis of these same tiles confirmed the presence of chrysotile asbestos. With further investigation, this method may prove applicable to other types of nonfriable materials.

FLOWCHART FOR QUALITATIVE ANALYSIS OF SPECIAL CASE BUILDING MATERIALS SUCH AS FLOOR TILES, ASPHALTIC MATERIALS, VISCOUS MATRIX MATERIALS, ETC.*



*Although this flowchart is applicable to all bulk materials, it is primarily intended to be used with known problem materials that are difficult to analyze by PLM due to low asbestos concentration, and/or small fiber size, and/or interfering binder/matrix. In addition to being qualitative, the results may also be semi-quantitative. It should not be assumed that all samples need to be analyzed by AEM and XRD. The flowchart simply illustrates options for methods of analysis. Alternate methods such as SEM may be applicable to some bulk materials.

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ANALYSIS OF ASBESTOS FIBERS IN SOIL BY POLARIZED LIGHT MICROSCOPY

SYNOPSIS: A semi-quantitative method for identifying and quantifying asbestos fibers in soil using polarized light microscopy is provided. This method is based on NIOSH Method 9002, EPA Method 600/R-93/116, and CARB Method 435, with project specific modifications intended specifically for application at the Libby Superfund Site. Sampling and plan developers and data users are cautioned to understand how data are generated from this SOP.

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Revision	Date	Principal Changes
0	3/3/2003	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 sample preparation and analytical process. Authors: Douglas Kent and Nikki MacDonald, ESAT Region 8

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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 and has been refined to focus testing on Libby Amphibole 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 a preparation process described below. This method is appropriate for the analysis of all types of asbestos fibers (chrysotile and amphiboles), including those that are characteristic of the Libby Asbestos Superfund Site, Libby Amphibole asbestos (LA).

3.0 RESPONSIBILITIES

3.1 It is the responsibility of the laboratory supervisor to ensure that all analyses and quality assurance (QA) 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, QA/QC Coordinator (or equivalent), or Analytical Lead communicates with project managers at the United States Environmental Protection Agency ([EPA]; also referred to as the client), or their designate, any situations where a change from the SOP may be useful and/or required. The laboratory supervisor 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 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), Quality Management Plan (QMP), or an equivalent document(s) that meets all the requirements of the National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150. It is also the responsibility of the laboratory to ensure its testing activities stay in compliance with the requirements of NVLAP Handbook 150 and the regulatory and accrediting agencies that provide oversight of the laboratory's operations and all Libby Asbestos 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 Asbestos Superfund Site is based on the National Institute of Occupational Safety and Health (NIOSH) Method 9002, 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 (particles 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 only the analysis of the fine fractions of soil samples. Coarse fractions of 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 are then prepared of the sample 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 estimated in terms of mass fraction (percent asbestos by weight) based on the use of project-specific reference materials. Samples are re-analyzed or re-prepped and re-analyzed, and prepared standards are analyzed, as part of the quality control (QC) program.

5.0 ACRONYMS

ACM	Asbestos Containing Material
CARB	State of California Air Resources Board
EDD	Electronic Data Deliverable
EDS	Energy Dispersive Spectrometry
EDXA	Energy Dispersive X-ray Analysis
EPA	United States Environmental Protection Agency
HEPA	High Efficiency Particulate Air
LA	Libby Amphibole asbestos
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
PE	Performance Evaluation
PLM	Polarized Light Microscopy
PLM-VE	Visual Area Estimation technique employed by Polarized Light Microscopy
PPE	Personal Protective Equipment
QA	Quality Assurance
QAM	Quality Assurance Manual
QC	Quality Control
QMP	Quality Management Plan
RI	Refractive Index
SEM	Scanning Electron Microscopy

SOP	Standard Operating Procedure
SRM	Standard Reference Material
TEM	Transmission Electron Microscopy
µm	Microns (1,000 µm = 1mm)
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, Chemical Hygiene Plan, 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 each reagent (e.g., 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 distilled 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 QA Program

8.1.1 Each laboratory operates under a 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 Quality Management Plan, or equivalent, in which the laboratory's QA program is detailed. Additional QA/QC requirements specific to the PLM laboratory and the Libby project are described later 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 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 certainty of a measurement provided to the client, the client must be notified and informed of the nature of the departure from the SOP and 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 for the Libby project are expected to be familiar with routine chemical laboratory procedures, principles of optical mineralogy, and proficient in EPA Method 600/R-93/116, NIOSH Method 9002, and CARB Method 435.

9.1.2 Personnel at laboratories with less than one year of experience specific to the Libby Asbestos project 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 Asbestos Superfund Site.

9.2 Before performing any analyses, the 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 The laboratory has all items of equipment (including instrumentation, hardware, software, and reference materials) required for the correct performance of calibrations and tests.

10.2 All equipment is properly maintained and calibrated (as appropriate) prior to use. See Section 12 for further details regarding microscope calibration.

10.3 Following is a general list of the equipment available at the PLM laboratory to perform this SOP:

10.3.1 Polarized Light Microscope, with:

10.3.1.1 Light source and replacement bulbs
10.3.1.2 Binocular observation tube
10.3.1.3 Blue daylight filter
10.3.1.4 Oculars (10X)
10.3.1.5 Objectives: 10X, 20X, and 40X (or similar magnification)
10.3.1.6 10X Dispersion Staining Objective
10.3.1.7 360 degree rotatable and centerable stage
10.3.1.8 Polarizer and analyzer aligned at 90 degrees to one another
10.3.1.9 Bertrand lens (optional)
10.3.1.10 Substage condenser with iris diaphragm
10.3.1.11 Accessory slot for compensator plate
10.3.1.12 First order red (550 nanometer) compensator plate

10.3.1.13 Crosshair reticle
10.3.1.14 Adjustment tools
10.3.2 HEPA-filtered hood, class 1 biohazard hood, or glove box with continuous airflow (negative pressure)
10.3.3 Binocular stereomicroscope, 10-50X magnification (approximate)
10.3.4 Light source for stereomicroscope
10.3.5 Muffle furnace
10.3.6 Analytical balance
10.3.7 SOP-specific Electronic Data Deliverable (EDD), most recent version
10.3.8 Mortars (agate or porcelain)
10.3.9 Pestles (agate or porcelain)
10.3.10 Anemometer
10.3.11 Wet/dry vacuum with HEPA filtration
10.3.12 Decontamination equipment (e.g. baby wipes, wet mop with bucket, etc.)

11.0 STANDARDS, REAGENTS AND SUPPLIES

11.1 High Dispersion RI Liquid from 1.620 to 1.640 (1.625 is a common choice)

11.2 1.550 High Dispersion RI Liquid

11.3 1.680 to 1.700 RI Liquid

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, amosite, 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 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 Asbestos Superfund 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 Asbestos Superfund 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 Data recording sheet or bench sheet (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 Distilled 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 Kimwipes (or other appropriate wiping material)

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, and Controlled PE Reference Materials of LA in soils.

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 greater than 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 equal to or less than 1% are calibrated using the Controlled PE Reference Materials.

12.2 General Maintenance and Calibration of the Polarized Light Microscope

12.2.1 Chrysotile, amosite, crocidolite, and anthophyllite all have the optical property of parallel extinction. Because this is one of the optical properties used to identify these minerals, the polars of the PLM must be aligned north-south (N-S) and east-west (E-W), and the polars must be kept at 90 degrees to each other.

12.2.1.1 A mineral grain's extinction angle cannot be measured accurately if the polars are not correctly aligned.

12.2.2 LA and some non-asbestos minerals (wollastonite, hornblende, etc.) will often display an inclined (or oblique) extinction angle.

12.2.3 The lower polar must be properly aligned E-W so RI's in the parallel and perpendicular directions can be measured correctly.

12.2.4 The polars should be kept at 90 degrees to each other so the field of view in crossed polars is as dark as possible.

12.2.5 The microscope's optics must be kept clean and properly aligned so optimal image quality can be produced.

12.2.6 Check the microscope's alignment each working day prior to use.

12.2.6.1 The microscope must be re-aligned any time it is found to be out of alignment. Follow all the procedures outlined in Sections 12.3

through 12.8 for re-calibrating the microscope.

12.2.7 Each day the microscope is used, record an entry in the microscope's instrument maintenance logbook. Record the date and analyst's initials confirming that all microscope alignment checks were made prior to analysis.

12.2.7.1 An individual instrument maintenance logbook must be kept for each microscope in use at the laboratory.

12.2.7.2 All maintenance activities performed on the microscope must be recorded in the appropriate logbook.

12.2.7.3 Each day the microscope is used to analyze samples, a data entry must be made in the logbook indicating that the microscope was properly calibrated that day prior to use.

12.3 Checking Microscope Alignment

12.3.1 Place a permanently-mounted test 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 of anthophyllite 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 of anthophyllite become completely extinct in either the N-S or E-W direction, pull the analyzer out to make sure the fibers of anthophyllite 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 in view when the microscope stage is rotated.

12.3.4 The light path through the scope must be centered (specifically, the condenser and iris diaphragm must be centered on the optic axis).

12.3.5 The crosshairs should 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 strong cleavage traces between the sheets of mica.

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 anthophyllite or polypropylene 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 the analyzer out 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 shall 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 surfaces of the lenses.

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.

12.9.3 If dust gets inside the microscope, it is necessary to completely disassemble and clean the microscope.

12.9.3.1 The microscope must be re-calibrated after being re-assembled and this must be recorded in the microscope's maintenance logbook.

12.9.3.2 Disassembly of the microscope should only be performed by qualified personnel.

13.0 DETAILED METHOD FOR ASBESTOS TESTING OF SOIL AND SOIL-LIKE MATERIALS

13.1 Stereomicroscopic Examination

13.1.1 All sample preparation activities, including stereomicroscopic examination, slide mounts, etc., must be performed in a HEPA-filtered hood, class 1 biohazard hood, or glove box with continuous airflow (negative pressure).

13.1.2 Due to the sample preparation requirements described in the current revision of SOP ISSI-LIBBY-01, Soil Sample Preparation, samples should never be wet. If the sample is wet, contact EPA or designate.

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 container 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 larger.

13.1.5 With the stereomicroscope, visually examine the entire sample for homogeneity and the presence of any suspect fibers.

13.1.6 If individual fibers suspected of being asbestos are observed, 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 often called “fiber-picks” and are referred to as fiber-picks in this SOP.

13.1.6.1 Each microscope slide must be wiped with lint-free wipes prior to use to avoid contamination.

13.1.6.2 Mount individual fibers in 1.550 RI oil if chrysotile is suspected, 1.620 to 1.640 RI oil if LA or anthophyllite is suspected, or 1.680 to 1.700 RI oil if amosite or crocidolite is suspected.

13.1.6.3 Only one drop of RI liquid is necessary to prepare the 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 sample appearance, 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 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 Even if no fibers are visible, prepare the sample as described in Section 13.3.

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 data sheet in the comments field. Alternatively, attach a separate analytical data sheet (specific to ashing samples) with the necessary measurements, and indicate the attachment in the comments section.

13.3 Preparation of Samples for PLM Visual Area Estimation

13.3.1 Quantitative analysis preparation typically consists of preparing random mounts of a sample. The objective is to produce random mounts of a representative sub-sample from the original sample.

13.3.2 View the sample through the stereomicroscope to determine if it is sufficiently homogenized and all particles are reduced to a small enough size.

13.3.2.1 Soil samples processed according to the current revision of SOP ISSI-LIBBY-01, Soil Sample Preparation, should be ground to a maximum particle size of approximately 250 μm.

13.3.2.2 Additional homogenization of the sample at the laboratory using a mortar and pestle may be required if any remaining inhomogeneities or coarse particles are observed in the sample. When further grinding the sample, care should be taken to not pulverize the LA to a fiber size unidentifiable by PLM techniques. The material in the slide mounts must be coarse enough that fibers of LA can still be identified by PLM and still be as representative as possible of the sample as a whole.

13.3.3 Oil immersion mounts of randomly selected sub-samples of the homogenized material are prepared in RI liquids for PLM analysis.

13.3.3.1 Prepare a minimum of five random mount slides for each sample.

13.3.3.2 Each microscope slide must be wiped clean with an appropriate wipe prior to use in order to avoid contamination.

13.3.3.3 Place one to two drops of the appropriate RI liquid onto each slide.

13.3.3.3.1 Prepare at least two slides with a RI liquid in the range of 1.620 to 1.640 for easier measurement of the optical properties of LA. Generally, 1.625 RI liquid is used for LA.

13.3.3.3.2 The refractive indices of the oils used for the remaining slides is left to the analyst's discretion based upon the suspected mineralogy present in the sample material.

13.3.3.4 Use a spatula, the curved edge of a scalpel blade, or other similar utensil to collect randomly selected sub-samples of the homogenized

sample material, and place this into the RI liquid on the slides.

13.3.3.5 With the utensil, gently stir the sample material in the RI liquid to produce a homogeneous mixture.

13.3.3.6 Cover each mixture of RI liquid and sample material with a glass cover slip.

13.3.3.7 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.

13.3.3.7.1 Use this action to spread the mixture of RI liquid and sample material over the approximate area of the cover slip.

13.3.3.7.2 The material under the cover slip should be spread out evenly with no or very few overlapping particles.

13.3.3.8 Wipe any loose sample material or excess RI liquid from the slide with lint-free wipes.

13.3.3.9 The prepared slide can now be safely removed from the hood for analysis by PLM.

13.4 Supplemental Stereomicroscopic Evaluation

13.4.1 Following random slide mount preparation, it may be useful agitate or tap the sample container to cause the particulate to settle and the amphibole fibers to sort to the surface.

13.4.1.1 Re-examine the sample using the stereomicroscope, and repeat procedures 13.1.6, above.

13.4.1.2 This “tapping” method 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.

13.4.1.3 The representative sub-sample material used for preparing random slide mounts must remain homogeneous.

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 come into contact with the sample, with distilled water and paper towels.

13.4.2.2 Dispose of gloves after 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 oil or to oil 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 Libby soil samples 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 Morphology
- 13.5.4.2 Color and pleochroism (if pleochroism is present)
- 13.5.4.3 Refractive indices, both alpha and gamma
- 13.5.4.4 Birefringence
- 13.5.4.5 Extinction characteristics
- 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 have been 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 Superfund Site. Also included are the minerals magnesio-arfvedsonite and magnesio-riebeckite, which are known to occur at the Libby Asbestos Superfund Site in smaller quantities.
OA	Other amphibole asbestos	Regulated amphibole asbestos (amosite, crocidolite, and anthophyllite) that are not thought to occur in significant amounts at the mine in Libby.
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 serpentine occurs in an asbestiform morhology, 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 morphologies.
- 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) morphology, it is classified as non-asbestiform serpentine (antigorite if it is platy or lizardite if it occurs as a massive, fine-grained matrix) and not as asbestos (chrysotile).

13.5.7.5 If serpentine is observed and has a fibrous morphology, it is classified as chrysotile asbestos.

13.5.7.6 The morphology of chrysotile is fibrous and sometimes silky.

13.5.7.7 The fibers are flexible. Chrysotile sometimes occurs as tangled mats of many fibers.

13.5.7.8 Chrysotile can only be seen in PLM as chrysotile bundles; the individual fibrils that make up a chrysotile bundle are beyond the resolution of all light microscopy.

13.5.7.9 Bundles of chrysotile are often splayed.

13.5.7.10 Kinked chevron-style folds are sometimes seen in chrysotile.

13.5.7.11 Chrysotile is usually colorless in PLM, although it sometimes shows a slight golden, yellow, or pale golden-green color in PLM.

13.5.7.12 Chrysotile that has been exposed to very high temperatures is distinctly brown under plain light.

13.5.7.13 Chrysotile is never pleochroic.

13.5.7.14 Small particles of opaque magnetite can sometimes be seen in large, intact bundles of chrysotile.

13.5.7.15 The range for the lower RI (alpha, or α) for chrysotile is 1.545 to 1.553 as reported in the certificate for NIST SRM 1866b, although the range for chrysotile encountered in field samples may be somewhat wider.

13.5.7.16 The range for the higher RI (gamma, or γ) for chrysotile is 1.552 to 1.560 as reported in the certificate for NIST SRM 1866b, although the range for chrysotile encountered in field samples may be somewhat wider.

13.5.7.17 Exposure to high heat and dehydration of the crystal lattice will increase the refractive indices of chrysotile.

13.5.7.18 The birefringence (expressed numerically as δ, the difference between α and γ) of chrysotile is low, usually around 0.008. In practice, this means that most chrysotile bundles of fine to medium size observed in samples will have low first order gray to medium gray interference colors under crossed polars. Larger, thicker fibers can show first order white to yellow interference colors; higher colors may be seen in the thickest bundles.

13.5.7.19 Chrysotile is most easily visible in plane light in the higher RI liquids, such as 1.62 or 1.68. However, measurement of the refractive indices of chrysotile should be done with the fibers mounted in the 1.550 oil.

13.5.7.20 Chrysotile is almost always length slow (positive sign of elongation), although length fast chrysotile has been observed on very rare occasions.

13.5.7.21 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) morphology. Bundles of amosite are composed of many lesser needles of amosite. Needles of amosite are often straight and only somewhat flexible.

13.5.8.3 Amosite is usually colorless, green, brown, or greenish-brown in plane light. Heated amosite is brown to dark brown and can be nearly opaque. Amosite is sometimes weakly pleochroic.

13.5.8.4 The range for the lower RI (α) for amosite is 1.675 to 1.681 as reported in the certificate for NIST SRM 1866b, although the range for amosite encountered in field samples may be somewhat wider.

13.5.8.5 The range for the higher RI (γ) for amosite is 1.697 to 1.704 as reported in the certificate for NIST SRM 1866b, although the range for amosite encountered in field samples may be somewhat wider.

13.5.8.6 Exposure to high heat and dehydration of the crystal lattice will increase the RI's of amosite.

13.5.8.7 The birefringence of amosite is moderate, usually about 0.020. Most fibers observed will have first order white to yellow interference colors under crossed polars; although, higher colors (first order magenta to second order or sometimes even higher) can be seen in the thicker bundles.

13.5.8.8 RI measurements should be done with the fibers mounted in 1.680 to 1.700 RI oil.

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.

13.5.9.2 Crocidolite has an acicular morphology very similar to that of amosite. The fibers are only somewhat flexible.

13.5.9.3 Crocidolite is distinctly blue or blue-green in plane light and is pleochroic.

13.5.9.4 Normally, the range for the lower RI (α) for crocidolite is 1.680 to 1.698 (EPA, 1993).

13.5.9.5 Normally, the range for the higher RI (γ) for crocidolite is 1.685 to 1.706 (EPA, 1993).

13.5.9.6 The strong color of crocidolite makes measurement of the refractive indices very difficult. For this reason, select finer fibers of crocidolite, which have less color, when measuring refractive indices.

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 oil.

13.5.9.9 Because crocidolite is length fast, the lower RI (α) should be measured with the fiber oriented in the E-W direction (parallel to the lower polar), and the higher RI (γ) should be measured with the fiber oriented in the perpendicular (N-S) direction.

13.5.9.10 Even though riebeckite is a monoclinic mineral, the extremely narrow fibers that form bundles of crocidolite cause crocidolite to have parallel extinction.

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

13.5.10.1 Anthophyllite occurs as straight to slightly curved fibers or fiber bundles. The morphology of anthophyllite is lamellar to acicular.

13.5.10.2 Anthophyllite is a rare type of asbestos used in construction materials.

13.5.10.3 Anthophyllite is colorless to pale brown in plane light. It is sometimes weakly pleochroic.

13.5.10.4 The range for the lower RI (α) for anthophyllite is 1.593 to 1.694 (Deer et al., 1997). The commercial-grade anthophyllite in SRM 1867a has an α of 1.615.

13.5.10.5 The range for the higher RI (γ) for anthophyllite is 1.613 to 1.722 (Deer et al., 1997). The commercial-grade anthophyllite in SRM 1867a has a γ of 1.636.

13.5.10.6 The birefringence of anthophyllite is moderate, usually about 0.020.

13.5.10.7 Generally, RI measurements on anthophyllite should be done with the fibers mounted in 1.620 to 1.640 oil.

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, $Na_2Mg_3Fe^{3+}{}_2Si_8O_{22}(OH)_2$.

13.5.11.2 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 a group of minerals, such as tremolite, actinolite, winchite, richterite, magnesio-riebeckite, and magnesio-arfvedsonite. The optical properties for each individual mineral are provided below and in Attachment 4. As seen, 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. Rather, if the sample exhibits the optical properties of a mineral listed in this section, the specific optical properties (such as refractive indices, birefringence, extinction angle, and sign of elongation) shall be noted on the analytical data sheet and EDD, and the mineral identified as LA.

13.5.11.3 The morphology 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 morphologies that may be encountered during PLM analysis.

13.5.11.6 Laboratories should use the Controlled Libby Amphibole Asbestos 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 designate if they do not have these reference materials.

13.5.11.7 Color of LA in plane light is highly varied. Tremolite is usually colorless in plane light. 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 in plane light is yellowish green, brownish green, or grey-blue (Deer et al, 1997). Magnesio-riebeckite in plane light is blue, grey-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 0.015 to 0.02.

13.5.11.9 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 approximately 30 degrees or even higher for richterite.

13.5.11.10 RI measurements on LA should be done with the fibers mounted in 1.620 to 1.640 RI oil (1.625 is a commonly-used choice).

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, Optical Properties of Fibrous Amphiboles, for additional information on the optical properties of LA used in LA identification.

13.6 Refractometry

13.6.1 Calibration of Refractive Index Liquids

13.6.1.1 Accurate measurement of a mineral's refractive indices 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 an oil immersion mount of the appropriate certified precision optical glass in the oil 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 (see page 7 of Attachment 3).

13.6.1.4 Next determine λ_0. This is the wavelength at which the RI of the oil is equal to the RI of the certified precision optical glass. Observe the

central stop dispersion staining color shown by the glass, and consult the dispersion staining color chart (McCrone, 1987). If the glass particles show a range of dispersion staining colors, use the most predominant color when determining λ_0. Record the predominant dispersion staining color and corresponding λ_0 on the worksheet.

13.6.1.5 Consult the Excel spreadsheet developed by Shu-Chun Su, Ph.D., "Create_RI_Liquid_Calibration_Conversion_Tables.xls", for the appropriate conversion table (see Attachment 2). These tables are used to convert λ_0 and t into n_d^{25}, which is the calibrated RI of the oil at a wavelength of 589 nm and a temperature of 25°C. Determine the value of n_d^{25} from the appropriate table for the known values λ_0 and t.

13.6.1.6 Additional conversion tables for oils not included in the spreadsheet can be generated by entering the dispersion coefficients and values of n_d of the oil and the glass, and the value of dn/dt (change of RI with temperature) of the oil into the first sheet of the workbook. All of these values are clearly provided by the manufacturer of the glass and oil.

13.6.1.7 Record the value of n_d^{25} on the worksheet. This is the calibrated RI of the oil 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 actual calibrated RI of the oil and the original RI of the oil is greater than 0.004, then the oil may not be used for analysis of samples.

13.6.1.10 Repeat the above steps for each oil in routine use.

13.6.2 Measurement of refractive indices (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 oil 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 oil. However, this method does not require a specific mineral-oil chart to be developed before it is used. For this reason the Becke Line method can be used to measure the RI's of other materials besides LA and regulated asbestos minerals.

13.6.3 Measurement of Refractive Indices by the Dispersion Staining Method

13.6.3.1 Mount the fibers in the appropriate oil (1.550 for fibers suspected of being chrysotile, 1.620 to 1.640 oil for fibers suspected of being LA or anthophyllite, or 1.680 to 1.700 oil for fibers suspected of being amosite or crocidolite).

13.6.3.2 In order for the correct dispersion staining colors to be displayed, a clean surface of the mineral must be in direct contact with the RI liquid.

13.6.3.3 If 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.

13.6.3.4 Examine the slide in plane light using the 10X dispersion staining objective. The dispersion staining objective and its central stop should be centered.

13.6.3.5 Stop down the condenser iris diaphragm until dispersion colors are observed.

13.6.3.6 Read the thermometer to find ambient temperature of the laboratory's air to the nearest 2°C.

13.6.3.7 To measure α, orient the fiber E-W (parallel to the lower polar) if the fiber is suspected of being crocidolite, or N-S if the fiber is suspected of being chrysotile, amosite, or anthophyllite. LA shows biaxial optics and requires a more detailed treatment, described below in Section 13.6.5.

13.6.3.8 Next, observe the dispersion staining color that is displayed.

13.6.3.9 Light of a wavelength higher or lower than the matching wavelength (given the symbol λ_0, where the RI of the oil matches the RI of the mineral) is refracted around the central stop and passes through.

13.6.3.10 Light of a wavelength equal or approximately equal to the matching wavelength is blocked.

13.6.3.11 The observed color is the summation of the remaining light.

13.6.3.12 Consult the dispersion staining color chart (McCrone, 1987) and find the matching wavelength (λ_0) that corresponds to the observed color.

13.6.3.13 When measuring α and a range of dispersion staining colors is displayed, choose the color that produces the lowest RI, i.e., the color that corresponds to the longest λ_0.

13.6.3.14 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).

13.6.3.15 For the appropriate RI oil and mineral combination, find the column for the laboratory's temperature and row for λ_0; record the corresponding value of RI.

13.6.3.16 To measure γ, rotate the stage 90 degrees.

13.6.3.17 The fiber should now be perpendicular to the lower polar (N-S) if the fiber is suspected of being crocidolite, or parallel to the lower polar (E-W) if the fiber is suspected of being chrysotile, amosite, or anthophyllite. Refer to Section 13.6.5 for orienting fibers of LA when measuring γ.

13.6.3.18 Observe the dispersion staining colors and find the corresponding λ_0. When measuring γ, choose the color that produces the highest RI, i.e., the color that corresponds to the shortest λ_0.

13.6.3.19 Consult the appropriate chart for the asbestos type and oil being used; record the value of RI for the temperature and λ_0.

Note: There are two charts for each mineral and oil combination - one for α and one for γ. Be sure to use the appropriate chart when measuring α or γ.

13.6.4 Measurement of Refractive Indices 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 to or the same as the RI of the oil. Becke line colors are usually best observed using high magnification (200X to 500X).

13.6.4.2 To measure refractive indices using the Becke line method, mount the fibers in an oil whose RI is close to that of the mineral.

13.6.4.3 Observe the Becke line colors with the fiber oriented in the parallel and perpendicular directions.

13.6.4.4 As a rule, the Becke line moves into whichever medium (the grain or the oil) that 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 oil for some wavelengths of light in the visible spectrum and when the RI of the grain is less than the oil 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 oil, the RI of the grain is less than that of the oil.

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 oil, the RI of the grain is higher than that of the oil.

13.6.4.8 A perfect match occurs when n_d (the RI for the wavelength of sodium light, 589 nm) is the same for both the grain and the oil. When the n_d of mineral matches the n_d of the oil, an orange Becke line with just a touch of red moves into the grain and a bluish line moves into the oil when the stage is lowered.

13.6.4.9 If a perfect match cannot be obtained, mount the mineral in two oils 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 (1999) may be used to approximate the size of the difference between the RI of the oil 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.

13.6.5.2 Even though chrysotile is a monoclinic mineral, it does not show biaxial optics because of the scrolled nature of the fibers.

13.6.5.3 When an asbestos fiber shows biaxial optics, it is easy to measure a RI called α' that is between true α and beta (β) when attempting to measure α.

13.6.5.4 True α can only be observed when a crystal is oriented in exactly the correct position.

13.6.5.5 For the monoclinic minerals that display biaxial optics (LA), the crystals need to be oriented so the X and Z axes of the biaxial indicratix corresponding to the directions of α and γ are parallel to the lower polar when measuring these indices, and they are not necessarily oriented with the crystallographic axes. As a general rule, when these fibers show inclined extinction, select the fibers that show the highest extinction angle when measuring α and γ. RI measurements should be made on a fiber where the plane of X and Z in the biaxial indicatrix lies as close to parallel to the plane of the microscope stage as possible, such that the microscopist is looking directly down Y, which corresponds to the β RI (and also the b crystallographic axis for tremolite, actinolite, winchite, richterite, and magnesio-arfvedsonite). Fibers at or close to this orientation will tend

to show the highest extinction angle.

13.6.5.6 Next, when measuring α for LA, orient the fiber approximately N-S, at the 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.

13.6.5.7 Repeat this for a number of crystals. If the crystals show different Becke line colors or dispersion staining colors, measure α for the crystals that display the lowest RI.

13.6.5.8 Similarly, it is easy to measure a RI called γ' that is between β and true γ when attempting to measure γ. True γ can only be observed when a crystal is oriented in exactly the correct position.

13.6.5.9 Orient a fiber of LA approximately E-W, so that the fiber is extinct under crossed polars, when measuring γ. 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 crystals. If the crystals show different Becke line colors or dispersion staining colors, measure γ for those that display the highest RI.

13.6.5.10 Biaxial Optics of Anthophyllite

13.6.5.10.1 When measuring α (the lower RI) for anthophyllite, the fiber should be oriented in the perpendicular (N-S) direction. When fibers of anthophyllite are oriented in the N-S position, they can show either α or β, or anywhere in between, depending on their orientation. It is therefore necessary to examine a number of fibers oriented in the N-S position to find true α (α will be shown for the fibers that show the lowest RI).

13.6.5.10.2 When measuring γ (the higher RI) for anthophyllite, the fiber should be oriented in the parallel (E-W) position. Fibers of anthophyllite lying flat on the slide will always show γ and not γ', because the c-axis of the fiber will lie within the plane of the slide.

13.7 Quantification of Asbestos Content

13.7.1 General

13.7.1.1 Asbestos is reported as mass fraction percent for LA and is reported as area fraction percent for chrysotile, amosite, crocidolite, and anthophyllite.

13.7.1.2 Asbestos must be positively identified, and its optical properties measured and recorded, before asbestos 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 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 phases 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 calibrated visual estimate. Making accurate calibrated visual estimates is a skill that must be learned and analysts generally improve over time.

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 fraction of asbestos as a percentage of the total material present over many fields of view. Area fraction estimation may be difficult, especially at low concentration values and because the desired output for LA is an estimate of mass fraction (percent asbestos by weight). As a result, all visual estimates of LA content will be performed using a set of site-specific reference materials (calibration standards) 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 analysis using the same approach as for field samples.

13.7.3.2 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 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.

13.7.3.3 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.

13.7.3.4 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.

[1] The nominal mass fraction of the reference materials (calibration standards) is based on the gravimetric fraction of the material that is soil and the amount that is spiking material, adjusted for the fraction of the spiking material that is LA. For example, if the spiking material were estimated to contain 85% LA by mass, then the 1.0% calibration standard would contain 1.18 grams of spiking material (1.00 grams of LA) per 100 grams of calibration standard. Because the estimate of LA content of the spiking material is approximate, the true concentration of a calibration material may not be precisely equal to the nominal value.

13.7.3.5 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.

13.7.3.6 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).

13.7.3.7 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 cut-offs" that place LA concentrations of each sample into one of four categories (see Section 13.8.5, below).

13.7.3.8 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 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 13.2 of this SOP, 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. Stereomicroscopic examination is often an iterative process used in conjunction with analysis by PLM. Refer to Attachment 8 for a flow diagram 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 counted as LA and contributes to the bin category (described in Table 13.2), regardless of its morphology type or aspect ratio. This includes prismatic LA, as well as more fibrous varieties, such as bundles with fibers crossing at various angles giving the bundle a “matted” appearance. Refer to Attachment 5 for examples of a wide range of LA morphologies. Also refer to Attachment 6 for photomicrographs of representative examples of LA morphologies as imaged by the United States Geological Survey (USGS) by Scanning Electron Microscopy (SEM).

13.7.5.2 Using the two 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 (wt.%)	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 was 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 mass percent is estimated quantitatively.

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 detected in each 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 slides 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 slides 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 without an entry in the Qualifier column** is used for all samples in which LA is observed either by stereomicroscopy or by PLM in slides 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 fraction as ND if these analytes are not detected, "<1" if these analytes were detected but at a concentration of less than 1% by area, or to the nearest whole percentage (1%, 2%, 3%, etc.) if these analytes were 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 morphologies. Commonly encountered non-asbestos fibers are listed below.

13.8.3 Talc $Mg_3Si_4O_{10}(OH)_2$

13.8.3.1 Talc usually occurs in a platy or fibrous morphology that looks similar to that of chrysotile.

13.8.3.2 Talc has a higher birefringence than chrysotile.

13.8.3.3 The birefringence of talc is 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.

13.8.3.4 Talc has higher refractive indices (α = 1.54 to 1.56, γ = 1.57 to 1.60) than chrysotile.

13.8.3.5 Talc's refractive indices are less than those of tremolite, actinolite, or anthophyllite.

13.8.4 Wollastonite $CaSiO_3$

13.8.4.1 Wollastonite is one of the pyroxenoid minerals and has a characteristically bladed or prismatic morphology.

13.8.4.2 Wollastonite is colorless in plane light.

13.8.4.3 The refractive indices of wollastonite are very close to that of tremolite. However, wollastonite has a lower birefringence (difference between α and γ = 0.013 to 0.017) than tremolite.

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 Crystals of wollastonite can be spun about their long axis until they change from length slow to length fast or vice versa. Crystals of tremolite will always remain consistently length slow regardless of their optical orientation.

13.8.4.6 One way to spin a wollastonite grain about its long axis is to 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 crystals 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.

13.8.5.2 Kyanite usually has a bladed or columnar morphology.

13.8.5.3 Kyanite is colorless to light blue in plane light. Its blue color is much more subdued than that of crocidolite.

13.8.5.4 Kyanite has positive relief in 1.680 oil. Its refractive indices are higher than those of crocidolite or amosite (for kyanite, α = 1.710 to 1.718, γ = 1.724 to 1.734).

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 amphiboles, often found in soils in areas where certain types of igneous and metamorphic rocks are found. Hornblende is often found in soil samples from the Libby area.

13.8.6.2 Edenite, $NaCa_2(Mg,Fe^{2+})_5Si_7AlO_{22}(OH)_2$, is an amphibole that may be present at the mine at Libby (Meeker et al. 2003). Edenite is part of the hornblende group, and for this reason, for the purposes of this SOP, should not be classified as LA if it is encountered in a field sample.

13.8.6.3 Hornblende generally has slender prismatic to bladed crystals. The traces of cleavage planes are usually visible within of the crystals.

13.8.6.4 Hornblende does not occur in a highly fibrous morphology like LA often does.

13.8.6.5 Hornblende is distinctly colored and pleochroic. Hornblende is usually green, yellow-green, brown, green-brown, or blue-green in plane light.

13.8.6.6 Refractive indices vary with composition, but usually α = 1.645 to 1.665 and γ = 1.660 to 1.690. This is higher than LA.

13.8.6.7 Birefringence is moderate.

13.8.6.8 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 mentioned here because they are among the most common pyroxenes, but analysts should be aware that there are others.

13.8.7.2 Calcic clinopyroxene can be found in soils from areas where certain types of igneous and metamorphic rocks occur and has been found in field samples from the Libby area.

13.8.7.3 The morphology of calcic clinopyroxene is usually prismatic to columnar. As a group, the pyroxenes tend to form less slender, elongated crystals than the amphiboles. Traces of cleavage planes are usually visible within crystals of the pyroxenes.

13.8.7.4 Augite is colorless, pale green, greenish-brown, pale brown, or gray in plane light. Diopside is colorless, but as iron content increases through the diopside-hedenbergite, the mineral develops a green color.

13.8.7.5 Calcic clinopyroxene can be weakly pleochroic.

13.8.7.6 Calcic clinopyroxene has high refractive indices (α = 1.66 to 1.75, γ = 1.69 to 1.77). The pyroxenes as a group tend to have high refractive indices.

13.8.7.7 Birefringence is moderate, as with the majority of other pyroxenes.

13.8.7.8 Calcic clinopyroxene can have a very high extinction angle, up to 48 degrees.

13.8.7.9 Calcic clinopyroxene is generally length slow, but the sign of elongation becomes ambiguous in crystals showing a very high extinction angle.

13.8.8 Fiberglass (Amorphous Silica, SiO_2)

13.8.8.1 Fiberglass is almost always isotropic (appears black at all orientations under crossed polars).

13.8.8.2 Some fiberglass that is coated with other materials, or fiberglass that has been de-vitrified (partial re-crystallization of amorphous silica) due to prolonged exposure to very high temperatures, may show some slight interference colors under crossed polars.

13.8.8.3 The morphology of fiberglass is usually straight, solid, cylindrical tubes. Usually the diameter of the tube varies little along the length of the fiber.

13.8.8.4 Most fiberglass is colorless under plane light. However, the addition of impurities can impart various colors to fiberglass. Some can be yellow, dark brown, or dark green.

13.8.8.5 The RI of fiberglass varies considerably depending on the glass's composition (i.e. the addition of impurities, such as aluminum or iron). However, the RI of most types of fiberglass is close to 1.6.

13.8.9 Cellulose

13.8.9.1 Cellulose often has the morphology of ribbons that are wider than they are thick. The interiors of cellulose fibers often show a cellular or structured network.

13.8.9.2 Cellulose can be straight, curved, kinked, or crooked.

13.8.9.3 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.

13.8.9.4 Cellulose displays undulatory (incomplete) extinction.

13.8.9.5 Cellulose usually has a higher birefringence than chrysotile.

13.8.9.6 Fibers of cellulose will often show first order white or yellow or higher interference colors under crossed polars.

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. This diatomaceous earth is mined and is used in a variety of construction materials.

13.8.10.2 Not all diatoms are fibrous, but many are.

13.8.10.3 Fibrous diatoms generally have the morphology of cylindrical tubes, sometimes with tapered ends.

13.8.10.4 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.

13.8.10.5 Many diatom shells have complex internal structure.

13.8.10.6 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

13.8.12.1 Synthetic fibers can be any color, including clear, pink, red, purple, blue, green, yellow, etc.

13.8.12.2 Synthetic fibers typically lack the splayed ends that chrysotile bundles commonly exhibit. Many synthetic fibers display a cylindrical morphology.

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 The synthetic fiber polyethylene has a wispy habit very similar to that of chrysotile.

13.8.12.6 Polyethylene has a higher birefringence than chrysotile.

13.8.12.7 Polyethylene fibers will melt if the slide is placed on the hot plate whereas chrysotile will not.

13.8.13 Rutile (TiO_2)

13.8.13.1 Titanium oxide occurs naturally as the mineral rutile, TiO_2. Rutile generally occurs as small prisms or fine acicular needles.

13.8.13.2 Refractive indices are extremely high (α = 2.6 to 2.7, γ = 2.8 to 2.9).
13.8.13.3 Rutile can be gray, brown, reddish-brown, or nearly opaque.
13.8.13.4 Needles of rutile have high birefringence, are length slow, and show parallel extinction.
13.8.13.5 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 crystals and are referred to as rutilated quartz.

14.0 RECORDING DATA AND RESULTS

14.1 Data Recording Sheets

14.1.1 Analysts record, by hand, on analytical data recording sheets (bench sheets), analytical results at the time the observations are made. Refer to Attachment 1 for a PLM-VE data recording 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, record the following data for each asbestos type that is present in the sample.
14.3.2 Morphology
14.3.3 Fiber color
14.3.4 Pleochroism (Yes or No)
14.3.5 Indices of refraction (α and γ)
14.3.6 Birefringence
14.3.6.1 Low if birefringence is less than 0.010; medium if birefringence is 0.010 to 0.050; high if birefringence is greater than 0.050.
14.3.7 Extinction characteristics
14.3.7.1 Parallel or oblique/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
14.3.10 Bin assignment for LA (see Section 13.7.5)

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 QA type as Not QA, 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.
15.1.2 All of the data recorded on the bench sheet is entered into an EDD in the form of an Excel spreadsheet.
15.1.2.1 The EDD was developed specifically for the Libby project, and the laboratory should check with the client to be sure it is using the most recent version of the spreadsheet.
15.1.2.2 Only one EDD is produced for each work order number.
15.1.2.3 Data entry instructions are provided on the spreadsheet.
15.1.3 After entering all data into the EDD, save the file by clicking on the macro button located on “Visual_data entry” worksheet.
15.1.3.1 The file name is generated automatically by concatenating Information entered on the “General_data entry” worksheet.
15.1.3.2 The information used to create the file name is the laboratory name, work order number, and analysis type (visual estimation).
15.1.4 The directory where the macro will save the file depends on how the template spreadsheet was opened.
15.1.4.1 Be sure there is a blank spreadsheet template in each folder where EDD’s will be saved.
15.1.4.2 If Excel is opened, and then the blank template spreadsheet is opened, the file will be saved in the same directory where the original blank template spreadsheet was opened from.
15.1.4.3 Do not open the blank template spreadsheet from Windows Explorer, because then the file will be saved at the computer’s default directory for Excel (generally, this default directory is C:\Documents and Settings\My Documents).
15.1.5 The EDD serves as an electronic version of the test report submitted to the client.
15.1.5.1 A hard copy of the test report is also mailed or couriered to the client following delivery of the EDD (see Section 15.3 for further details about hardcopy data reports).
15.1.5.2 The laboratory retains all original records for use in resolving any questions until otherwise instructed by EPA.

15.2 Data Package Generation

15.2.1 Hardcopy reports of the raw analytical data are submitted to EPA, or their project oversight contractors, for archival.

15.2.2 A completed data package consists of a cover sheet signed and initialed by approved signatories and the following documentation:

15.2.2.1 Number of samples received, and copies of the signed chains of custody.

15.2.2.2 The date of sample receipt and condition of samples.

15.2.2.3 The 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 Verification that microscope slides were wiped clean before use.

15.2.2.5 Calibration data for the RI liquids used in the analysis.

15.2.2.6 Verification that the microscope was properly calibrated before use.

15.2.2.7 Verification that reference materials were used for comparison when performing calibrated visual estimates of asbestos content.

15.2.2.8 Visual Estimate hard copy data forms, as presented in the EDD and containing the analytical data (including all cross-check and self-check QC’s performed on any samples in the work order number).

15.2.2.9 Copies of the handwritten bench sheets containing the analyst’s original data and observations.

15.2.3 Refer to Attachment 3, the Data Package Checklist, for a complete list of items required for each data package.

15.2.4 Each test report is identified by a unique Laboratory Information Management System (LIMS) number called a Work Order Number, Job Number, or equivalent.

15.2.5 When opinions and interpretations are provided in a test report, the laboratory will:

15.2.5.1 Document the basis on which the opinions and interpretations were made.

15.2.5.2 Clearly indicate on the test report which items are opinions and interpretations.

15.2.6 Once the data package 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 EPA’s data management software.

15.3.1.2 A scanned .pdf of all items in the data package described above, including the cover sheet signed by an approved signatory, the signed chains of custody, and the analyst’s original bench sheets. 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, a hardcopy of the data package is sent to the client through the mail.

16.0 QUALITY ASSURANCE AND QUALITY CONTROL

16.1 General

16.1.1 The laboratory operates 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 (self-check analysis) or by a different analyst (cross-check analysis).

16.1.3.2 Repeated analyses on calibration standards of known asbestos concentration.

16.1.3.3 NIST proficiency testing.

16.1.3.4 Inter-laboratory analyses.

16.1.4 Records are 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 Libby Amphibole 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 Regular analysis of the calibration standards provides a routine check of analyst precision and accuracy.

16.3.1.3 Calibration standards are read at a frequency on 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.

16.3.4 The Laboratory Manager, QA/QC Coordinator, or designate other than the analyst performing the test, will 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. For example, the reported value of blind calibration standards below 1% should fall in the correct concentration bin.

16.3.6 Repeated analysis of the calibration standards provides a benchmark upon which analysts may 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 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 data package. Rather, laboratories are responsible for maintaining an internal system for tracking analyses of these calibration standards.

16.4 Self-Check and Cross-Check QC Analyses (Duplicates and Replicates)

16.4.1 For each set of samples, 10% of the samples must be re-analyzed within the laboratory.

16.4.2 A QC analysis (self-check or cross-check) 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 self-check QC analyses on client samples will be 1 per 50 samples analyzed (2%). Self-check analyses should be performed as a remount of the sample (see Section 13.3 for slide preparation procedures).

16.4.4 The frequency of cross-check QC analyses on client samples will be 8 per 100 samples analyzed (8%). Cross-check analyses should be done on the five original slide preparations.

16.4.4.1 All analysts performing QC analyses must be experienced with PLM analysis of soil samples from the Libby Asbestos Superfund 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 cross-check QC samples to another Libby laboratory with the proper experience and qualifications.

16.4.5 The self-check and cross-check analysis is acceptable if results are within a bin

category (i.e., ± 1 bin) for reported concentrations below 1% LA. For all asbestos types greater than 1%, it is recommended that precision is tracked using control charting or a similar tool.

16.4.6 Corrective action(s) must be taken immediately if re-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 to EPA, or their designate.

16.4.7 When performing a QC analysis, it is necessary to mark LDS or LDC in the "QA 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 Asbestos Superfund Site. The purpose of this program is to help detect and minimize laboratory biases and 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 EPA or their designate.

16.5.3 Results of the inter-laboratory analyses are reviewed by EPA, or their designate.

16.5.4 The inter-laboratory analysis is acceptable if results are within a bin category (i.e., ± 1 bin) for reported concentrations below 1% LA.

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 EPA, or their designate. 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 analyst re-training.

17.0 REFERENCES

17.1 Bandli, B.R. et al. (2003). Optical, compositional, morphological, and X-ray data on eleven Eleven particles of amphibole from Libby, Montana, U.S.A. Canadian Mineralogist, 41, 1241-1253.

18.2 Bloss, F.D. (1999). Becke Line Chart. Optical Crystallography. Mineralogical Society of America, Monograph Series Publication No. 5.

18.3 Cornelis Klein and Cornelius S. Hurlbut, Jr. (1985). Manual of Mineralogy. John Wiley & Sons.

18.4 Deer, W.A., Howie, R.A., and Zussman, J. (1997). Rock Forming Minerals Volume 2B: Double Chain Silicates. 2nd Edition. The Geological Society, London.

18.5 EPA Method 600/R-93/116: Method for the Determination of Asbestos in Bulk Building Materials. (July 1993).

18.6 Federal Register, 40 CFR Part 763, Volume 52, No 210, "Asbestos Containing Materials in Schools; Final Rule and Notice."

18.7 McCrone Research Institute (1987). Dispersion Staining Color Chart.

18.8 Meeker, G.P., Bern, A.M., Brownfield, I.K., Lowers, H.A., Sutley, S.J., Hoefen, T.M., and Vance, J.S. (2003). The Composition and Morphology of Amphiboles from the Rainy Creek Complex Near Libby Montana. American Mineralogist, 88, 1955-1969.

18.9 NIOSH Method 9002: Asbestos (bulk) by PLM. Manual of Analytical Methods, 4th Edition. (August 1994).

18.10 NIST Handbook 150, 2006 Edition, "NVLAP Procedures and General Requirements."

18.11 State of California Air Resources Board Method 435: Determination of Asbestos Content of Serpentine Aggregate. (June 1991).

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

18.13 Su, Shu-Chun (1996). Rapidly and Accurately Determining Refractive Indices of Asbestos Fibers by Using Dispersion Staining Method.

18.14 Terry and Chilingar (1955). Comparison Chart for Visual Percentage Estimation.

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

18.16 Wylie, A.G. and Verkouteren, J.R. (2000). Amphibole asbestos from Libby, Montana: Aspects of nomenclature. American Mineralogist, 85, 1540-1542.

19.0 LIST OF ATTACHMENTS

19.1 Attachment 1: PLM-VE Data Recording Sheet

19.2 Attachment 2: RI Liquid Calibration Conversion Tables

19.3 Attachment 3: Data Package Checklist from PLM Data Sheet and EDD

19.4 Attachment 4: Optical Properties of Fibrous Amphiboles

19.5 Attachment 5: PLM Photomicrographs Demonstrating a Wide Range of Libby Amphibole Morphologies

19.6 Attachment 6: SEM Photomicrographs of Representative Examples of Libby Amphibole Morphologies

19.7 Attachment 7: Photomicrographs of Representative Fields of View of 0.2% and 1.0% Libby Amphibole Controlled PE Reference Materials

19.8 Attachment 8: Flow Diagram for Determining LA Content by Complementary Use of Stereomicroscopic Examination and PLM Visual Estimation

ATTACHMENT 1

PLM-VE Data Recording Sheet

PLM VISUAL ESTIMATION DATA RECORDING SHEET

Laboratory Name _____ Date Received _____

Job Number _____ SOP Name/Revision _____

Note: Data Recording Sheet is formatted to print on 11x17 paper.

EPA Index ID	Index Suffix Char.	Index Suffix No.	QA Type (NOT QA, LDS, LDC)	Lab Sample ID	Date Analyzed	Analyst Name	Stereomicroscopy Examination					Libby Amphibole		Other Amphibole			Chrysotile		Deviation?	Comments (list below)	OPTICAL PROPERTIES FOR LA (see key for appropriate data inputs)							
							Sample Appearance	Est. % LA		Est. % OA and C											Morph.	Fiber Color	Sign Elong. (+/)	Pleoch. (Y/N)	Angle Extinct.	Ref. Index α	Ref. Index γ	Biref.
								Qual (ND, Tr, <)	Mass Fract (%)	Qual (ND, <)	Area Fract (%)	Qual (ND, Tr, <)	Mass Fract (%)	Qual (ND, <)	Area Fract (%)	OA Type (AMOS, ANTH, CROC, UNK)	Qual (ND, <)	Area Fract (%)										

Comments (Use back if needed)

ATTACHMENT 2

RI Liquid Calibration Conversion Tables

Prepared by Dr. Shu-Chun Su, Hercules, Inc.

See attached Excel spreadsheet entitled

"Create_RI_Liquid_Calibration_Conversion_Tables.xls"

ATTACHMENT 3

Data Package Checklist

From PLM (VE and PC) Data Sheet and EDD.xls

STANDARD LABORATORY DATA PACKAGE CHECKLIST
Analytical Test Report
Bulk Asbestos Analysis by Polarized Light Microscopy (PLM)

Prepared For:

City/State: _____

Laboratory Name: _____
City/State: _____
Laboratory Job No.: _____
Method Utilized (SOP and Rev. No.): SRC-LIBBY-03/Revision 2
Circle One: Visual Estimation Point Counting Approach

Report Reviewed by: _____

STANDARD LABORATORY DATA PACKAGE CHECKLIST

Instructions: **For PLM analytical results raw data packages, complete and sign the following checklist. Attach supporting documentation as outlined below. Organize the supporting documentation in the order listed below. Paginate the completed raw data package.**

		Laboratory Verification (Initials and Date)	Validator Verification (Initials and Date)
1	**Number of samples received:**_____ *An SDG is defined as no more than 200 samples.* **Additional Supporting Documentation:** Attach COC forms having footer R (report).		
2	**Date of sample receipt and condition of samples:**_____ *For Condition of samples enter "OK" or "See SDG Case Narrative".*		
3	**SDG Case Narrative:** **Additional Supporting Documentation:** Attach SDG Narrative and any modification forms.		
4	**Check for contamination (daily):** Wipe microscope slides with lens paper before using. *Laboratory Verification initial and date signifies that this has been performed for the samples in this SDG.*		
5	**Verification of the refractive indices of the refractive index liquids once per month:** **Additional Supporting Documentation:** Provide information indicating a monthly record of checking each of the four liquids including liquid name, lot number and analyst initials. (See table - Results of RI Liquids Calibration)		
6	**Verification of microscope adjustments prior to each SDG:** *Laboratory Verification initial and date signifies that this has been performed for the samples in this SDG.*		
7	**Reference material - Visual Estimation Approach:** *Laboratory Verification initial and date signifies that this has been performed for the samples in this SDG.*		
	Reference material - Point Counting Approach: **Additional Supporting Documentation:** Provide calibration curve documentation, printed from the EDD spreadsheet.		
8	**VE and/or PC hard copy data forms (as presented in the EDD spreadsheet):** **Additional Supporting Documentation:** Copies of the Hard Copy Data Forms for all investigative samples and laboratory duplicates will be provided from systems that are entered electronically.		
9	**Bench sheets for data results:** **Additional Supporting Documentation**: Provide copies of the hand written or LIMS system generated raw data sheets for sample results.		

COCs

SDG NARRATIVE

Instructions: The following information should be included in all narratives. Please see the attached narrative template.

1 List the method or methods used.
2 For any modifications, reference the modification number and attach a copy of the signed document to the raw data
3 If sample condition is not "OK", explain why and any implications to the data.

SDG NARRATIVE EXAMPLE

SDG Narrative - PLM Analysis by SRC-LIBBY-03 Revision 2

Laboratory Job Number:_____

The samples were received in sealed coolers [or other container]. [Any special notations about the samples as received goes here such as damaged in shipping, missing sample, etc.] The sample set was assigned a laboratory job number, each sample was assigned a unique, sequential laboratory ID number, and the job was entered into the Laboratory Information System. The laboratory ID numbers, shipping information and signatures were recorded on the CDM Chain of Custody and the login information was summarized on the laboratory Chain of Custody.

Samples were analyzed in accord with SRC-LIBBY-03 Rev. 2 [with modifications described on Laboratory Modification document(s): LB-_____ (see attached)].

SAMPLE RESULTS

See Attached Sample Results

Instructions: These sample result forms are from the current version of the PLM (VE & PC) Data Sheet and EDD.xls file. They are labeled in this file as the VE or PC hard copy data form.

BENCH SHEETS

Instructions: Please provide handwritten or LIMS system generated raw data sheets for sample results.

REFRACTIVE INDEX LIQUIDS

Instructions: Please see and follow attached table from Shu-Chun Su, Technical Expert for NVLAP Asbestos Programs. (Suggested Format for Recording Results of RI Liquids Calibration using Cargille Glass Standard and Dispersion Staining Method - Version: February 1996)

The following components are included in the table:

1 Date
2 Nominal or Labeled n_D 25 degree Celsius
3 Cargille Glass
3a Nominal or Labeled R.I.
3b Lot No.
4 Central Stop DS Observation
4a Predominant DS Color
4b Corresponding $alpha_0$
5 Liquid or Room Temperature (degree Celsius)
6 Actual or Calibrated n_D 25 degree Celsius
7 Difference between Calibrated n_D 25 degree Celsius and Labeled n_D 25 degree Celsius
8 Accept or Reject
9 Analyst

RESULTS OF RI LIQUIDS CALIBRATION

Date[1]	RI Liquid[2]	Cargille Glass		CSDS Color of Glass4		Liquid or Room Temperature (°C)[7]	Calibrated RI of Liquid $(n_D^{25°C})_{clb}$[8]	Absolute Difference Between Calibrated and labeled RI $(n_D^{25°C})_{clb} - (n_D^{25°C})_{lbl}$[9]	Accept or Reject[10]	Initials of Analyst[11]
	Labeled RI $(nD25°C)_{lbl}$	Labeled RI[3]	Lot No.[4]	Predominant CSDS Color[5]	Corresponding λ_0 (nm)[6]					
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	

1. Date: 2. The $n_D^{25°C}$ on the label of RI liquid bottle or $(n_D^{25°C})_{lbl}$; 3. The RI value on the label of Cargille calibrated glass vial; 4. The Lot No. on the label of Cargille calibrated glass vial; 5. The predominant central stop dispersion color displayed by glass fragments (do not be confused by the false CSDS color due to edge effect (see p.3). 6. The matching wavelength, λ_0, corresponding to the CSDS color in Column 5; 7. The temperature of the RI liquid or the room if the liquid's temperature can be considered to be in equilibrium with the room atmosphere; 8. The reading based on the values in Columns 6 and 7 from the conversion table for the liquid-glass combination. This value is the actual or calibrated RI of the liquid at 589 nm and 25 °C or $(n_D^{25°C})_{clb}$; 9. Column 8 minus Column 2; 10. If the *absolute* value of Column 9 is less or equal to 0.004, circle A for *acceptable*. Otherwise, circle R for *rejected*. 11. Analyst's initials.

$(nD25°C)_{lbl} = n_D^t + (25 - t)\, dn/dt$ (The temperature correction is built in the conversion tables.)

Version: December 1998 (Shu-Chun Su, Technical Expert for NVLAP Asbestos Programs)

ATTACHMENT 4

Optical Properties of Fibrous Amphiboles

OPTICAL PROPERTIES OF FIBROUS AMPHIBOLES ASSOCIATED WITH LIBBY AMPHIBOLE[A]

Libby Amphibole asbestos (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 a group of minerals, such as tremolite, actinolite, winchite, richterite, magnesio-riebeckite, and magnesio-arfvedsonite. The optical properties for each individual mineral are provided below. As seen, 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 under this SOP. Rather, if the sample exhibits the optical properties of a mineral listed below, the specific optical properties (refractive indices, birefringence, extinction angle, and elongation sign) shall be noted on the analytical data sheet and electronic file, and the mineral identified as LA.

Mineral	Morphology and Color	Refractive Indices		Birefringence	Extinction	Elongation Sign
		α	γ			
Tremolite[7]	Straight to curved fibers and bundles. Colorless to pale green.	1.600-1.628 1.604-1.612 1.599-1.612 1.6063	1.625-1.655 1.627-1.635 1.625-1.637 1.6343	0.017-0.028	Oblique (up to 21 °);	+ (length slow)
Actinolite[7]		1.600-1.628 1.612-1.668 1.613-1.628 1.6126	1.625-1.655 1.635-1.688 1.638-1.655 1.6393	0.017-0.028		+ (length slow)
Winchite	Straight to curved fibers or bundles. Colorless to pale blue Pleochroism weak to moderate: X-colorless, Y=light blue-violet, Z=light blue[3]	**1.618-1.626**[1] **1.618-1.621**[2] 1.629[3] 1.636[4]	**1.634-1.642**[1] **1.634-1.637**[2] 1.650[3] 1.658[4]	**0.008-0.019**[1] **0.016**[2] 0.021[3] 0.022[4]	Oblique, **22°**[1] **15.8°**[2] Oblique, 7-29°[8]	+ (length slow)
Richterite	Straight to curved fibers or bundles. Colorless, pale yellow, brown, pale to dark green, or violet[8] Pleochroism weak to strong in pale yellow, orange, and red[5]	**1.622-1.623**[1] 1.605-1.624[5] 1.615[6]	**1.638-1.639**[1] 1.627-1.641[5] 1.636[6]	**0.012-0.017**[1] 0.017-0.022[5]	Oblique, **21-22°**[1] Oblique, 5-45°[8]	+ (length slow)
Magnesio-riebeckite	Prismatic to fibrous aggregates. Blue, grey-blue, pale blue to yellow. Can be pleochroic.[8]	1.650-1.673[8]	1.662-1.676[8]	Up to 0.015[8]	Oblique, 8-40°[8]	- (length fast)[8]
Magnesio-arfvedsonite	Prismatic to fibrous aggregates. Yellowish green, brownish green, or grey-blue. Can be pleochroic.[8]	1.623-1.660[8]	1.635-1.680[8]	0.012-0.026[8]	Oblique, 18-45°[8]	- (length fast)[8]

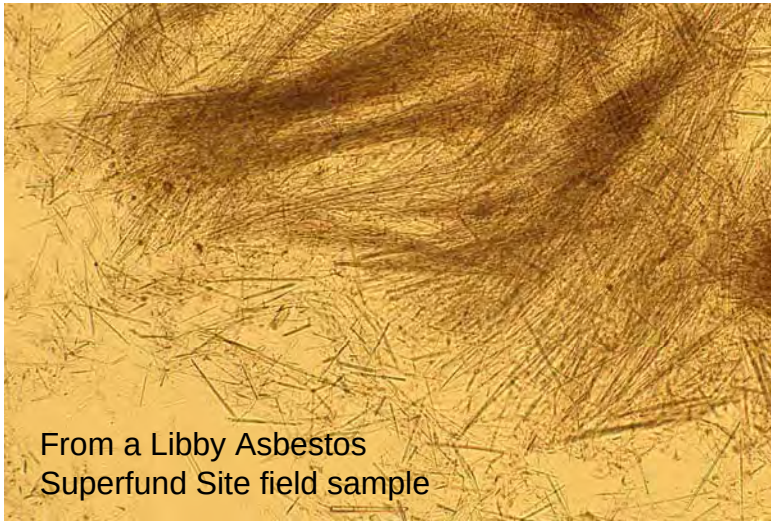
A. This table is adapted for use in the SOP from: Su, Shu-Chun, 2005. White paper: *Tables to Facilitate the Determination of Refractive Indices of Winchite and Richterite, (Libby, Montana) by Dispersion Staining*, **August 8, 2005** Data on this table were compiled from data of amphiboles from Libby, Montana and other localities. The data in **bold** are samples from Libby, Montana. The data of tremolite/actinolite are adapted from Table 2-2 of EPA/600/R-93/116.

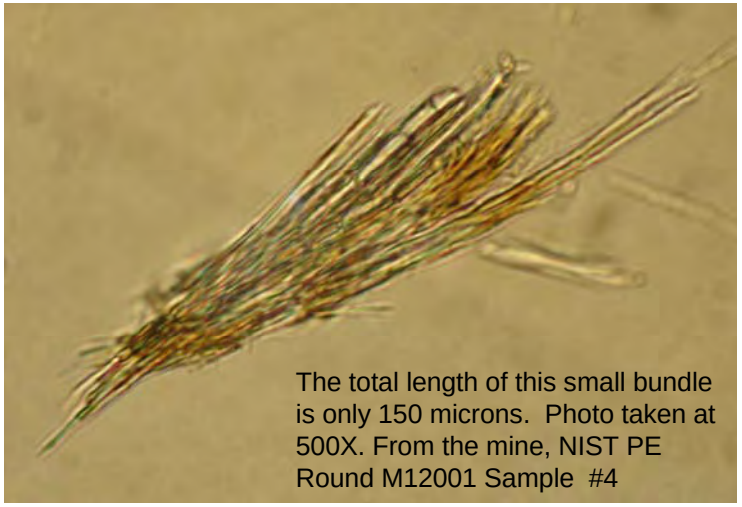
1. Bandli, B.R. et al. (2003) *Optical, compositional, morphological, and X-ray data on eleven particles of amphibole from Libby, Montana, U.S.A.* Canadian Mineralogist, 41, 1241-1253.
2. Wylie, A.G. and Verkouteren, J.R. (2000) *Amphibole asbestos from Libby, Montana: Aspects of nomenclature.* American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

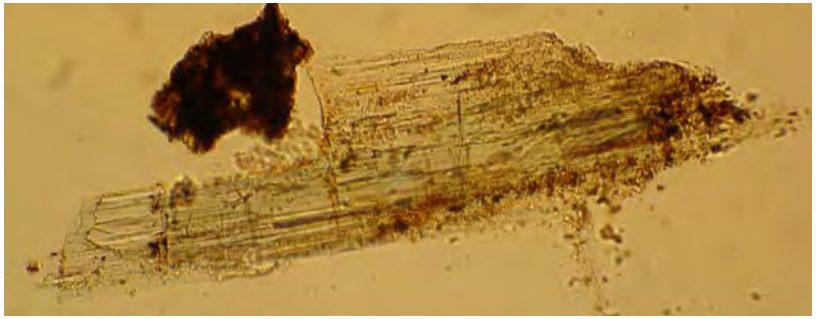
PLM Photomicrographs of Typical Libby Amphibole Morphology





Prismatic Libby Amphibole

The optical properties are the same as they are for more fibrous forms of LA. Colors of winhcite, 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 Libby Amphibole.



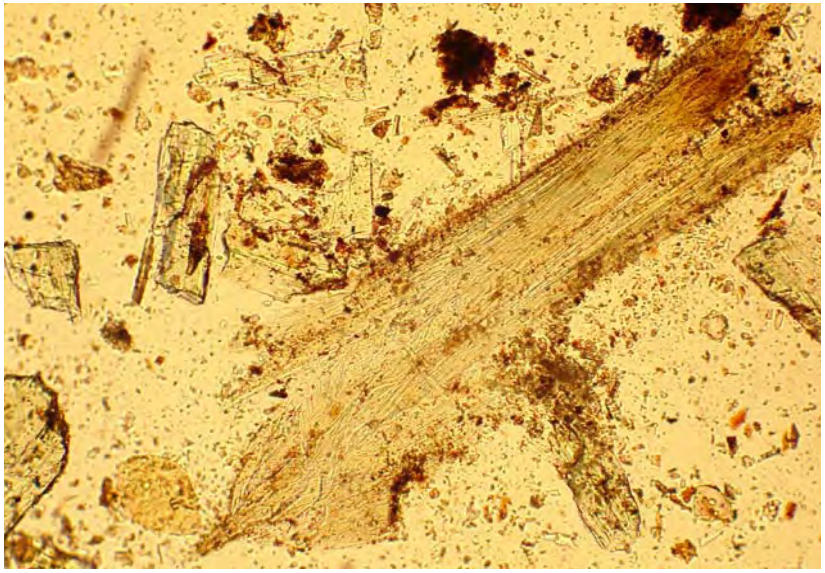
From a Libby Asbestos Superfund Site field sample

From the mine, NIST PE Round M12001 Sample #4

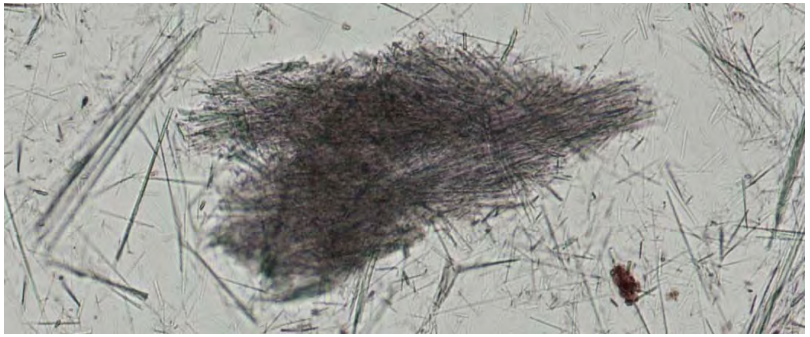
From the mine, NIST PE Round M12001 Sample #4

From a Libby Asbestos Superfund Site field sample

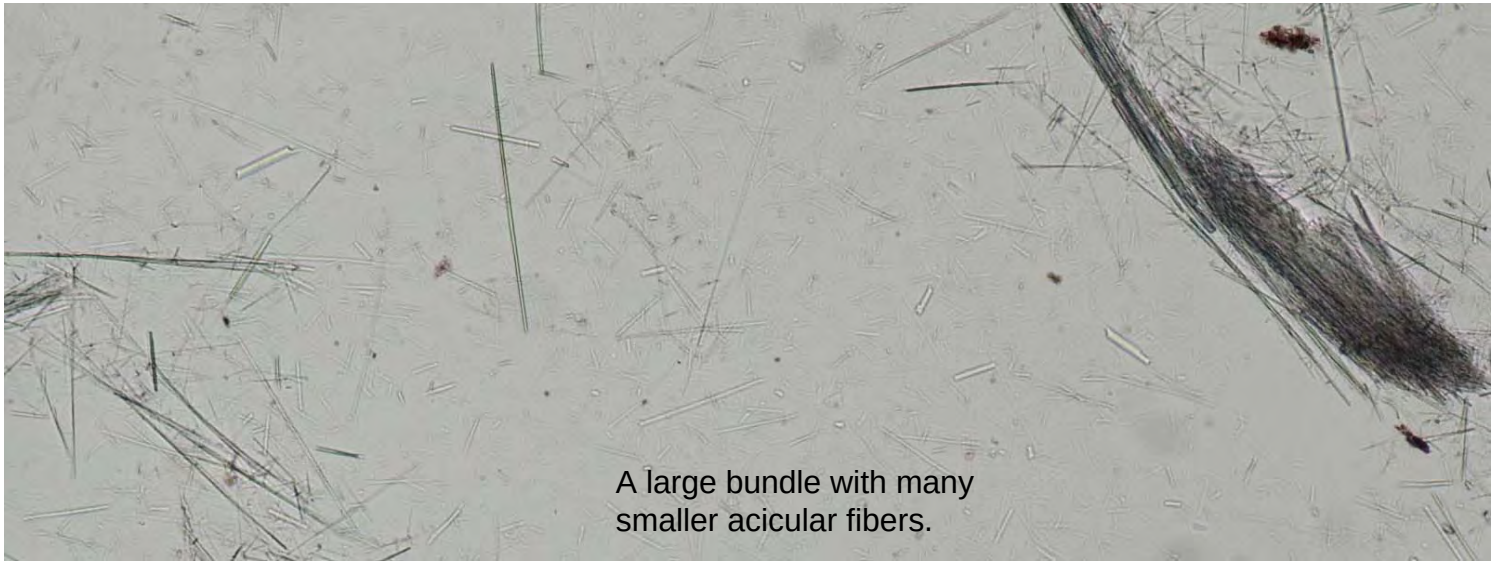
Some Libby Amphibole shows a “matted” or “felted” morphology. The internal structure of these bundles is still fibrous. The green high-relief prismatic crystals in the top right photo are hornblende. The bundles in the two top photos were found in Libby Asbestos Superfund Site field samples. The bundles 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.

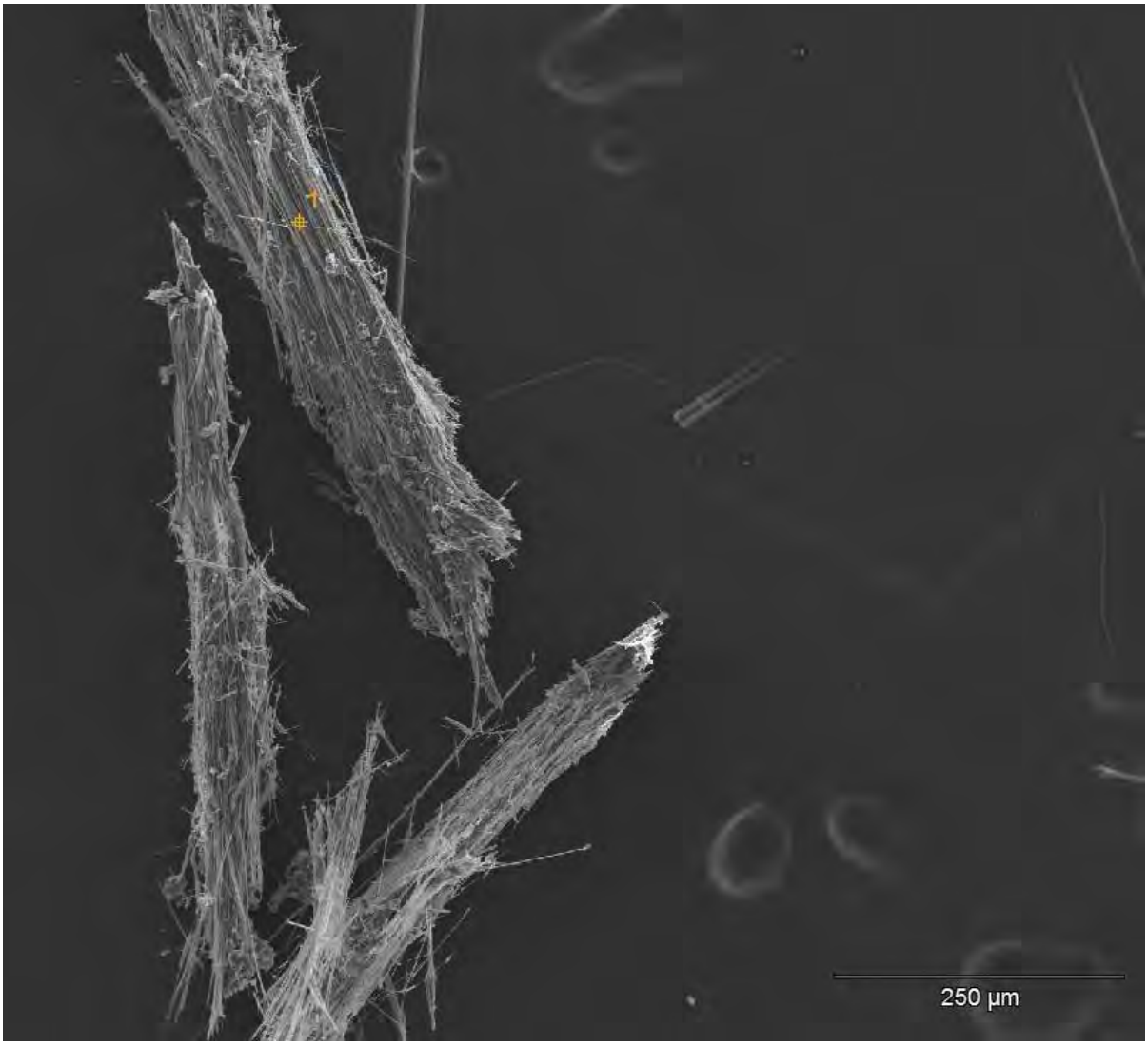
ATTACHMENT 6

SEM Photomicrographs of Representative Examples of Libby Amphibole Morphologies

SEM Photomicrographs of Representative Examples of Libby Amphibole Morphology

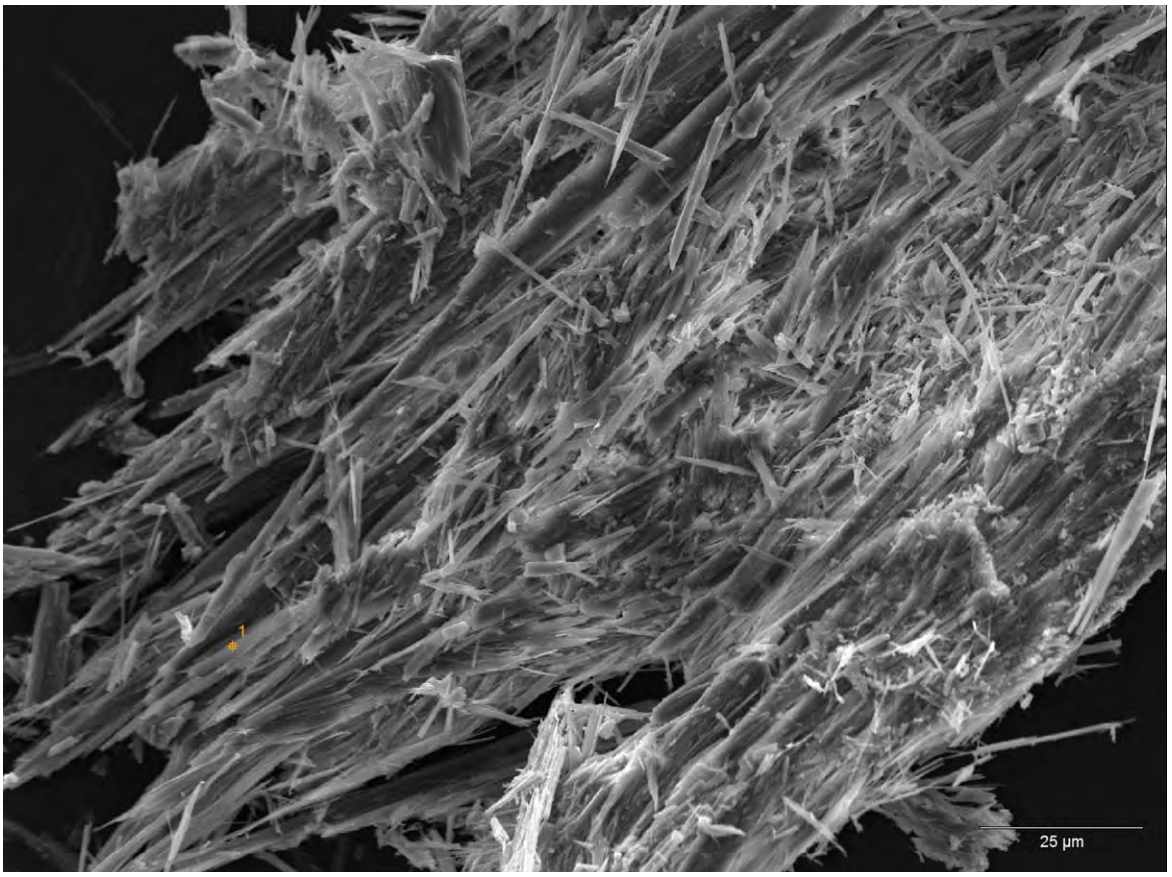
Individual bundles of Libby Amphibole were picked from soil samples at the ESAT Region 8 Laboratory and prepared for analysis by scanning electron microscopy (SEM). Slide mounts of these bundles were initially prepared in a refractive index liquid and the bundles were examined by PLM. Then the refractive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

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 morphologies 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.



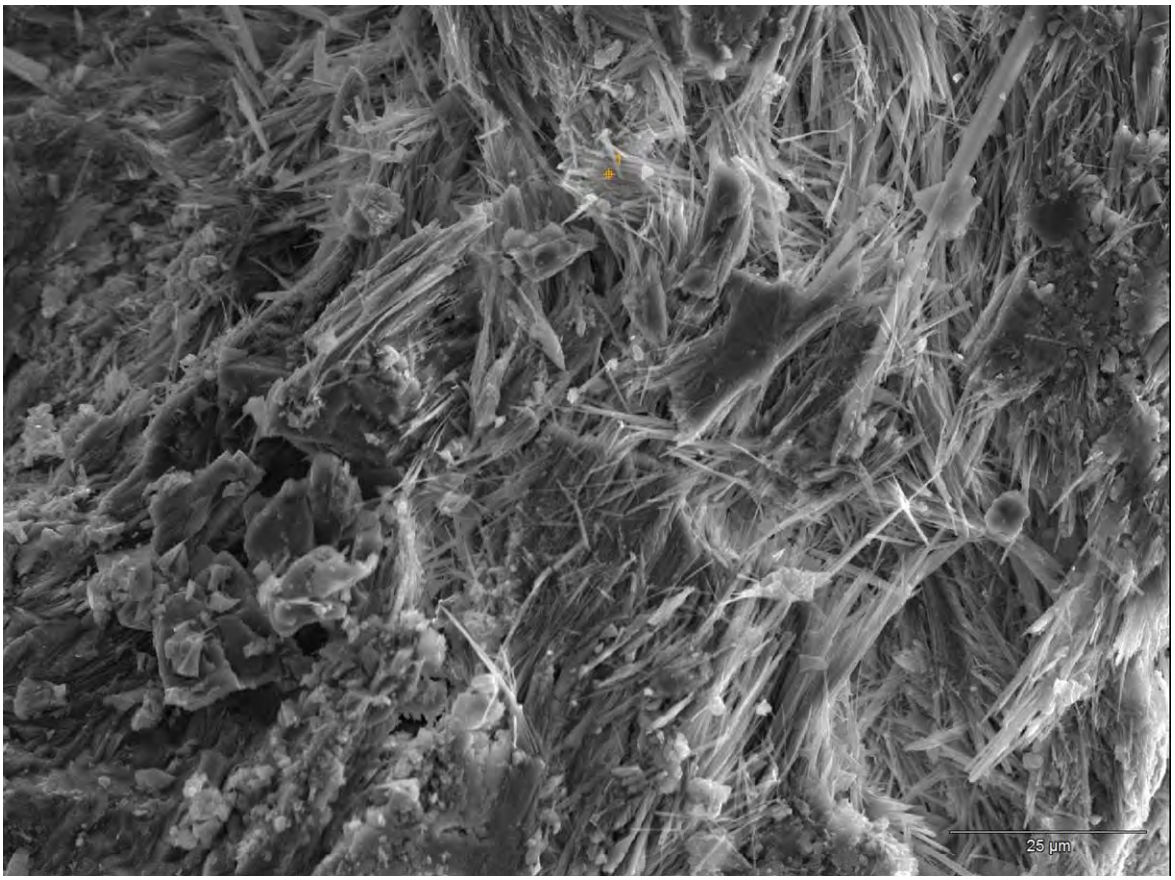
These are typical bundles of Libby Amphibole where the average aspect ratio of the fibers is high and most of the fibers are nearly parallel to one another. Note the scale in microns at the bottom of the photo. These three bundles are all of a size that can be seen with a stereomicroscope and picked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



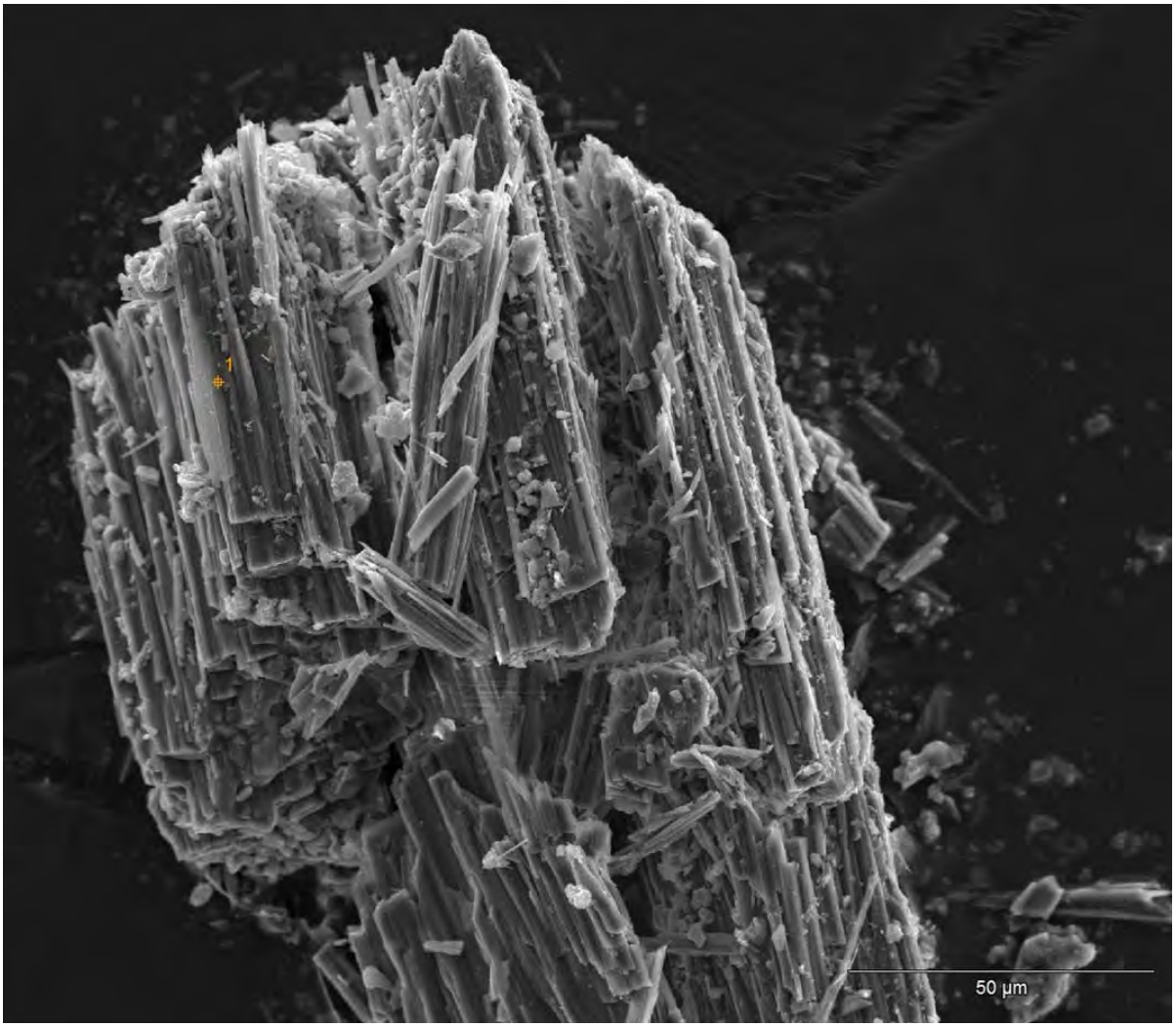
Varying degrees of parallelism can be seen in the fibers that compose bundles of Libby Amphibole. Note that the fibers in this bundle of LA are less parallel than the fibers in the bundles in the previous example.

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



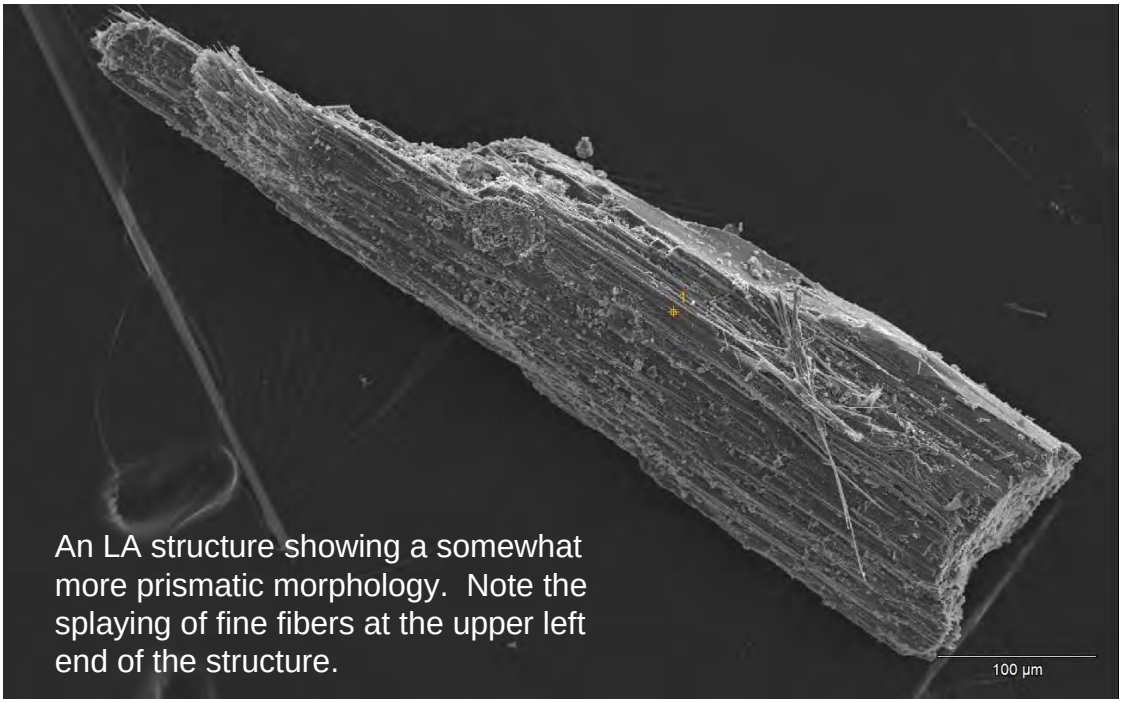
When this bundle of Libby Amphibole was viewed under PLM, its morphology was described as “felted”, or “matted”, with the fibers crossing at high angles to one another. This is how the bundle appeared when it was subsequently viewed by SEM. The fibrous nature of the “felted” or “matted” morphology is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

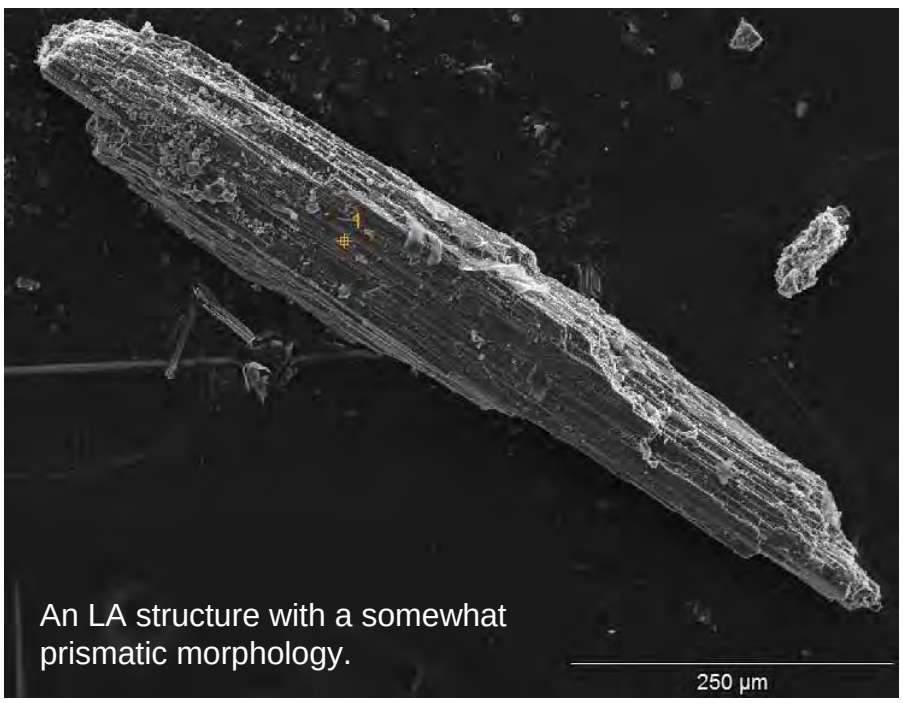


The average aspect ratio of the fibers in this bundle of LA is lower than those of the bundles in the previous examples. However, as seen by SEM, the bundle still splits readily into many small fibers.

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

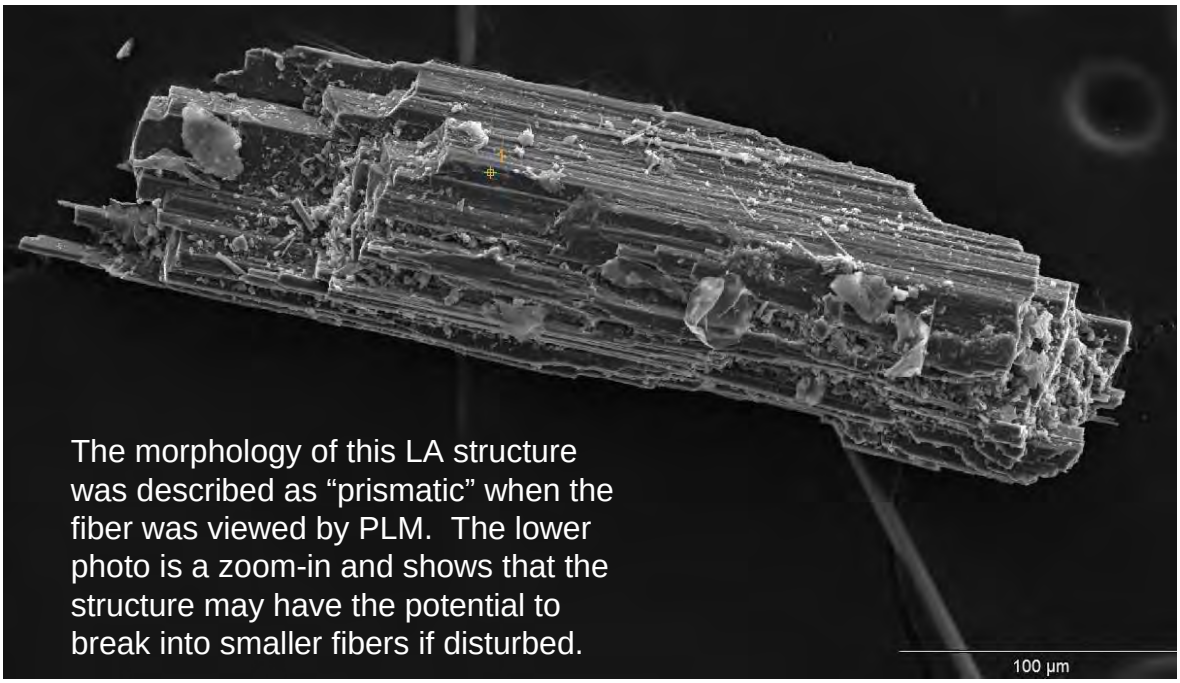


An LA structure showing a somewhat more prismatic morphology. Note the splaying of fine fibers at the upper left end of the structure.

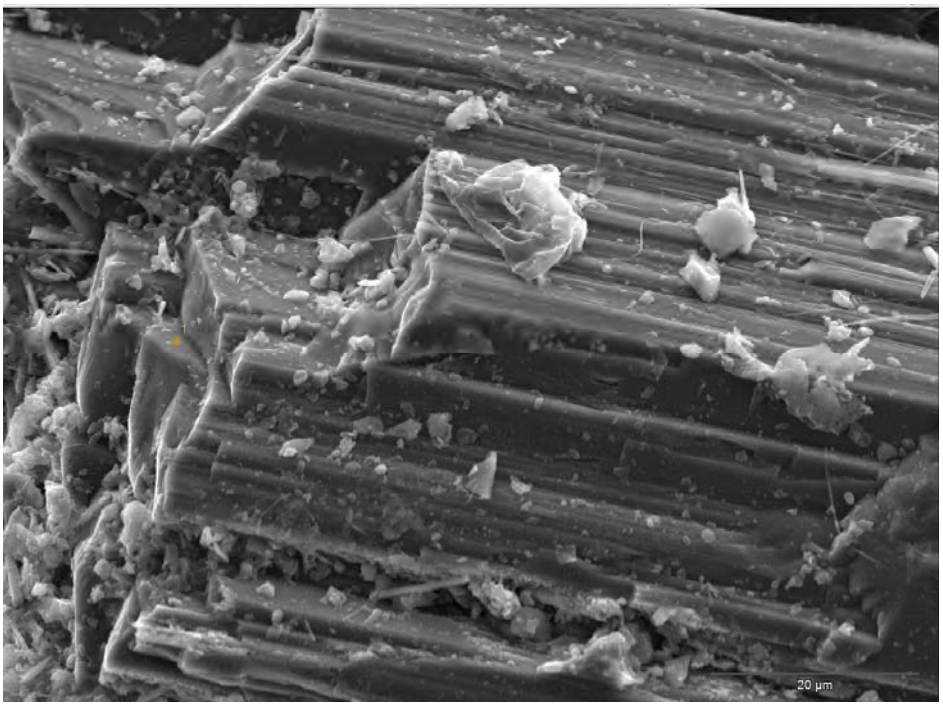


An LA structure with a somewhat prismatic morphology.

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



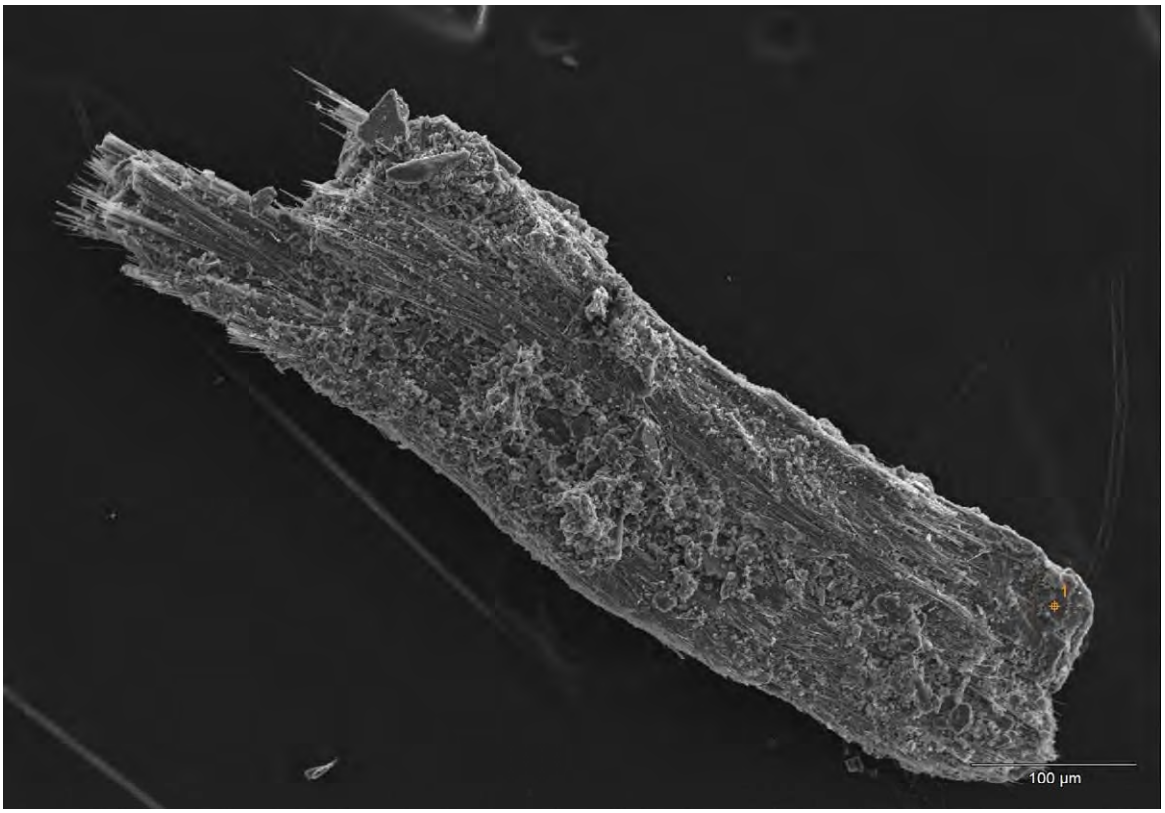
The morphology of this LA structure was described as “prismatic” when the fiber was viewed by PLM. The lower photo is a zoom-in and shows that the structure may have the potential to break into smaller fibers if disturbed.



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. Energy dispersive spectrometry (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 Libby Amphibole.

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 Libby Amphibole.

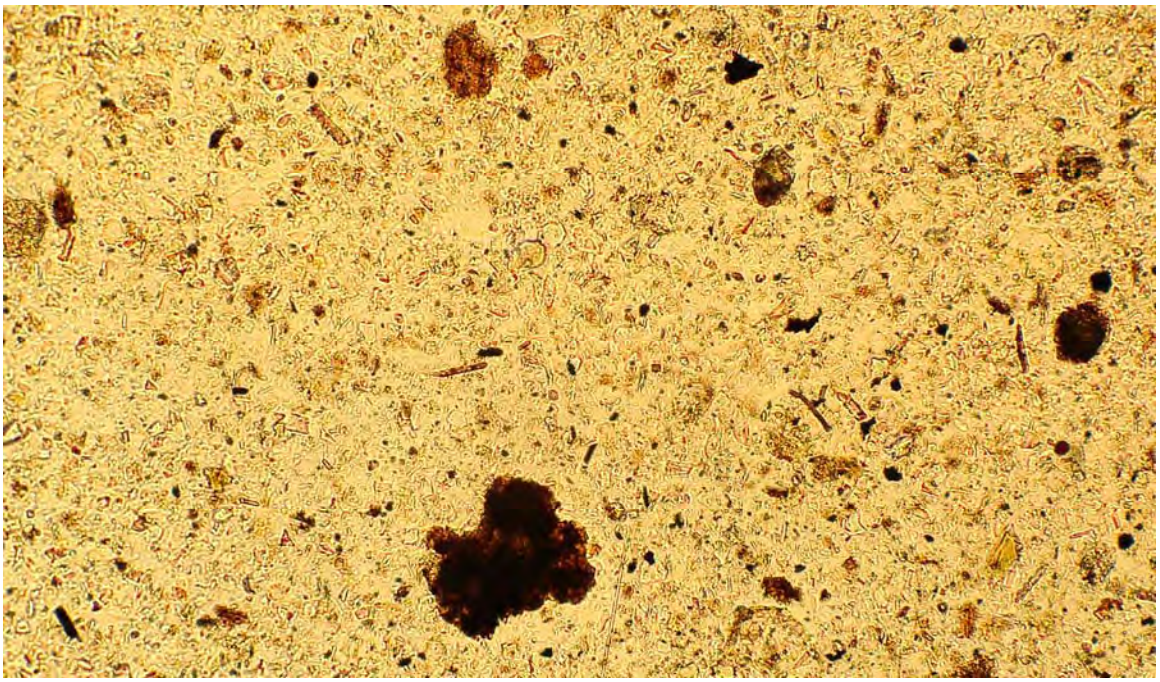
Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.

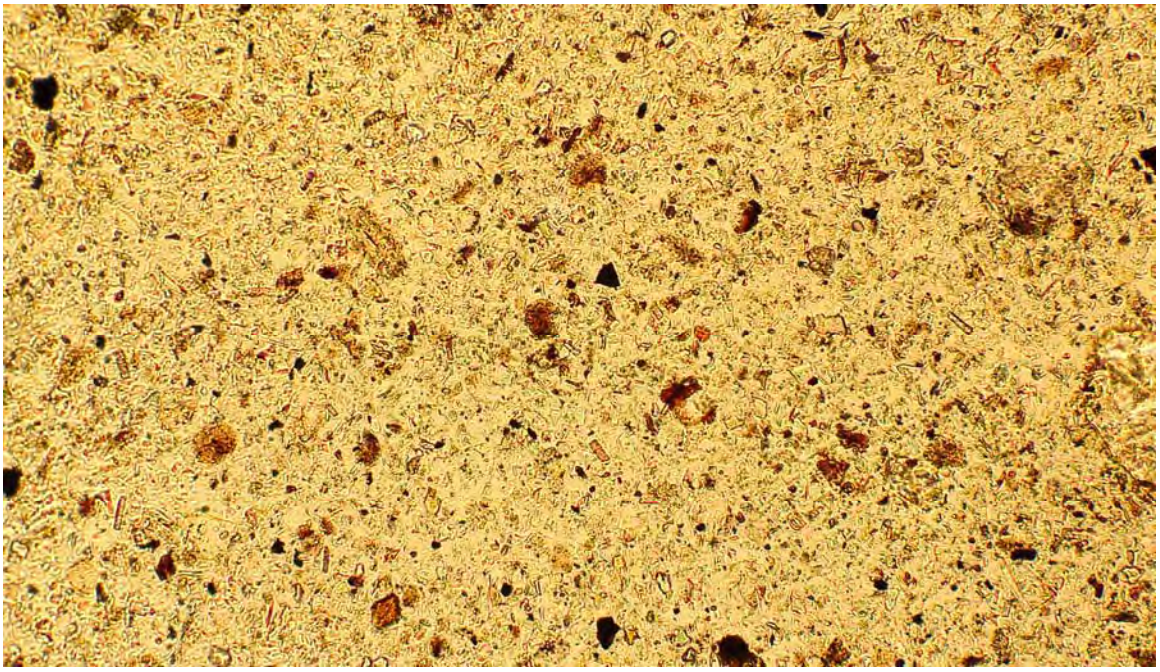
ATTACHMENT 7

Photomicrographs of Representative Fields of View of 0.2% and 1.0% Libby Amphibole Reference Materials

0.2% Libby Amphibole

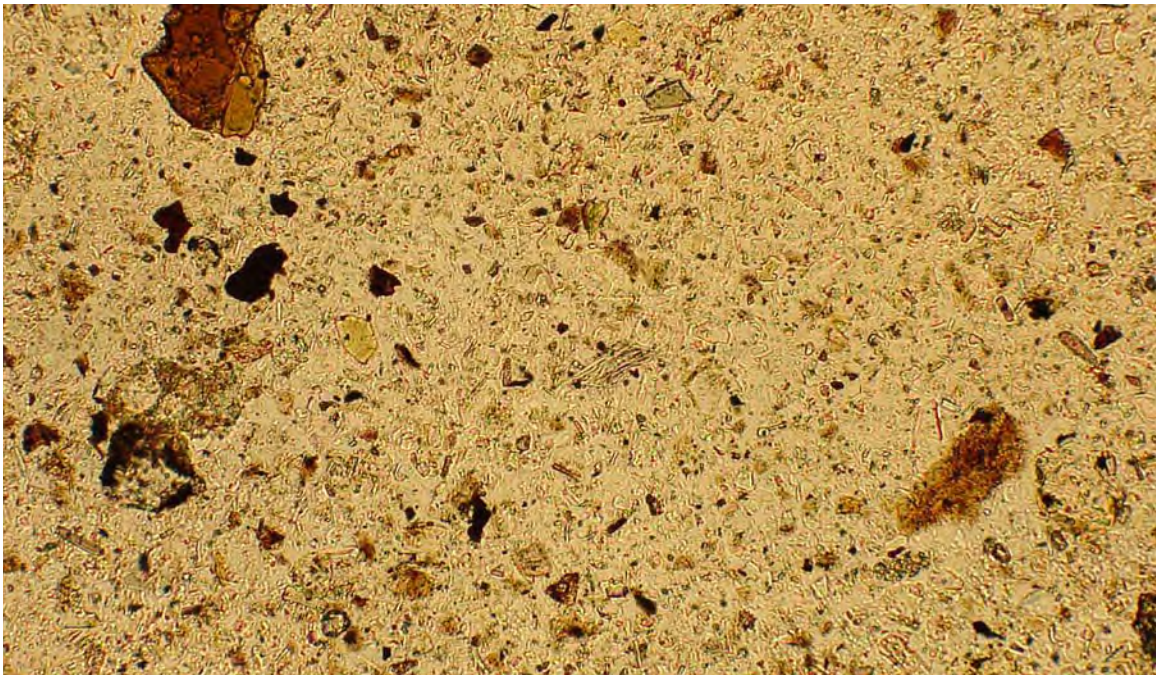
Photomicrographs of representative fields of view of the 0.2% Libby Amphibole by weight Controlled PE Reference Material. All photos taken at 100x, plane light in 1.55 refractive index oil. Width of each picture is approximately 1,500 microns.

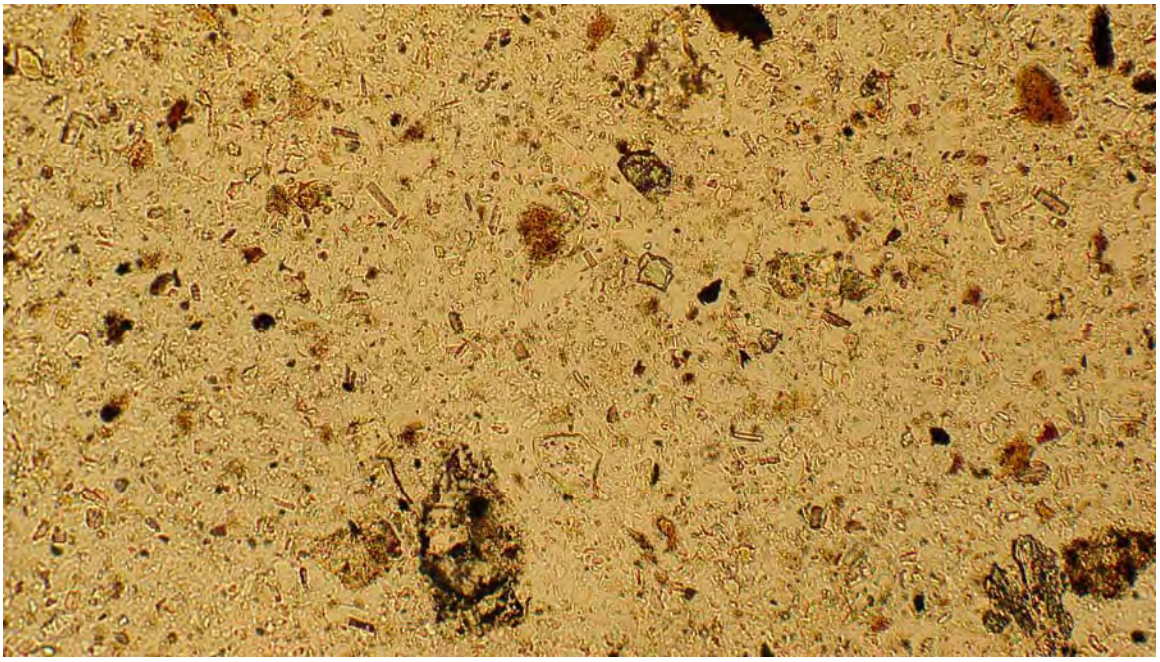




0.2% Libby Amphibole

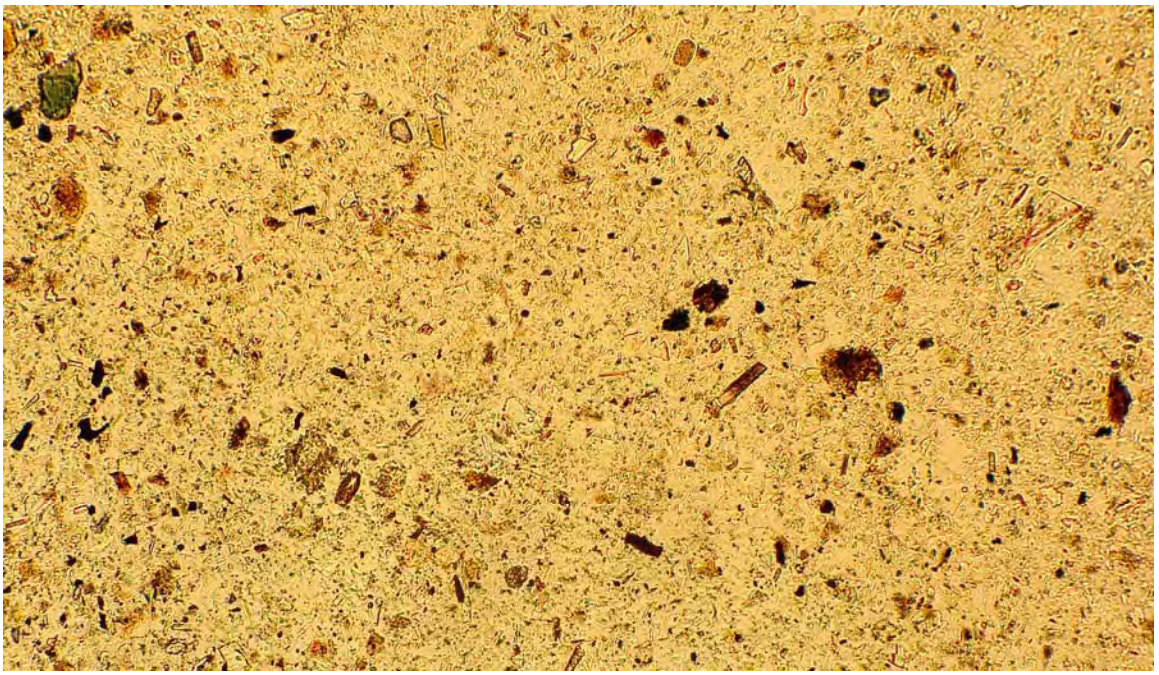
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

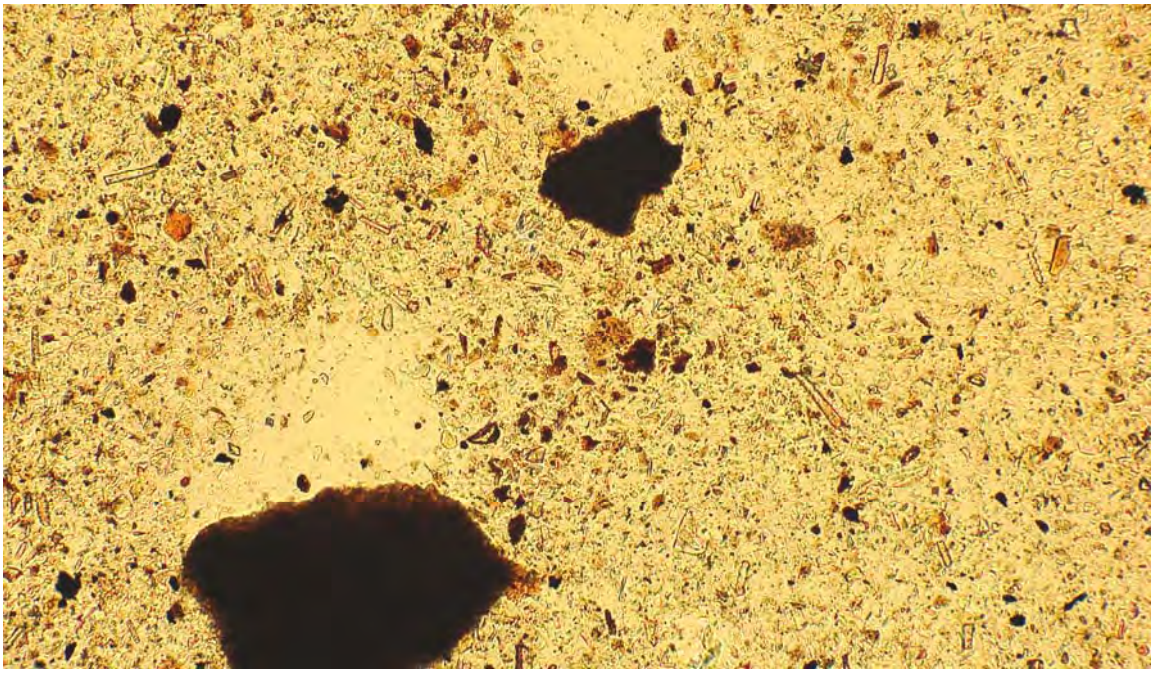




0.2% Libby Amphibole

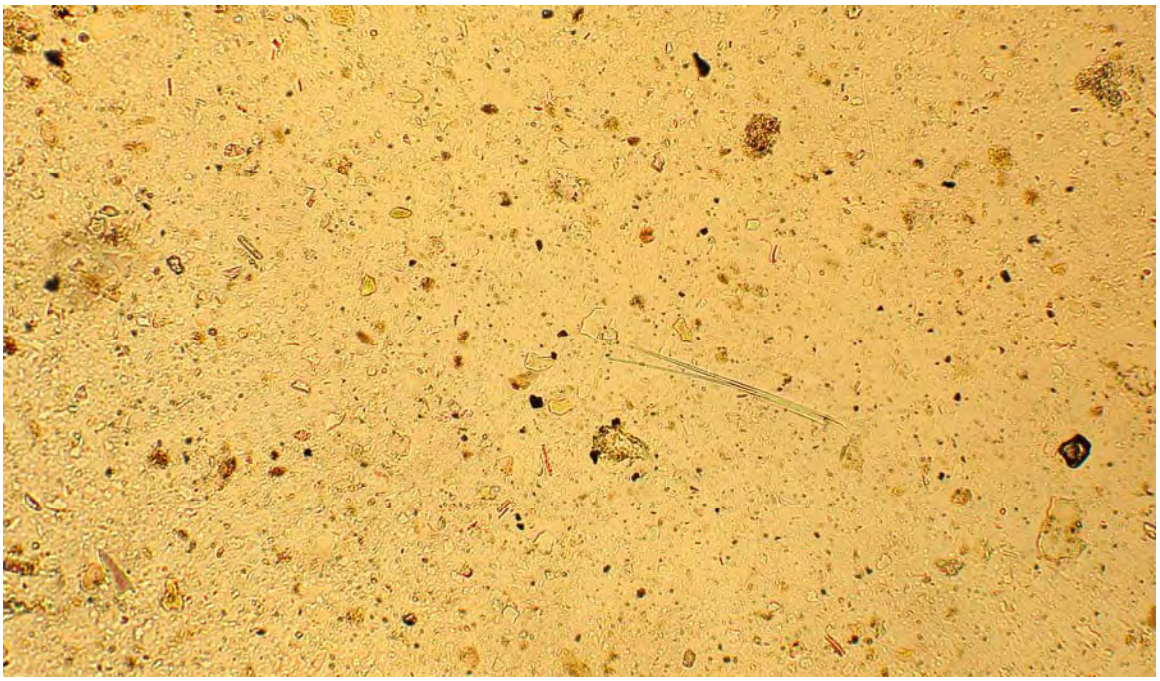
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

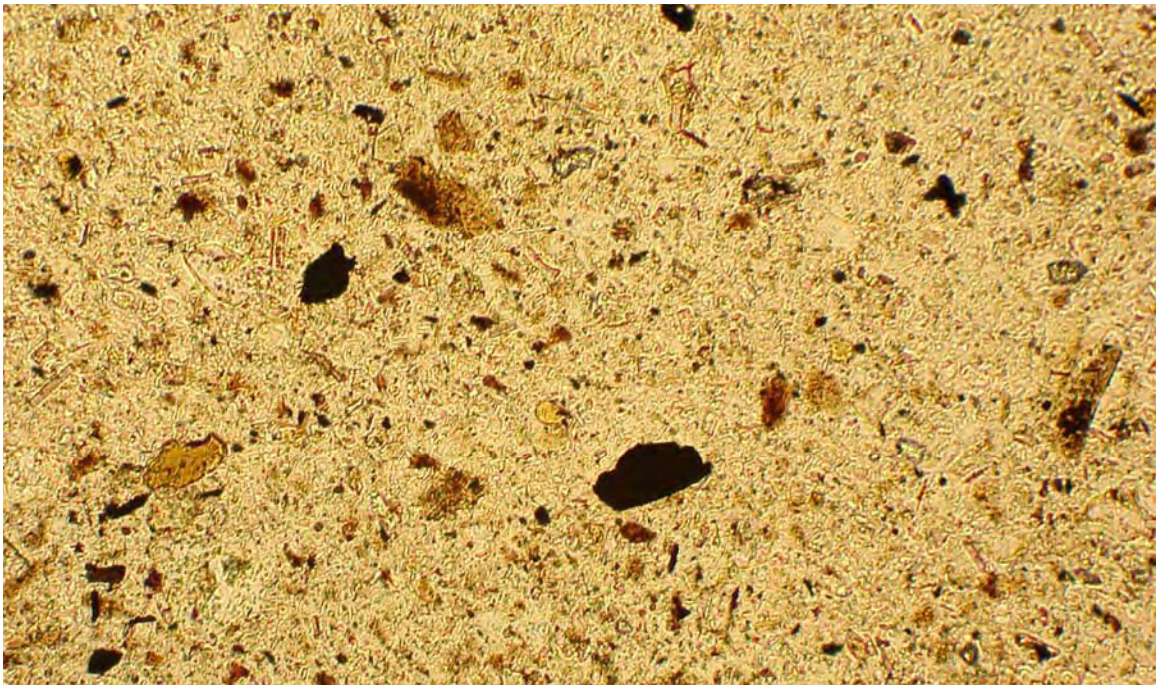




0.2% Libby Amphibole

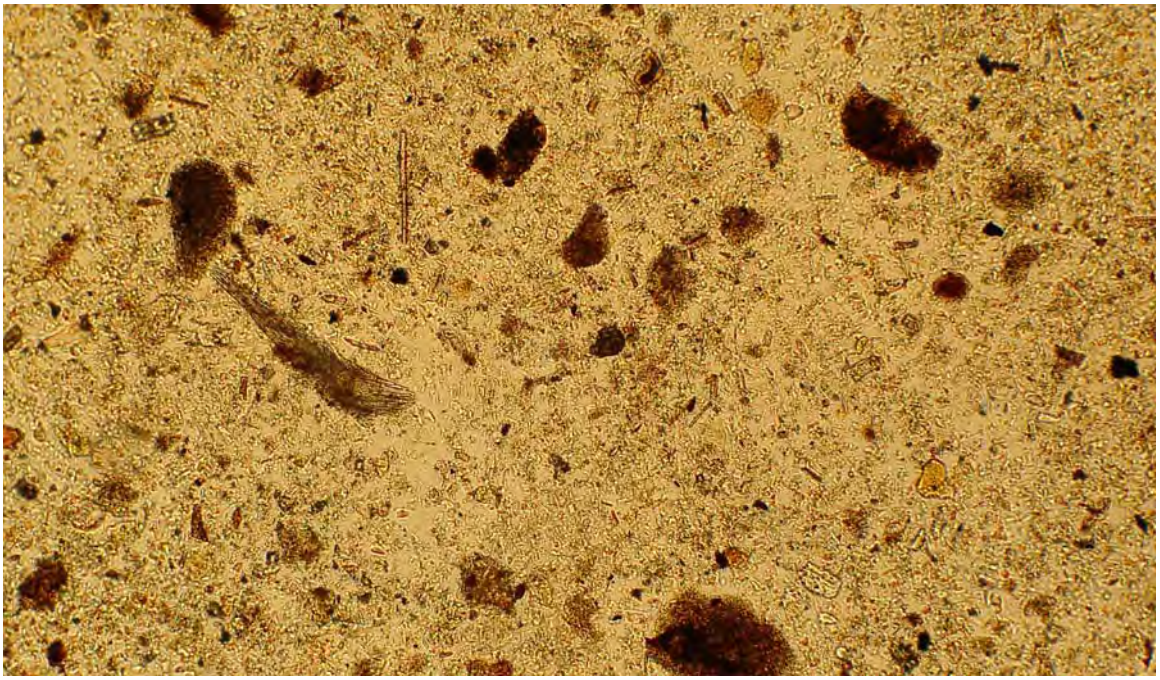
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

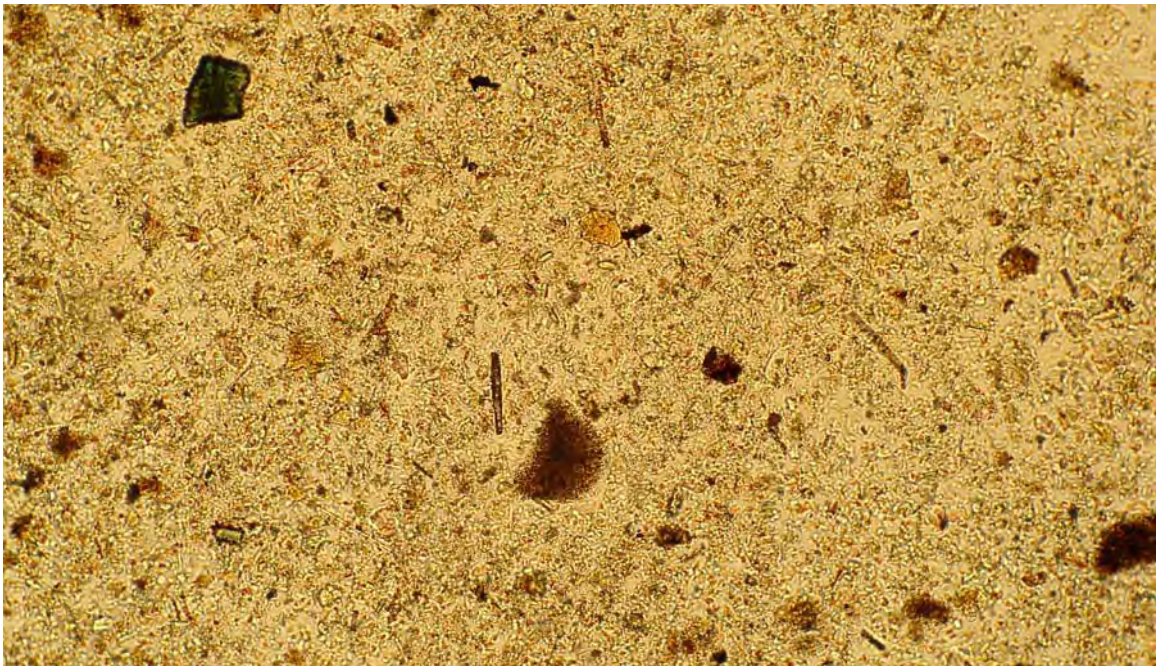




1.0% Libby Amphibole

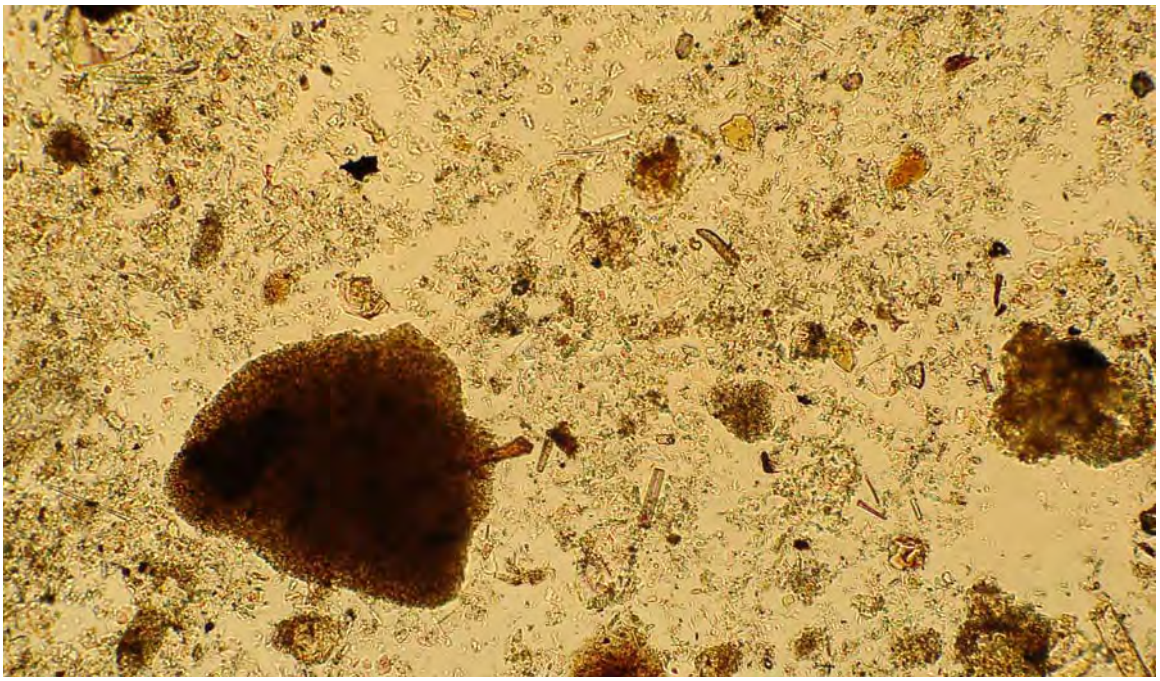
Photomicrographs of representative fields of view of the 1.0% Libby Amphibole by weight Controlled PE Reference Material. All photos taken at 100x, plane light in 1.55 refractive index oil. Width of each picture is approximately 1,500 microns.

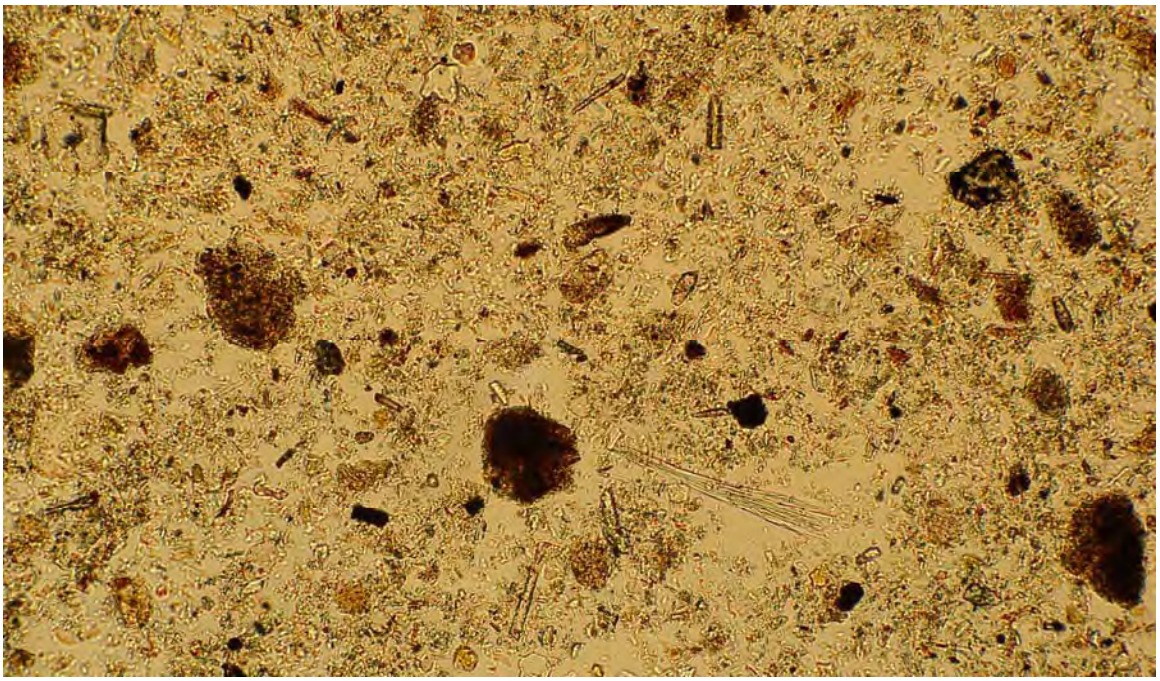




1.0% Libby Amphibole

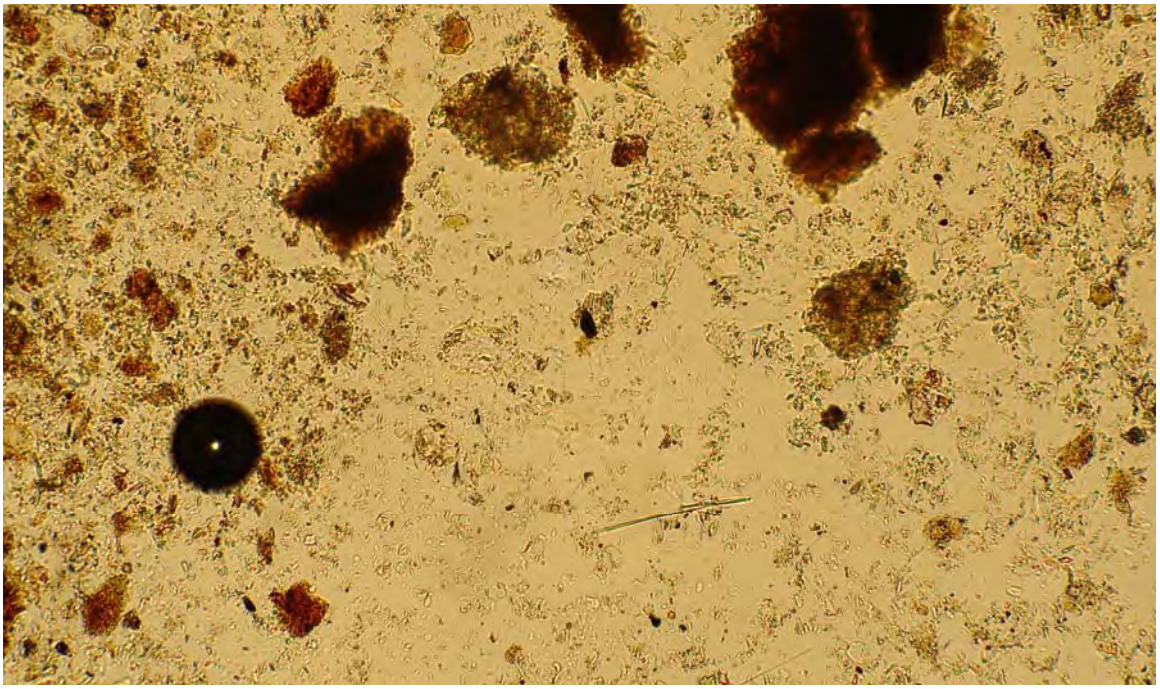
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

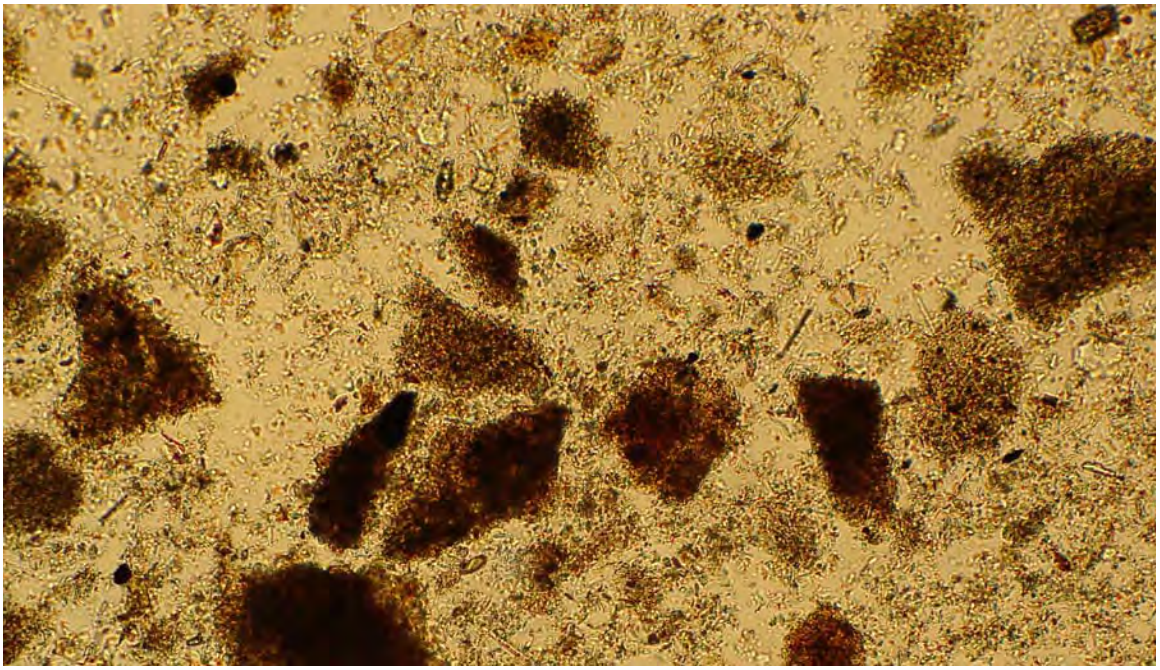




1.0% Libby Amphibole

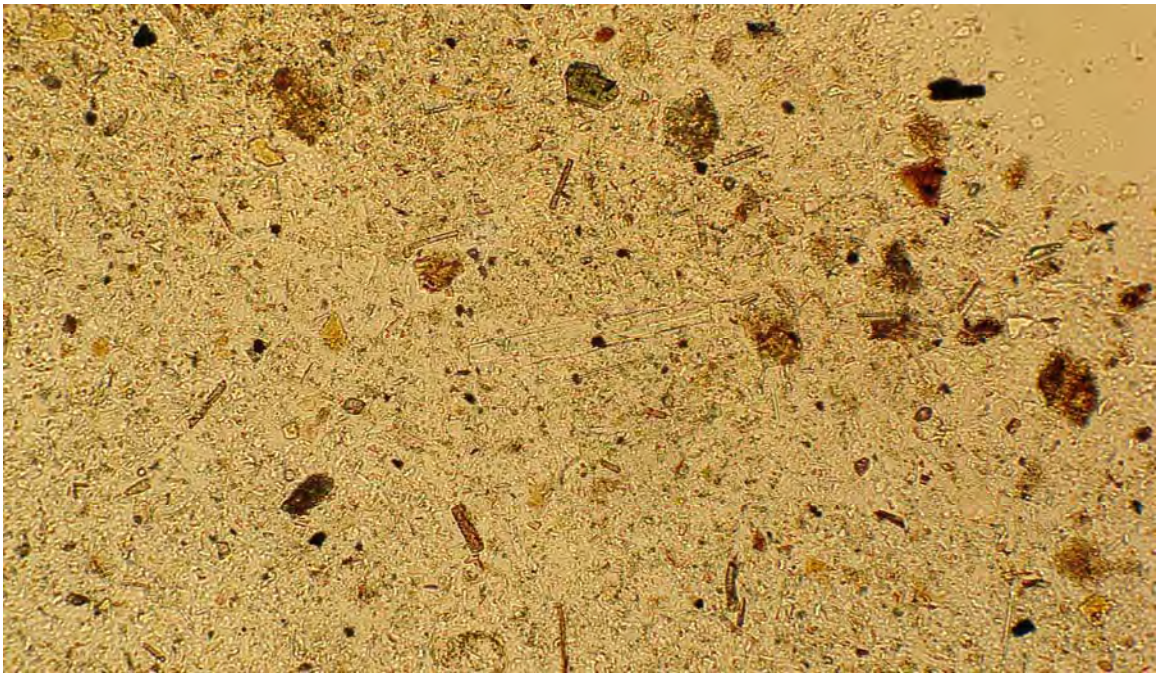
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

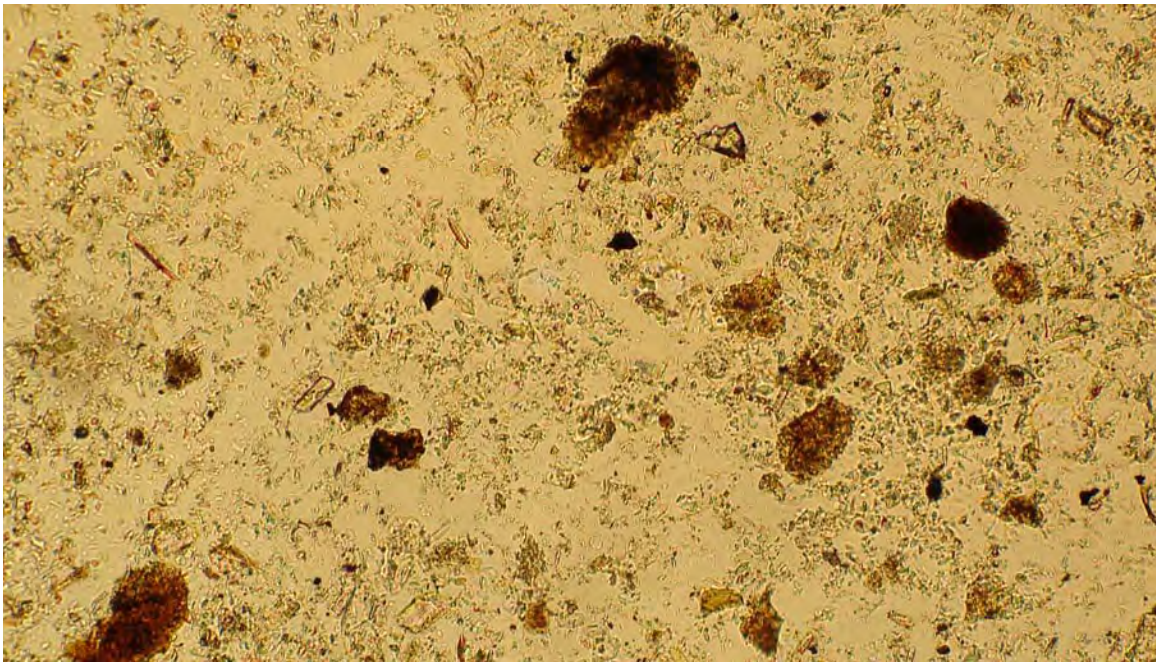




1.0% Libby Amphibole

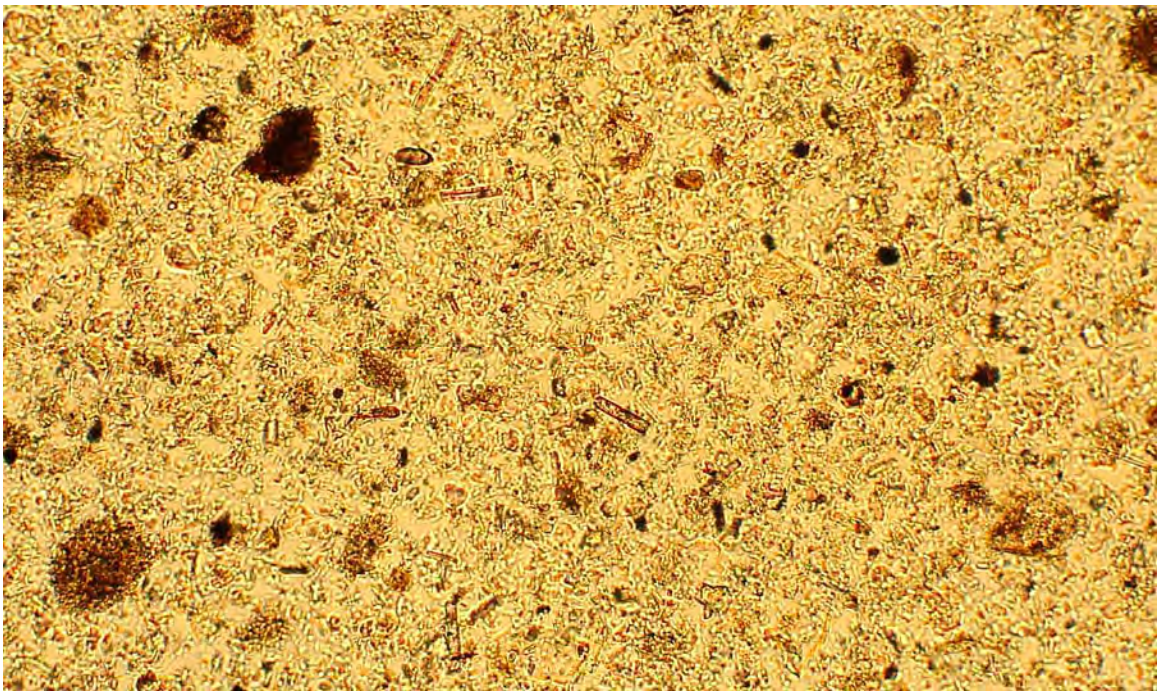
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

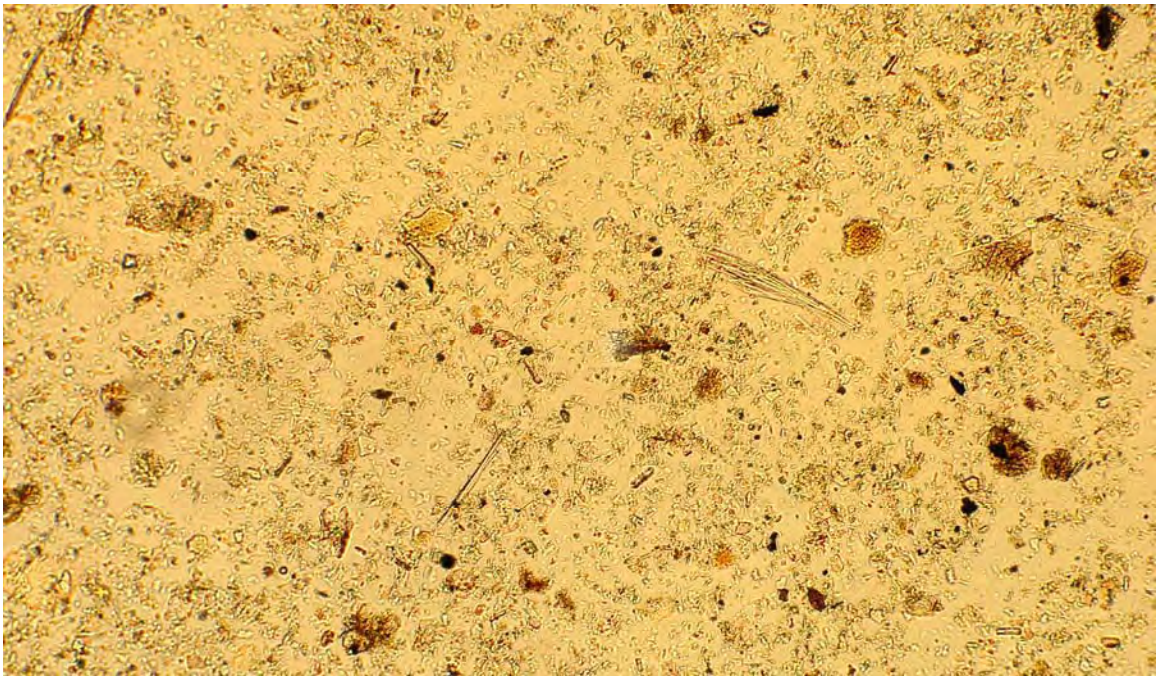




1.0% Libby Amphibole

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

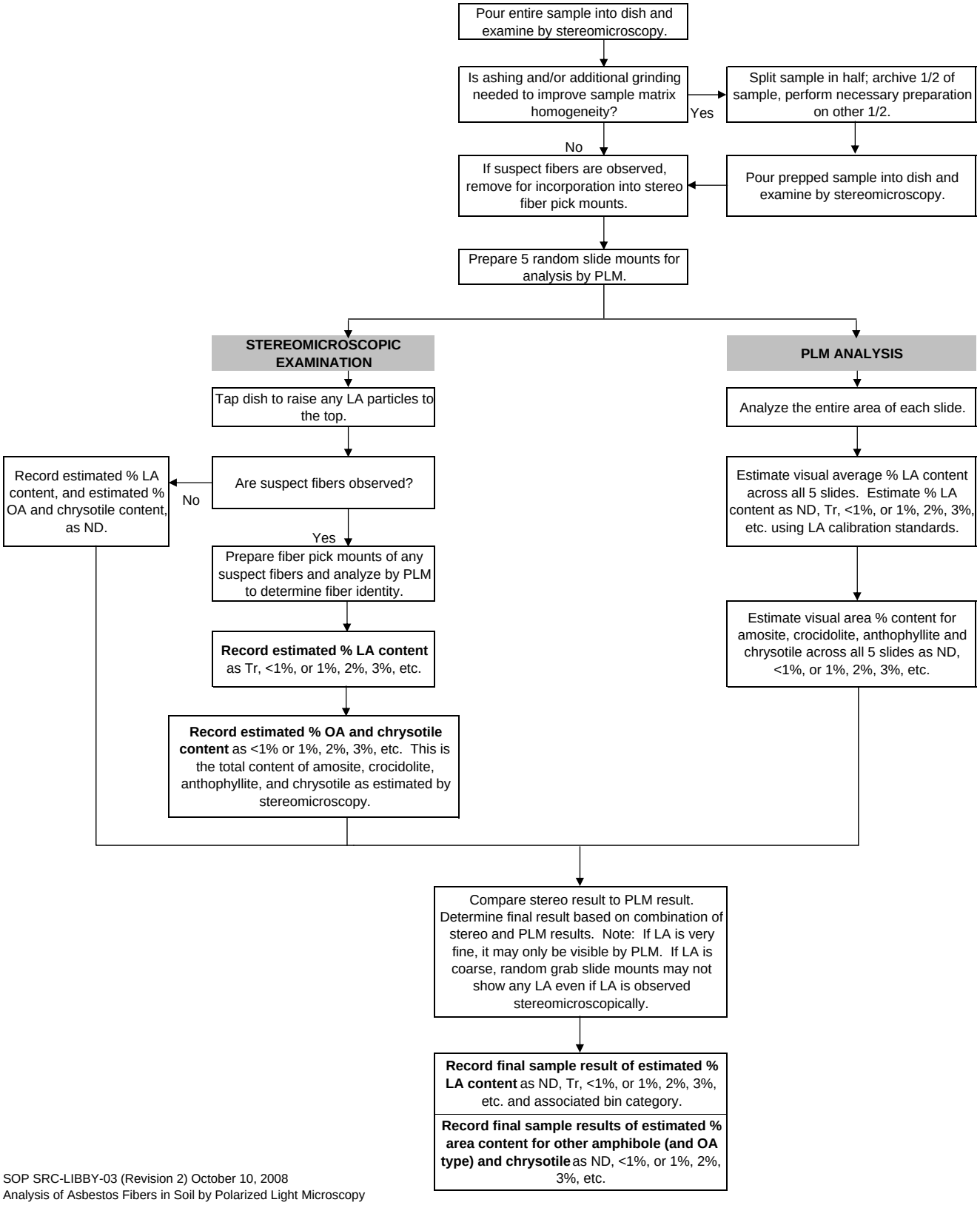




ATTACHMENT 8

Flow Chart for Determining Asbestos Content by Complementary Use of Stereomicroscopic Examination and PLM Visual Estimation

Flow Chart for Determining Asbestos Content by Complementary Use of Stereomicrosopic Examination and PLM Visual Estimation



ANALYSIS OF ASBESTOS FIBERS IN SOIL BY POLARIZED LIGHT MICROSCOPY

SYNOPSIS: A semi-quantitative method for identifying and quantifying asbestos fibers in soil using polarized light microscopy is provided. This method is based on NIOSH Method 9002, EPA Method 600/R-93/116, and CARB Method 435, with project specific modifications intended specifically for application at the Libby Superfund Site. Sampling and plan developers and data users are cautioned to understand how data are generated from this SOP.

APPROVALS:

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Revision	Date	Principal Changes
0	3/3/2003	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 sample preparation and analytical process. Authors: Douglas Kent and Nikki MacDonald, ESAT Region 8

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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 and has been refined to focus testing on Libby Amphibole 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 a preparation process described below. This method is appropriate for the analysis of all types of asbestos fibers (chrysotile and amphiboles), including those that are characteristic of the Libby Asbestos Superfund Site, Libby Amphibole asbestos (LA).

3.0 RESPONSIBILITIES

3.1 It is the responsibility of the laboratory supervisor to ensure that all analyses and quality assurance (QA) 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, QA/QC Coordinator (or equivalent), or Analytical Lead communicates with project managers at the United States Environmental Protection Agency ([EPA]; also referred to as the client), or their designate, any situations where a change from the SOP may be useful and/or required. The laboratory supervisor 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 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), Quality Management Plan (QMP), or an equivalent document(s) that meets all the requirements of the National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150. It is also the responsibility of the laboratory to ensure its testing activities stay in compliance with the requirements of NVLAP Handbook 150 and the regulatory and accrediting agencies that provide oversight of the laboratory's operations and all Libby Asbestos 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 Asbestos Superfund Site is based on the National Institute of Occupational Safety and Health (NIOSH) Method 9002, 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 (particles 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 only the analysis of the fine fractions of soil samples. Coarse fractions of 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 are then prepared of the sample 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 estimated in terms of mass fraction (percent asbestos by weight) based on the use of project-specific reference materials. Samples are re-analyzed or re-prepped and re-analyzed, and prepared standards are analyzed, as part of the quality control (QC) program.

5.0 ACRONYMS

ACM	Asbestos Containing Material
CARB	State of California Air Resources Board
EDD	Electronic Data Deliverable
EDS	Energy Dispersive Spectrometry
EDXA	Energy Dispersive X-ray Analysis
EPA	United States Environmental Protection Agency
HEPA	High Efficiency Particulate Air
LA	Libby Amphibole asbestos
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
PE	Performance Evaluation
PLM	Polarized Light Microscopy
PLM-VE	Visual Area Estimation technique employed by Polarized Light Microscopy
PPE	Personal Protective Equipment
QA	Quality Assurance
QAM	Quality Assurance Manual
QC	Quality Control
QMP	Quality Management Plan
RI	Refractive Index
SEM	Scanning Electron Microscopy

SOP	Standard Operating Procedure
SRM	Standard Reference Material
TEM	Transmission Electron Microscopy
µm	Microns (1,000 µm = 1mm)
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, Chemical Hygiene Plan, 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 each reagent (e.g., 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 distilled 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 QA Program

8.1.1 Each laboratory operates under a 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 Quality Management Plan, or equivalent, in which the laboratory's QA program is detailed. Additional QA/QC requirements specific to the PLM laboratory and the Libby project are described later 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 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 certainty of a measurement provided to the client, the client must be notified and informed of the nature of the departure from the SOP and 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 for the Libby project are expected to be familiar with routine chemical laboratory procedures, principles of optical mineralogy, and proficient in EPA Method 600/R-93/116, NIOSH Method 9002, and CARB Method 435.

9.1.2 Personnel at laboratories with less than one year of experience specific to the Libby Asbestos project 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 Asbestos Superfund Site.

9.2 Before performing any analyses, the 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 The laboratory has all items of equipment (including instrumentation, hardware, software, and reference materials) required for the correct performance of calibrations and tests.

10.2 All equipment is properly maintained and calibrated (as appropriate) prior to use. See Section 12 for further details regarding microscope calibration.

10.3 Following is a general list of the equipment available at the PLM laboratory to perform this SOP:

10.3.1 Polarized Light Microscope, with:

10.3.1.1 Light source and replacement bulbs
10.3.1.2 Binocular observation tube
10.3.1.3 Blue daylight filter
10.3.1.4 Oculars (10X)
10.3.1.5 Objectives: 10X, 20X, and 40X (or similar magnification)
10.3.1.6 10X Dispersion Staining Objective
10.3.1.7 360 degree rotatable and centerable stage
10.3.1.8 Polarizer and analyzer aligned at 90 degrees to one another
10.3.1.9 Bertrand lens (optional)
10.3.1.10 Substage condenser with iris diaphragm
10.3.1.11 Accessory slot for compensator plate
10.3.1.12 First order red (550 nanometer) compensator plate

10.3.1.13 Crosshair reticle
10.3.1.14 Adjustment tools
10.3.2 HEPA-filtered hood, class 1 biohazard hood, or glove box with continuous airflow (negative pressure)
10.3.3 Binocular stereomicroscope, 10-50X magnification (approximate)
10.3.4 Light source for stereomicroscope
10.3.5 Muffle furnace
10.3.6 Analytical balance
10.3.7 SOP-specific Electronic Data Deliverable (EDD), most recent version
10.3.8 Mortars (agate or porcelain)
10.3.9 Pestles (agate or porcelain)
10.3.10 Anemometer
10.3.11 Wet/dry vacuum with HEPA filtration
10.3.12 Decontamination equipment (e.g. baby wipes, wet mop with bucket, etc.)

11.0 STANDARDS, REAGENTS AND SUPPLIES

11.1 High Dispersion RI Liquid from 1.620 to 1.640 (1.625 is a common choice)

11.2 1.550 High Dispersion RI Liquid

11.3 1.680 to 1.700 RI Liquid

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, amosite, 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 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 Asbestos Superfund 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 Asbestos Superfund 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 Data recording sheet or bench sheet (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 Distilled 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 Kimwipes (or other appropriate wiping material)

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, and Controlled PE Reference Materials of LA in soils.

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 greater than 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 equal to or less than 1% are calibrated using the Controlled PE Reference Materials.

12.2 General Maintenance and Calibration of the Polarized Light Microscope

12.2.1 Chrysotile, amosite, crocidolite, and anthophyllite all have the optical property of parallel extinction. Because this is one of the optical properties used to identify these minerals, the polars of the PLM must be aligned north-south (N-S) and east-west (E-W), and the polars must be kept at 90 degrees to each other.

12.2.1.1 A mineral grain's extinction angle cannot be measured accurately if the polars are not correctly aligned.

12.2.2 LA and some non-asbestos minerals (wollastonite, hornblende, etc.) will often display an inclined (or oblique) extinction angle.

12.2.3 The lower polar must be properly aligned E-W so RI's in the parallel and perpendicular directions can be measured correctly.

12.2.4 The polars should be kept at 90 degrees to each other so the field of view in crossed polars is as dark as possible.

12.2.5 The microscope's optics must be kept clean and properly aligned so optimal image quality can be produced.

12.2.6 Check the microscope's alignment each working day prior to use.

12.2.6.1 The microscope must be re-aligned any time it is found to be out of alignment. Follow all the procedures outlined in Sections 12.3

through 12.8 for re-calibrating the microscope.

12.2.7 Each day the microscope is used, record an entry in the microscope's instrument maintenance logbook. Record the date and analyst's initials confirming that all microscope alignment checks were made prior to analysis.

12.2.7.1 An individual instrument maintenance logbook must be kept for each microscope in use at the laboratory.

12.2.7.2 All maintenance activities performed on the microscope must be recorded in the appropriate logbook.

12.2.7.3 Each day the microscope is used to analyze samples, a data entry must be made in the logbook indicating that the microscope was properly calibrated that day prior to use.

12.3 Checking Microscope Alignment

12.3.1 Place a permanently-mounted test 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 of anthophyllite 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 of anthophyllite become completely extinct in either the N-S or E-W direction, pull the analyzer out to make sure the fibers of anthophyllite 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 in view when the microscope stage is rotated.

12.3.4 The light path through the scope must be centered (specifically, the condenser and iris diaphragm must be centered on the optic axis).

12.3.5 The crosshairs should 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 strong cleavage traces between the sheets of mica.

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 anthophyllite or polypropylene 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 the analyzer out 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 shall 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 surfaces of the lenses.

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.

12.9.3 If dust gets inside the microscope, it is necessary to completely disassemble and clean the microscope.

12.9.3.1 The microscope must be re-calibrated after being re-assembled and this must be recorded in the microscope's maintenance logbook.

12.9.3.2 Disassembly of the microscope should only be performed by qualified personnel.

13.0 DETAILED METHOD FOR ASBESTOS TESTING OF SOIL AND SOIL-LIKE MATERIALS

13.1 Stereomicroscopic Examination

13.1.1 All sample preparation activities, including stereomicroscopic examination, slide mounts, etc., must be performed in a HEPA-filtered hood, class 1 biohazard hood, or glove box with continuous airflow (negative pressure).

13.1.2 Due to the sample preparation requirements described in the current revision of SOP ISSI-LIBBY-01, Soil Sample Preparation, samples should never be wet. If the sample is wet, contact EPA or designate.

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 container 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 larger.

13.1.5 With the stereomicroscope, visually examine the entire sample for homogeneity and the presence of any suspect fibers.

13.1.6 If individual fibers suspected of being asbestos are observed, 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 often called “fiber-picks” and are referred to as fiber-picks in this SOP.

13.1.6.1 Each microscope slide must be wiped with lint-free wipes prior to use to avoid contamination.

13.1.6.2 Mount individual fibers in 1.550 RI oil if chrysotile is suspected, 1.620 to 1.640 RI oil if LA or anthophyllite is suspected, or 1.680 to 1.700 RI oil if amosite or crocidolite is suspected.

13.1.6.3 Only one drop of RI liquid is necessary to prepare the 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 sample appearance, 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 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 Even if no fibers are visible, prepare the sample as described in Section 13.3.

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 data sheet in the comments field. Alternatively, attach a separate analytical data sheet (specific to ashing samples) with the necessary measurements, and indicate the attachment in the comments section.

13.3 Preparation of Samples for PLM Visual Area Estimation

13.3.1 Quantitative analysis preparation typically consists of preparing random mounts of a sample. The objective is to produce random mounts of a representative sub-sample from the original sample.

13.3.2 View the sample through the stereomicroscope to determine if it is sufficiently homogenized and all particles are reduced to a small enough size.

13.3.2.1 Soil samples processed according to the current revision of SOP ISSI-LIBBY-01, Soil Sample Preparation, should be ground to a maximum particle size of approximately 250 μm.

13.3.2.2 Additional homogenization of the sample at the laboratory using a mortar and pestle may be required if any remaining inhomogeneities or coarse particles are observed in the sample. When further grinding the sample, care should be taken to not pulverize the LA to a fiber size unidentifiable by PLM techniques. The material in the slide mounts must be coarse enough that fibers of LA can still be identified by PLM and still be as representative as possible of the sample as a whole.

13.3.3 Oil immersion mounts of randomly selected sub-samples of the homogenized material are prepared in RI liquids for PLM analysis.

13.3.3.1 Prepare a minimum of five random mount slides for each sample.

13.3.3.2 Each microscope slide must be wiped clean with an appropriate wipe prior to use in order to avoid contamination.

13.3.3.3 Place one to two drops of the appropriate RI liquid onto each slide.

13.3.3.3.1 Prepare at least two slides with a RI liquid in the range of 1.620 to 1.640 for easier measurement of the optical properties of LA. Generally, 1.625 RI liquid is used for LA.

13.3.3.3.2 The refractive indices of the oils used for the remaining slides is left to the analyst's discretion based upon the suspected mineralogy present in the sample material.

13.3.3.4 Use a spatula, the curved edge of a scalpel blade, or other similar utensil to collect randomly selected sub-samples of the homogenized

sample material, and place this into the RI liquid on the slides.

13.3.3.5 With the utensil, gently stir the sample material in the RI liquid to produce a homogeneous mixture.

13.3.3.6 Cover each mixture of RI liquid and sample material with a glass cover slip.

13.3.3.7 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.

13.3.3.7.1 Use this action to spread the mixture of RI liquid and sample material over the approximate area of the cover slip.

13.3.3.7.2 The material under the cover slip should be spread out evenly with no or very few overlapping particles.

13.3.3.8 Wipe any loose sample material or excess RI liquid from the slide with lint-free wipes.

13.3.3.9 The prepared slide can now be safely removed from the hood for analysis by PLM.

13.4 Supplemental Stereomicroscopic Evaluation

13.4.1 Following random slide mount preparation, it may be useful agitate or tap the sample container to cause the particulate to settle and the amphibole fibers to sort to the surface.

13.4.1.1 Re-examine the sample using the stereomicroscope, and repeat procedures 13.1.6, above.

13.4.1.2 This “tapping” method 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.

13.4.1.3 The representative sub-sample material used for preparing random slide mounts must remain homogeneous.

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 come into contact with the sample, with distilled water and paper towels.

13.4.2.2 Dispose of gloves after 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 oil or to oil 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 Libby soil samples 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 Morphology
- 13.5.4.2 Color and pleochroism (if pleochroism is present)
- 13.5.4.3 Refractive indices, both alpha and gamma
- 13.5.4.4 Birefringence
- 13.5.4.5 Extinction characteristics
- 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 have been 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 Superfund Site. Also included are the minerals magnesio-arfvedsonite and magnesio-riebeckite, which are known to occur at the Libby Asbestos Superfund Site in smaller quantities.
OA	Other amphibole asbestos	Regulated amphibole asbestos (amosite, crocidolite, and anthophyllite) that are not thought to occur in significant amounts at the mine in Libby.
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 serpentine occurs in an asbestiform morhology, 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 morphologies.
- 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) morphology, it is classified as non-asbestiform serpentine (antigorite if it is platy or lizardite if it occurs as a massive, fine-grained matrix) and not as asbestos (chrysotile).

13.5.7.5 If serpentine is observed and has a fibrous morphology, it is classified as chrysotile asbestos.

13.5.7.6 The morphology of chrysotile is fibrous and sometimes silky.

13.5.7.7 The fibers are flexible. Chrysotile sometimes occurs as tangled mats of many fibers.

13.5.7.8 Chrysotile can only be seen in PLM as chrysotile bundles; the individual fibrils that make up a chrysotile bundle are beyond the resolution of all light microscopy.

13.5.7.9 Bundles of chrysotile are often splayed.

13.5.7.10 Kinked chevron-style folds are sometimes seen in chrysotile.

13.5.7.11 Chrysotile is usually colorless in PLM, although it sometimes shows a slight golden, yellow, or pale golden-green color in PLM.

13.5.7.12 Chrysotile that has been exposed to very high temperatures is distinctly brown under plain light.

13.5.7.13 Chrysotile is never pleochroic.

13.5.7.14 Small particles of opaque magnetite can sometimes be seen in large, intact bundles of chrysotile.

13.5.7.15 The range for the lower RI (alpha, or α) for chrysotile is 1.545 to 1.553 as reported in the certificate for NIST SRM 1866b, although the range for chrysotile encountered in field samples may be somewhat wider.

13.5.7.16 The range for the higher RI (gamma, or γ) for chrysotile is 1.552 to 1.560 as reported in the certificate for NIST SRM 1866b, although the range for chrysotile encountered in field samples may be somewhat wider.

13.5.7.17 Exposure to high heat and dehydration of the crystal lattice will increase the refractive indices of chrysotile.

13.5.7.18 The birefringence (expressed numerically as δ, the difference between α and γ) of chrysotile is low, usually around 0.008. In practice, this means that most chrysotile bundles of fine to medium size observed in samples will have low first order gray to medium gray interference colors under crossed polars. Larger, thicker fibers can show first order white to yellow interference colors; higher colors may be seen in the thickest bundles.

13.5.7.19 Chrysotile is most easily visible in plane light in the higher RI liquids, such as 1.62 or 1.68. However, measurement of the refractive indices of chrysotile should be done with the fibers mounted in the 1.550 oil.

13.5.7.20 Chrysotile is almost always length slow (positive sign of elongation), although length fast chrysotile has been observed on very rare occasions.

13.5.7.21 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) morphology. Bundles of amosite are composed of many lesser needles of amosite. Needles of amosite are often straight and only somewhat flexible.

13.5.8.3 Amosite is usually colorless, green, brown, or greenish-brown in plane light. Heated amosite is brown to dark brown and can be nearly opaque. Amosite is sometimes weakly pleochroic.

13.5.8.4 The range for the lower RI (α) for amosite is 1.675 to 1.681 as reported in the certificate for NIST SRM 1866b, although the range for amosite encountered in field samples may be somewhat wider.

13.5.8.5 The range for the higher RI (γ) for amosite is 1.697 to 1.704 as reported in the certificate for NIST SRM 1866b, although the range for amosite encountered in field samples may be somewhat wider.

13.5.8.6 Exposure to high heat and dehydration of the crystal lattice will increase the RI's of amosite.

13.5.8.7 The birefringence of amosite is moderate, usually about 0.020. Most fibers observed will have first order white to yellow interference colors under crossed polars; although, higher colors (first order magenta to second order or sometimes even higher) can be seen in the thicker bundles.

13.5.8.8 RI measurements should be done with the fibers mounted in 1.680 to 1.700 RI oil.

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.

13.5.9.2 Crocidolite has an acicular morphology very similar to that of amosite. The fibers are only somewhat flexible.

13.5.9.3 Crocidolite is distinctly blue or blue-green in plane light and is pleochroic.

13.5.9.4 Normally, the range for the lower RI (α) for crocidolite is 1.680 to 1.698 (EPA, 1993).

13.5.9.5 Normally, the range for the higher RI (γ) for crocidolite is 1.685 to 1.706 (EPA, 1993).

13.5.9.6 The strong color of crocidolite makes measurement of the refractive indices very difficult. For this reason, select finer fibers of crocidolite, which have less color, when measuring refractive indices.

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 oil.

13.5.9.9 Because crocidolite is length fast, the lower RI (α) should be measured with the fiber oriented in the E-W direction (parallel to the lower polar), and the higher RI (γ) should be measured with the fiber oriented in the perpendicular (N-S) direction.

13.5.9.10 Even though riebeckite is a monoclinic mineral, the extremely narrow fibers that form bundles of crocidolite cause crocidolite to have parallel extinction.

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

13.5.10.1 Anthophyllite occurs as straight to slightly curved fibers or fiber bundles. The morphology of anthophyllite is lamellar to acicular.

13.5.10.2 Anthophyllite is a rare type of asbestos used in construction materials.

13.5.10.3 Anthophyllite is colorless to pale brown in plane light. It is sometimes weakly pleochroic.

13.5.10.4 The range for the lower RI (α) for anthophyllite is 1.593 to 1.694 (Deer et al., 1997). The commercial-grade anthophyllite in SRM 1867a has an α of 1.615.

13.5.10.5 The range for the higher RI (γ) for anthophyllite is 1.613 to 1.722 (Deer et al., 1997). The commercial-grade anthophyllite in SRM 1867a has a γ of 1.636.

13.5.10.6 The birefringence of anthophyllite is moderate, usually about 0.020.

13.5.10.7 Generally, RI measurements on anthophyllite should be done with the fibers mounted in 1.620 to 1.640 oil.

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, $Na_2Mg_3Fe^{3+}{}_2Si_8O_{22}(OH)_2$.

13.5.11.2 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 a group of minerals, such as tremolite, actinolite, winchite, richterite, magnesio-riebeckite, and magnesio-arfvedsonite. The optical properties for each individual mineral are provided below and in Attachment 4. As seen, 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. Rather, if the sample exhibits the optical properties of a mineral listed in this section, the specific optical properties (such as refractive indices, birefringence, extinction angle, and sign of elongation) shall be noted on the analytical data sheet and EDD, and the mineral identified as LA.

13.5.11.3 The morphology 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 morphologies that may be encountered during PLM analysis.

13.5.11.6 Laboratories should use the Controlled Libby Amphibole Asbestos 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 designate if they do not have these reference materials.

13.5.11.7 Color of LA in plane light is highly varied. Tremolite is usually colorless in plane light. 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 in plane light is yellowish green, brownish green, or grey-blue (Deer et al, 1997). Magnesio-riebeckite in plane light is blue, grey-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 0.015 to 0.02.

13.5.11.9 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 approximately 30 degrees or even higher for richterite.

13.5.11.10 RI measurements on LA should be done with the fibers mounted in 1.620 to 1.640 RI oil (1.625 is a commonly-used choice).

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, Optical Properties of Fibrous Amphiboles, for additional information on the optical properties of LA used in LA identification.

13.6 Refractometry

13.6.1 Calibration of Refractive Index Liquids

13.6.1.1 Accurate measurement of a mineral's refractive indices 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 an oil immersion mount of the appropriate certified precision optical glass in the oil 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 (see page 7 of Attachment 3).

13.6.1.4 Next determine λ_0. This is the wavelength at which the RI of the oil is equal to the RI of the certified precision optical glass. Observe the

central stop dispersion staining color shown by the glass, and consult the dispersion staining color chart (McCrone, 1987). If the glass particles show a range of dispersion staining colors, use the most predominant color when determining λ_0. Record the predominant dispersion staining color and corresponding λ_0 on the worksheet.

13.6.1.5 Consult the Excel spreadsheet developed by Shu-Chun Su, Ph.D., "Create_RI_Liquid_Calibration_Conversion_Tables.xls", for the appropriate conversion table (see Attachment 2). These tables are used to convert λ_0 and t into n_d^{25}, which is the calibrated RI of the oil at a wavelength of 589 nm and a temperature of 25°C. Determine the value of n_d^{25} from the appropriate table for the known values λ_0 and t.

13.6.1.6 Additional conversion tables for oils not included in the spreadsheet can be generated by entering the dispersion coefficients and values of n_d of the oil and the glass, and the value of dn/dt (change of RI with temperature) of the oil into the first sheet of the workbook. All of these values are clearly provided by the manufacturer of the glass and oil.

13.6.1.7 Record the value of n_d^{25} on the worksheet. This is the calibrated RI of the oil 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 actual calibrated RI of the oil and the original RI of the oil is greater than 0.004, then the oil may not be used for analysis of samples.

13.6.1.10 Repeat the above steps for each oil in routine use.

13.6.2 Measurement of refractive indices (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 oil 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 oil. However, this method does not require a specific mineral-oil chart to be developed before it is used. For this reason the Becke Line method can be used to measure the RI's of other materials besides LA and regulated asbestos minerals.

13.6.3 Measurement of Refractive Indices by the Dispersion Staining Method

13.6.3.1 Mount the fibers in the appropriate oil (1.550 for fibers suspected of being chrysotile, 1.620 to 1.640 oil for fibers suspected of being LA or anthophyllite, or 1.680 to 1.700 oil for fibers suspected of being amosite or crocidolite).

13.6.3.2 In order for the correct dispersion staining colors to be displayed, a clean surface of the mineral must be in direct contact with the RI liquid.

13.6.3.3 If 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.

13.6.3.4 Examine the slide in plane light using the 10X dispersion staining objective. The dispersion staining objective and its central stop should be centered.

13.6.3.5 Stop down the condenser iris diaphragm until dispersion colors are observed.

13.6.3.6 Read the thermometer to find ambient temperature of the laboratory's air to the nearest 2°C.

13.6.3.7 To measure α, orient the fiber E-W (parallel to the lower polar) if the fiber is suspected of being crocidolite, or N-S if the fiber is suspected of being chrysotile, amosite, or anthophyllite. LA shows biaxial optics and requires a more detailed treatment, described below in Section 13.6.5.

13.6.3.8 Next, observe the dispersion staining color that is displayed.

13.6.3.9 Light of a wavelength higher or lower than the matching wavelength (given the symbol λ_0, where the RI of the oil matches the RI of the mineral) is refracted around the central stop and passes through.

13.6.3.10 Light of a wavelength equal or approximately equal to the matching wavelength is blocked.

13.6.3.11 The observed color is the summation of the remaining light.

13.6.3.12 Consult the dispersion staining color chart (McCrone, 1987) and find the matching wavelength (λ_0) that corresponds to the observed color.

13.6.3.13 When measuring α and a range of dispersion staining colors is displayed, choose the color that produces the lowest RI, i.e., the color that corresponds to the longest λ_0.

13.6.3.14 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).

13.6.3.15 For the appropriate RI oil and mineral combination, find the column for the laboratory's temperature and row for λ_0; record the corresponding value of RI.

13.6.3.16 To measure γ, rotate the stage 90 degrees.

13.6.3.17 The fiber should now be perpendicular to the lower polar (N-S) if the fiber is suspected of being crocidolite, or parallel to the lower polar (E-W) if the fiber is suspected of being chrysotile, amosite, or anthophyllite. Refer to Section 13.6.5 for orienting fibers of LA when measuring γ.

13.6.3.18 Observe the dispersion staining colors and find the corresponding λ_0. When measuring γ, choose the color that produces the highest RI, i.e., the color that corresponds to the shortest λ_0.

13.6.3.19 Consult the appropriate chart for the asbestos type and oil being used; record the value of RI for the temperature and λ_0.

Note: There are two charts for each mineral and oil combination - one for α and one for γ. Be sure to use the appropriate chart when measuring α or γ.

13.6.4 Measurement of Refractive Indices 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 to or the same as the RI of the oil. Becke line colors are usually best observed using high magnification (200X to 500X).

13.6.4.2 To measure refractive indices using the Becke line method, mount the fibers in an oil whose RI is close to that of the mineral.

13.6.4.3 Observe the Becke line colors with the fiber oriented in the parallel and perpendicular directions.

13.6.4.4 As a rule, the Becke line moves into whichever medium (the grain or the oil) that 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 oil for some wavelengths of light in the visible spectrum and when the RI of the grain is less than the oil 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 oil, the RI of the grain is less than that of the oil.

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 oil, the RI of the grain is higher than that of the oil.

13.6.4.8 A perfect match occurs when n_d (the RI for the wavelength of sodium light, 589 nm) is the same for both the grain and the oil. When the n_d of mineral matches the n_d of the oil, an orange Becke line with just a touch of red moves into the grain and a bluish line moves into the oil when the stage is lowered.

13.6.4.9 If a perfect match cannot be obtained, mount the mineral in two oils 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 (1999) may be used to approximate the size of the difference between the RI of the oil 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.

13.6.5.2 Even though chrysotile is a monoclinic mineral, it does not show biaxial optics because of the scrolled nature of the fibers.

13.6.5.3 When an asbestos fiber shows biaxial optics, it is easy to measure a RI called α' that is between true α and beta (β) when attempting to measure α.

13.6.5.4 True α can only be observed when a crystal is oriented in exactly the correct position.

13.6.5.5 For the monoclinic minerals that display biaxial optics (LA), the crystals need to be oriented so the X and Z axes of the biaxial indicratix corresponding to the directions of α and γ are parallel to the lower polar when measuring these indices, and they are not necessarily oriented with the crystallographic axes. As a general rule, when these fibers show inclined extinction, select the fibers that show the highest extinction angle when measuring α and γ. RI measurements should be made on a fiber where the plane of X and Z in the biaxial indicatrix lies as close to parallel to the plane of the microscope stage as possible, such that the microscopist is looking directly down Y, which corresponds to the β RI (and also the b crystallographic axis for tremolite, actinolite, winchite, richterite, and magnesio-arfvedsonite). Fibers at or close to this orientation will tend

to show the highest extinction angle.

13.6.5.6 Next, when measuring α for LA, orient the fiber approximately N-S, at the 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.

13.6.5.7 Repeat this for a number of crystals. If the crystals show different Becke line colors or dispersion staining colors, measure α for the crystals that display the lowest RI.

13.6.5.8 Similarly, it is easy to measure a RI called γ' that is between β and true γ when attempting to measure γ. True γ can only be observed when a crystal is oriented in exactly the correct position.

13.6.5.9 Orient a fiber of LA approximately E-W, so that the fiber is extinct under crossed polars, when measuring γ. 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 crystals. If the crystals show different Becke line colors or dispersion staining colors, measure γ for those that display the highest RI.

13.6.5.10 Biaxial Optics of Anthophyllite

13.6.5.10.1 When measuring α (the lower RI) for anthophyllite, the fiber should be oriented in the perpendicular (N-S) direction. When fibers of anthophyllite are oriented in the N-S position, they can show either α or β, or anywhere in between, depending on their orientation. It is therefore necessary to examine a number of fibers oriented in the N-S position to find true α (α will be shown for the fibers that show the lowest RI).

13.6.5.10.2 When measuring γ (the higher RI) for anthophyllite, the fiber should be oriented in the parallel (E-W) position. Fibers of anthophyllite lying flat on the slide will always show γ and not γ', because the c-axis of the fiber will lie within the plane of the slide.

13.7 Quantification of Asbestos Content

13.7.1 General

13.7.1.1 Asbestos is reported as mass fraction percent for LA and is reported as area fraction percent for chrysotile, amosite, crocidolite, and anthophyllite.

13.7.1.2 Asbestos must be positively identified, and its optical properties measured and recorded, before asbestos 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 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 phases 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 calibrated visual estimate. Making accurate calibrated visual estimates is a skill that must be learned and analysts generally improve over time.

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 fraction of asbestos as a percentage of the total material present over many fields of view. Area fraction estimation may be difficult, especially at low concentration values and because the desired output for LA is an estimate of mass fraction (percent asbestos by weight). As a result, all visual estimates of LA content will be performed using a set of site-specific reference materials (calibration standards) 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 analysis using the same approach as for field samples.

13.7.3.2 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 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.

13.7.3.3 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.

13.7.3.4 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.

[1] The nominal mass fraction of the reference materials (calibration standards) is based on the gravimetric fraction of the material that is soil and the amount that is spiking material, adjusted for the fraction of the spiking material that is LA. For example, if the spiking material were estimated to contain 85% LA by mass, then the 1.0% calibration standard would contain 1.18 grams of spiking material (1.00 grams of LA) per 100 grams of calibration standard. Because the estimate of LA content of the spiking material is approximate, the true concentration of a calibration material may not be precisely equal to the nominal value.

13.7.3.5 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.

13.7.3.6 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).

13.7.3.7 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 cut-offs" that place LA concentrations of each sample into one of four categories (see Section 13.8.5, below).

13.7.3.8 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 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 13.2 of this SOP, 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. Stereomicroscopic examination is often an iterative process used in conjunction with analysis by PLM. Refer to Attachment 8 for a flow diagram 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 counted as LA and contributes to the bin category (described in Table 13.2), regardless of its morphology type or aspect ratio. This includes prismatic LA, as well as more fibrous varieties, such as bundles with fibers crossing at various angles giving the bundle a “matted” appearance. Refer to Attachment 5 for examples of a wide range of LA morphologies. Also refer to Attachment 6 for photomicrographs of representative examples of LA morphologies as imaged by the United States Geological Survey (USGS) by Scanning Electron Microscopy (SEM).

13.7.5.2 Using the two 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 (wt.%)	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 was 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 mass percent is estimated quantitatively.

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 detected in each 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 slides 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 slides 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 without an entry in the Qualifier column** is used for all samples in which LA is observed either by stereomicroscopy or by PLM in slides 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 fraction as ND if these analytes are not detected, "<1" if these analytes were detected but at a concentration of less than 1% by area, or to the nearest whole percentage (1%, 2%, 3%, etc.) if these analytes were 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 morphologies. Commonly encountered non-asbestos fibers are listed below.

13.8.3 Talc $Mg_3Si_4O_{10}(OH)_2$

13.8.3.1 Talc usually occurs in a platy or fibrous morphology that looks similar to that of chrysotile.

13.8.3.2 Talc has a higher birefringence than chrysotile.

13.8.3.3 The birefringence of talc is 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.

13.8.3.4 Talc has higher refractive indices (α = 1.54 to 1.56, γ = 1.57 to 1.60) than chrysotile.

13.8.3.5 Talc's refractive indices are less than those of tremolite, actinolite, or anthophyllite.

13.8.4 Wollastonite $CaSiO_3$

13.8.4.1 Wollastonite is one of the pyroxenoid minerals and has a characteristically bladed or prismatic morphology.

13.8.4.2 Wollastonite is colorless in plane light.

13.8.4.3 The refractive indices of wollastonite are very close to that of tremolite. However, wollastonite has a lower birefringence (difference between α and γ = 0.013 to 0.017) than tremolite.

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 Crystals of wollastonite can be spun about their long axis until they change from length slow to length fast or vice versa. Crystals of tremolite will always remain consistently length slow regardless of their optical orientation.

13.8.4.6 One way to spin a wollastonite grain about its long axis is to 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 crystals 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.

13.8.5.2 Kyanite usually has a bladed or columnar morphology.

13.8.5.3 Kyanite is colorless to light blue in plane light. Its blue color is much more subdued than that of crocidolite.

13.8.5.4 Kyanite has positive relief in 1.680 oil. Its refractive indices are higher than those of crocidolite or amosite (for kyanite, α = 1.710 to 1.718, γ = 1.724 to 1.734).

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 amphiboles, often found in soils in areas where certain types of igneous and metamorphic rocks are found. Hornblende is often found in soil samples from the Libby area.

13.8.6.2 Edenite, $NaCa_2(Mg,Fe^{2+})_5Si_7AlO_{22}(OH)_2$, is an amphibole that may be present at the mine at Libby (Meeker et al. 2003). Edenite is part of the hornblende group, and for this reason, for the purposes of this SOP, should not be classified as LA if it is encountered in a field sample.

13.8.6.3 Hornblende generally has slender prismatic to bladed crystals. The traces of cleavage planes are usually visible within of the crystals.

13.8.6.4 Hornblende does not occur in a highly fibrous morphology like LA often does.

13.8.6.5 Hornblende is distinctly colored and pleochroic. Hornblende is usually green, yellow-green, brown, green-brown, or blue-green in plane light.

13.8.6.6 Refractive indices vary with composition, but usually α = 1.645 to 1.665 and γ = 1.660 to 1.690. This is higher than LA.

13.8.6.7 Birefringence is moderate.

13.8.6.8 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 mentioned here because they are among the most common pyroxenes, but analysts should be aware that there are others.

13.8.7.2 Calcic clinopyroxene can be found in soils from areas where certain types of igneous and metamorphic rocks occur and has been found in field samples from the Libby area.

13.8.7.3 The morphology of calcic clinopyroxene is usually prismatic to columnar. As a group, the pyroxenes tend to form less slender, elongated crystals than the amphiboles. Traces of cleavage planes are usually visible within crystals of the pyroxenes.

13.8.7.4 Augite is colorless, pale green, greenish-brown, pale brown, or gray in plane light. Diopside is colorless, but as iron content increases through the diopside-hedenbergite, the mineral develops a green color.

13.8.7.5 Calcic clinopyroxene can be weakly pleochroic.

13.8.7.6 Calcic clinopyroxene has high refractive indices (α = 1.66 to 1.75, γ = 1.69 to 1.77). The pyroxenes as a group tend to have high refractive indices.

13.8.7.7 Birefringence is moderate, as with the majority of other pyroxenes.

13.8.7.8 Calcic clinopyroxene can have a very high extinction angle, up to 48 degrees.

13.8.7.9 Calcic clinopyroxene is generally length slow, but the sign of elongation becomes ambiguous in crystals showing a very high extinction angle.

13.8.8 Fiberglass (Amorphous Silica, SiO_2)

13.8.8.1 Fiberglass is almost always isotropic (appears black at all orientations under crossed polars).

13.8.8.2 Some fiberglass that is coated with other materials, or fiberglass that has been de-vitrified (partial re-crystallization of amorphous silica) due to prolonged exposure to very high temperatures, may show some slight interference colors under crossed polars.

13.8.8.3 The morphology of fiberglass is usually straight, solid, cylindrical tubes. Usually the diameter of the tube varies little along the length of the fiber.

13.8.8.4 Most fiberglass is colorless under plane light. However, the addition of impurities can impart various colors to fiberglass. Some can be yellow, dark brown, or dark green.

13.8.8.5 The RI of fiberglass varies considerably depending on the glass's composition (i.e. the addition of impurities, such as aluminum or iron). However, the RI of most types of fiberglass is close to 1.6.

13.8.9 Cellulose

13.8.9.1 Cellulose often has the morphology of ribbons that are wider than they are thick. The interiors of cellulose fibers often show a cellular or structured network.

13.8.9.2 Cellulose can be straight, curved, kinked, or crooked.

13.8.9.3 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.

13.8.9.4 Cellulose displays undulatory (incomplete) extinction.

13.8.9.5 Cellulose usually has a higher birefringence than chrysotile.

13.8.9.6 Fibers of cellulose will often show first order white or yellow or higher interference colors under crossed polars.

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. This diatomaceous earth is mined and is used in a variety of construction materials.

13.8.10.2 Not all diatoms are fibrous, but many are.

13.8.10.3 Fibrous diatoms generally have the morphology of cylindrical tubes, sometimes with tapered ends.

13.8.10.4 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.

13.8.10.5 Many diatom shells have complex internal structure.

13.8.10.6 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

13.8.12.1 Synthetic fibers can be any color, including clear, pink, red, purple, blue, green, yellow, etc.

13.8.12.2 Synthetic fibers typically lack the splayed ends that chrysotile bundles commonly exhibit. Many synthetic fibers display a cylindrical morphology.

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 The synthetic fiber polyethylene has a wispy habit very similar to that of chrysotile.

13.8.12.6 Polyethylene has a higher birefringence than chrysotile.

13.8.12.7 Polyethylene fibers will melt if the slide is placed on the hot plate whereas chrysotile will not.

13.8.13 Rutile (TiO_2)

13.8.13.1 Titanium oxide occurs naturally as the mineral rutile, TiO_2. Rutile generally occurs as small prisms or fine acicular needles.

13.8.13.2 Refractive indices are extremely high (α = 2.6 to 2.7, γ = 2.8 to 2.9).
13.8.13.3 Rutile can be gray, brown, reddish-brown, or nearly opaque.
13.8.13.4 Needles of rutile have high birefringence, are length slow, and show parallel extinction.
13.8.13.5 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 crystals and are referred to as rutilated quartz.

14.0 RECORDING DATA AND RESULTS

14.1 Data Recording Sheets

14.1.1 Analysts record, by hand, on analytical data recording sheets (bench sheets), analytical results at the time the observations are made. Refer to Attachment 1 for a PLM-VE data recording 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, record the following data for each asbestos type that is present in the sample.
14.3.2 Morphology
14.3.3 Fiber color
14.3.4 Pleochroism (Yes or No)
14.3.5 Indices of refraction (α and γ)
14.3.6 Birefringence
14.3.6.1 Low if birefringence is less than 0.010; medium if birefringence is 0.010 to 0.050; high if birefringence is greater than 0.050.
14.3.7 Extinction characteristics
14.3.7.1 Parallel or oblique/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
14.3.10 Bin assignment for LA (see Section 13.7.5)

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 QA type as Not QA, 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.
15.1.2 All of the data recorded on the bench sheet is entered into an EDD in the form of an Excel spreadsheet.
15.1.2.1 The EDD was developed specifically for the Libby project, and the laboratory should check with the client to be sure it is using the most recent version of the spreadsheet.
15.1.2.2 Only one EDD is produced for each work order number.
15.1.2.3 Data entry instructions are provided on the spreadsheet.
15.1.3 After entering all data into the EDD, save the file by clicking on the macro button located on “Visual_data entry” worksheet.
15.1.3.1 The file name is generated automatically by concatenating Information entered on the “General_data entry” worksheet.
15.1.3.2 The information used to create the file name is the laboratory name, work order number, and analysis type (visual estimation).
15.1.4 The directory where the macro will save the file depends on how the template spreadsheet was opened.
15.1.4.1 Be sure there is a blank spreadsheet template in each folder where EDD’s will be saved.
15.1.4.2 If Excel is opened, and then the blank template spreadsheet is opened, the file will be saved in the same directory where the original blank template spreadsheet was opened from.
15.1.4.3 Do not open the blank template spreadsheet from Windows Explorer, because then the file will be saved at the computer’s default directory for Excel (generally, this default directory is C:\Documents and Settings\My Documents).
15.1.5 The EDD serves as an electronic version of the test report submitted to the client.
15.1.5.1 A hard copy of the test report is also mailed or couriered to the client following delivery of the EDD (see Section 15.3 for further details about hardcopy data reports).
15.1.5.2 The laboratory retains all original records for use in resolving any questions until otherwise instructed by EPA.

15.2 Data Package Generation

15.2.1 Hardcopy reports of the raw analytical data are submitted to EPA, or their project oversight contractors, for archival.

15.2.2 A completed data package consists of a cover sheet signed and initialed by approved signatories and the following documentation:

15.2.2.1 Number of samples received, and copies of the signed chains of custody.

15.2.2.2 The date of sample receipt and condition of samples.

15.2.2.3 The 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 Verification that microscope slides were wiped clean before use.

15.2.2.5 Calibration data for the RI liquids used in the analysis.

15.2.2.6 Verification that the microscope was properly calibrated before use.

15.2.2.7 Verification that reference materials were used for comparison when performing calibrated visual estimates of asbestos content.

15.2.2.8 Visual Estimate hard copy data forms, as presented in the EDD and containing the analytical data (including all cross-check and self-check QC’s performed on any samples in the work order number).

15.2.2.9 Copies of the handwritten bench sheets containing the analyst’s original data and observations.

15.2.3 Refer to Attachment 3, the Data Package Checklist, for a complete list of items required for each data package.

15.2.4 Each test report is identified by a unique Laboratory Information Management System (LIMS) number called a Work Order Number, Job Number, or equivalent.

15.2.5 When opinions and interpretations are provided in a test report, the laboratory will:

15.2.5.1 Document the basis on which the opinions and interpretations were made.

15.2.5.2 Clearly indicate on the test report which items are opinions and interpretations.

15.2.6 Once the data package 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 EPA’s data management software.

15.3.1.2 A scanned .pdf of all items in the data package described above, including the cover sheet signed by an approved signatory, the signed chains of custody, and the analyst’s original bench sheets. 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, a hardcopy of the data package is sent to the client through the mail.

16.0 QUALITY ASSURANCE AND QUALITY CONTROL

16.1 General

16.1.1 The laboratory operates 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 (self-check analysis) or by a different analyst (cross-check analysis).

16.1.3.2 Repeated analyses on calibration standards of known asbestos concentration.

16.1.3.3 NIST proficiency testing.

16.1.3.4 Inter-laboratory analyses.

16.1.4 Records are 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 Libby Amphibole 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 Regular analysis of the calibration standards provides a routine check of analyst precision and accuracy.

16.3.1.3 Calibration standards are read at a frequency on 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.

16.3.4 The Laboratory Manager, QA/QC Coordinator, or designate other than the analyst performing the test, will 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. For example, the reported value of blind calibration standards below 1% should fall in the correct concentration bin.

16.3.6 Repeated analysis of the calibration standards provides a benchmark upon which analysts may 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 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 data package. Rather, laboratories are responsible for maintaining an internal system for tracking analyses of these calibration standards.

16.4 Self-Check and Cross-Check QC Analyses (Duplicates and Replicates)

16.4.1 For each set of samples, 10% of the samples must be re-analyzed within the laboratory.

16.4.2 A QC analysis (self-check or cross-check) 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 self-check QC analyses on client samples will be 1 per 50 samples analyzed (2%). Self-check analyses should be performed as a remount of the sample (see Section 13.3 for slide preparation procedures).

16.4.4 The frequency of cross-check QC analyses on client samples will be 8 per 100 samples analyzed (8%). Cross-check analyses should be done on the five original slide preparations.

16.4.4.1 All analysts performing QC analyses must be experienced with PLM analysis of soil samples from the Libby Asbestos Superfund 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 cross-check QC samples to another Libby laboratory with the proper experience and qualifications.

16.4.5 The self-check and cross-check analysis is acceptable if results are within a bin

category (i.e., ± 1 bin) for reported concentrations below 1% LA. For all asbestos types greater than 1%, it is recommended that precision is tracked using control charting or a similar tool.

16.4.6 Corrective action(s) must be taken immediately if re-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 to EPA, or their designate.

16.4.7 When performing a QC analysis, it is necessary to mark LDS or LDC in the "QA 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 Asbestos Superfund Site. The purpose of this program is to help detect and minimize laboratory biases and 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 EPA or their designate.

16.5.3 Results of the inter-laboratory analyses are reviewed by EPA, or their designate.

16.5.4 The inter-laboratory analysis is acceptable if results are within a bin category (i.e., ± 1 bin) for reported concentrations below 1% LA.

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 EPA, or their designate. 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 analyst re-training.

17.0 REFERENCES

17.1 Bandli, B.R. et al. (2003). Optical, compositional, morphological, and X-ray data on eleven Eleven particles of amphibole from Libby, Montana, U.S.A. Canadian Mineralogist, 41, 1241-1253.

18.2 Bloss, F.D. (1999). Becke Line Chart. Optical Crystallography. Mineralogical Society of America, Monograph Series Publication No. 5.

18.3 Cornelis Klein and Cornelius S. Hurlbut, Jr. (1985). Manual of Mineralogy. John Wiley & Sons.

18.4 Deer, W.A., Howie, R.A., and Zussman, J. (1997). Rock Forming Minerals Volume 2B: Double Chain Silicates. 2nd Edition. The Geological Society, London.

18.5 EPA Method 600/R-93/116: Method for the Determination of Asbestos in Bulk Building Materials. (July 1993).

18.6 Federal Register, 40 CFR Part 763, Volume 52, No 210, "Asbestos Containing Materials in Schools; Final Rule and Notice."

18.7 McCrone Research Institute (1987). Dispersion Staining Color Chart.

18.8 Meeker, G.P., Bern, A.M., Brownfield, I.K., Lowers, H.A., Sutley, S.J., Hoefen, T.M., and Vance, J.S. (2003). The Composition and Morphology of Amphiboles from the Rainy Creek Complex Near Libby Montana. American Mineralogist, 88, 1955-1969.

18.9 NIOSH Method 9002: Asbestos (bulk) by PLM. Manual of Analytical Methods, 4th Edition. (August 1994).

18.10 NIST Handbook 150, 2006 Edition, "NVLAP Procedures and General Requirements."

18.11 State of California Air Resources Board Method 435: Determination of Asbestos Content of Serpentine Aggregate. (June 1991).

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

18.13 Su, Shu-Chun (1996). Rapidly and Accurately Determining Refractive Indices of Asbestos Fibers by Using Dispersion Staining Method.

18.14 Terry and Chilingar (1955). Comparison Chart for Visual Percentage Estimation.

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

18.16 Wylie, A.G. and Verkouteren, J.R. (2000). Amphibole asbestos from Libby, Montana: Aspects of nomenclature. American Mineralogist, 85, 1540-1542.

19.0 LIST OF ATTACHMENTS

19.1 Attachment 1: PLM-VE Data Recording Sheet

19.2 Attachment 2: RI Liquid Calibration Conversion Tables

19.3 Attachment 3: Data Package Checklist from PLM Data Sheet and EDD

19.4 Attachment 4: Optical Properties of Fibrous Amphiboles

19.5 Attachment 5: PLM Photomicrographs Demonstrating a Wide Range of Libby Amphibole Morphologies

19.6 Attachment 6: SEM Photomicrographs of Representative Examples of Libby Amphibole Morphologies

19.7 Attachment 7: Photomicrographs of Representative Fields of View of 0.2% and 1.0% Libby Amphibole Controlled PE Reference Materials

19.8 Attachment 8: Flow Diagram for Determining LA Content by Complementary Use of Stereomicroscopic Examination and PLM Visual Estimation

ATTACHMENT 1

PLM-VE Data Recording Sheet

PLM VISUAL ESTIMATION DATA RECORDING SHEET

Laboratory Name _____ Date Received _____

Job Number _____ SOP Name/Revision _____

Note: Data Recording Sheet is formatted to print on 11x17 paper.

EPA Index ID	Index Suffix Char.	Index Suffix No.	QA Type (NOT QA, LDS, LDC)	Lab Sample ID	Date Analyzed	Analyst Name	Stereomicroscopy Examination					Libby Amphibole		Other Amphibole			Chrysotile		Deviation?	Comments (list below)	OPTICAL PROPERTIES FOR LA (see key for appropriate data inputs)							
							Sample Appearance	Est. % LA		Est. % OA and C											Morph.	Fiber Color	Sign Elong. (+/)	Pleoch. (Y/N)	Angle Extinct.	Ref. Index α	Ref. Index γ	Biref.
								Qual (ND, Tr, <)	Mass Fract (%)	Qual (ND, <)	Area Fract (%)	Qual (ND, Tr, <)	Mass Fract (%)	Qual (ND, <)	Area Fract (%)	OA Type (AMOS, ANTH, CROC, UNK)	Qual (ND, <)	Area Fract (%)										

Comments (Use back if needed)

ATTACHMENT 2

RI Liquid Calibration Conversion Tables

Prepared by Dr. Shu-Chun Su, Hercules, Inc.

See attached Excel spreadsheet entitled

"Create_RI_Liquid_Calibration_Conversion_Tables.xls"

ATTACHMENT 3

Data Package Checklist

From PLM (VE and PC) Data Sheet and EDD.xls

STANDARD LABORATORY DATA PACKAGE CHECKLIST
Analytical Test Report
Bulk Asbestos Analysis by Polarized Light Microscopy (PLM)

Prepared For:

City/State: _____

Laboratory Name: _____
City/State: _____
Laboratory Job No.: _____
Method Utilized (SOP and Rev. No.): SRC-LIBBY-03/Revision 2
Circle One: Visual Estimation Point Counting Approach

Report Reviewed by: _____

STANDARD LABORATORY DATA PACKAGE CHECKLIST

Instructions: **For PLM analytical results raw data packages, complete and sign the following checklist. Attach supporting documentation as outlined below. Organize the supporting documentation in the order listed below. Paginate the completed raw data package.**

		Laboratory Verification (Initials and Date)	Validator Verification (Initials and Date)
1	**Number of samples received:**_____ *An SDG is defined as no more than 200 samples.* **Additional Supporting Documentation:** Attach COC forms having footer R (report).		
2	**Date of sample receipt and condition of samples:**_____ *For Condition of samples enter "OK" or "See SDG Case Narrative".*		
3	**SDG Case Narrative:** **Additional Supporting Documentation:** Attach SDG Narrative and any modification forms.		
4	**Check for contamination (daily):** Wipe microscope slides with lens paper before using. *Laboratory Verification initial and date signifies that this has been performed for the samples in this SDG.*		
5	**Verification of the refractive indices of the refractive index liquids once per month:** **Additional Supporting Documentation:** Provide information indicating a monthly record of checking each of the four liquids including liquid name, lot number and analyst initials. (See table - Results of RI Liquids Calibration)		
6	**Verification of microscope adjustments prior to each SDG:** *Laboratory Verification initial and date signifies that this has been performed for the samples in this SDG.*		
7	**Reference material - Visual Estimation Approach:** *Laboratory Verification initial and date signifies that this has been performed for the samples in this SDG.*		
	Reference material - Point Counting Approach: **Additional Supporting Documentation:** Provide calibration curve documentation, printed from the EDD spreadsheet.		
8	**VE and/or PC hard copy data forms (as presented in the EDD spreadsheet):** **Additional Supporting Documentation:** Copies of the Hard Copy Data Forms for all investigative samples and laboratory duplicates will be provided from systems that are entered electronically.		
9	**Bench sheets for data results:** **Additional Supporting Documentation**: Provide copies of the hand written or LIMS system generated raw data sheets for sample results.		

COCs

SDG NARRATIVE

Instructions: The following information should be included in all narratives. Please see the attached narrative template.

1 List the method or methods used.
2 For any modifications, reference the modification number and attach a copy of the signed document to the raw data
3 If sample condition is not "OK", explain why and any implications to the data.

SDG NARRATIVE EXAMPLE

SDG Narrative - PLM Analysis by SRC-LIBBY-03 Revision 2

Laboratory Job Number:_____

The samples were received in sealed coolers [or other container]. [Any special notations about the samples as received goes here such as damaged in shipping, missing sample, etc.] The sample set was assigned a laboratory job number, each sample was assigned a unique, sequential laboratory ID number, and the job was entered into the Laboratory Information System. The laboratory ID numbers, shipping information and signatures were recorded on the CDM Chain of Custody and the login information was summarized on the laboratory Chain of Custody.

Samples were analyzed in accord with SRC-LIBBY-03 Rev. 2 [with modifications described on Laboratory Modification document(s): LB-_____ (see attached)].

SAMPLE RESULTS

See Attached Sample Results

Instructions: These sample result forms are from the current version of the PLM (VE & PC) Data Sheet and EDD.xls file. They are labeled in this file as the VE or PC hard copy data form.

BENCH SHEETS

Instructions: Please provide handwritten or LIMS system generated raw data sheets for sample results.

REFRACTIVE INDEX LIQUIDS

Instructions: Please see and follow attached table from Shu-Chun Su, Technical Expert for NVLAP Asbestos Programs. (Suggested Format for Recording Results of RI Liquids Calibration using Cargille Glass Standard and Dispersion Staining Method - Version: February 1996)

The following components are included in the table:

1 Date
2 Nominal or Labeled n_D 25 degree Celsius
3 Cargille Glass
3a Nominal or Labeled R.I.
3b Lot No.
4 Central Stop DS Observation
4a Predominant DS Color
4b Corresponding $alpha_0$
5 Liquid or Room Temperature (degree Celsius)
6 Actual or Calibrated n_D 25 degree Celsius
7 Difference between Calibrated n_D 25 degree Celsius and Labeled n_D 25 degree Celsius
8 Accept or Reject
9 Analyst

RESULTS OF RI LIQUIDS CALIBRATION

Date[1]	RI Liquid[2]	Cargille Glass		CSDS Color of Glass4		Liquid or Room Temperature (°C)[7]	Calibrated RI of Liquid $(n_D^{25°C})_{clb}$[8]	Absolute Difference Between Calibrated and labeled RI $(n_D^{25°C})_{clb} - (n_D^{25°C})_{lbl}$[9]	Accept or Reject[10]	Initials of Analyst[11]
	Labeled RI $(nD25°C)_{lbl}$	Labeled RI[3]	Lot No.[4]	Predominant CSDS Color[5]	Corresponding λ_0 (nm)[6]					
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	

1. Date: 2. The $n_D^{25°C}$ on the label of RI liquid bottle or $(n_D^{25°C})_{lbl}$; 3. The RI value on the label of Cargille calibrated glass vial; 4. The Lot No. on the label of Cargille calibrated glass vial; 5. The predominant central stop dispersion color displayed by glass fragments (do not be confused by the false CSDS color due to edge effect (see p.3). 6. The matching wavelength, λ_0, corresponding to the CSDS color in Column 5; 7. The temperature of the RI liquid or the room if the liquid's temperature can be considered to be in equilibrium with the room atmosphere; 8. The reading based on the values in Columns 6 and 7 from the conversion table for the liquid-glass combination. This value is the actual or calibrated RI of the liquid at 589 nm and 25 °C or $(n_D^{25°C})_{clb}$; 9. Column 8 minus Column 2; 10. If the *absolute* value of Column 9 is less or equal to 0.004, circle A for *acceptable*. Otherwise, circle R for *rejected*. 11. Analyst's initials.

$(nD25°C)_{lbl} = n_D^t + (25 - t)\, dn/dt$ (The temperature correction is built in the conversion tables.)

Version: December 1998 (Shu-Chun Su, Technical Expert for NVLAP Asbestos Programs)

ATTACHMENT 4

Optical Properties of Fibrous Amphiboles

OPTICAL PROPERTIES OF FIBROUS AMPHIBOLES ASSOCIATED WITH LIBBY AMPHIBOLE[A]

Libby Amphibole asbestos (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 a group of minerals, such as tremolite, actinolite, winchite, richterite, magnesio-riebeckite, and magnesio-arfvedsonite. The optical properties for each individual mineral are provided below. As seen, 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 under this SOP. Rather, if the sample exhibits the optical properties of a mineral listed below, the specific optical properties (refractive indices, birefringence, extinction angle, and elongation sign) shall be noted on the analytical data sheet and electronic file, and the mineral identified as LA.

Mineral	Morphology and Color	Refractive Indices		Birefringence	Extinction	Elongation Sign
		α	γ			
Tremolite[7]	Straight to curved fibers and bundles. Colorless to pale green.	1.600-1.628 1.604-1.612 1.599-1.612 1.6063	1.625-1.655 1.627-1.635 1.625-1.637 1.6343	0.017-0.028	Oblique (up to 21 °);	+ (length slow)
Actinolite[7]		1.600-1.628 1.612-1.668 1.613-1.628 1.6126	1.625-1.655 1.635-1.688 1.638-1.655 1.6393	0.017-0.028		+ (length slow)
Winchite	Straight to curved fibers or bundles. Colorless to pale blue Pleochroism weak to moderate: X-colorless, Y=light blue-violet, Z=light blue[3]	**1.618-1.626**[1] **1.618-1.621**[2] 1.629[3] 1.636[4]	**1.634-1.642**[1] **1.634-1.637**[2] 1.650[3] 1.658[4]	**0.008-0.019**[1] **0.016**[2] 0.021[3] 0.022[4]	Oblique, **22°**[1] **15.8°**[2] Oblique, 7-29°[8]	+ (length slow)
Richterite	Straight to curved fibers or bundles. Colorless, pale yellow, brown, pale to dark green, or violet[8] Pleochroism weak to strong in pale yellow, orange, and red[5]	**1.622-1.623**[1] 1.605-1.624[5] 1.615[6]	**1.638-1.639**[1] 1.627-1.641[5] 1.636[6]	**0.012-0.017**[1] 0.017-0.022[5]	Oblique, **21-22°**[1] Oblique, 5-45°[8]	+ (length slow)
Magnesio-riebeckite	Prismatic to fibrous aggregates. Blue, grey-blue, pale blue to yellow. Can be pleochroic.[8]	1.650-1.673[8]	1.662-1.676[8]	Up to 0.015[8]	Oblique, 8-40°[8]	- (length fast)[8]
Magnesio-arfvedsonite	Prismatic to fibrous aggregates. Yellowish green, brownish green, or grey-blue. Can be pleochroic.[8]	1.623-1.660[8]	1.635-1.680[8]	0.012-0.026[8]	Oblique, 18-45°[8]	- (length fast)[8]

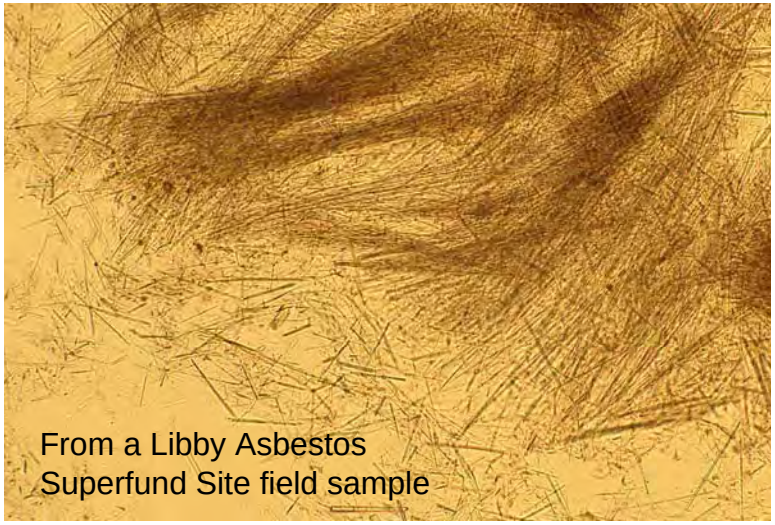
A. This table is adapted for use in the SOP from: Su, Shu-Chun, 2005. White paper: *Tables to Facilitate the Determination of Refractive Indices of Winchite and Richterite, (Libby, Montana) by Dispersion Staining*, **August 8, 2005** Data on this table were compiled from data of amphiboles from Libby, Montana and other localities. The data in **bold** are samples from Libby, Montana. The data of tremolite/actinolite are adapted from Table 2-2 of EPA/600/R-93/116.

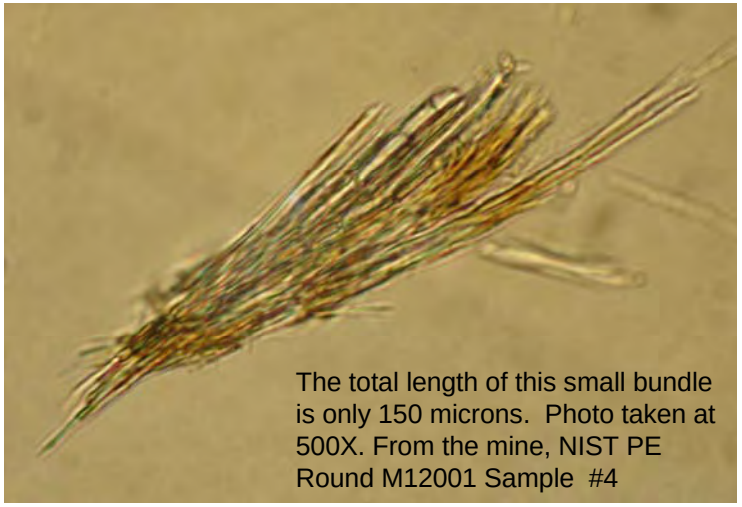
1. Bandli, B.R. et al. (2003) *Optical, compositional, morphological, and X-ray data on eleven particles of amphibole from Libby, Montana, U.S.A.* Canadian Mineralogist, 41, 1241-1253.
2. Wylie, A.G. and Verkouteren, J.R. (2000) *Amphibole asbestos from Libby, Montana: Aspects of nomenclature.* American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

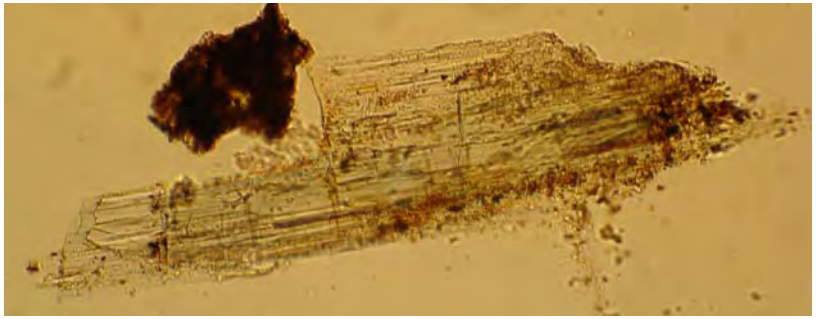
PLM Photomicrographs of Typical Libby Amphibole Morphology





Prismatic Libby Amphibole

The optical properties are the same as they are for more fibrous forms of LA. Colors of winhcite, 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 Libby Amphibole.



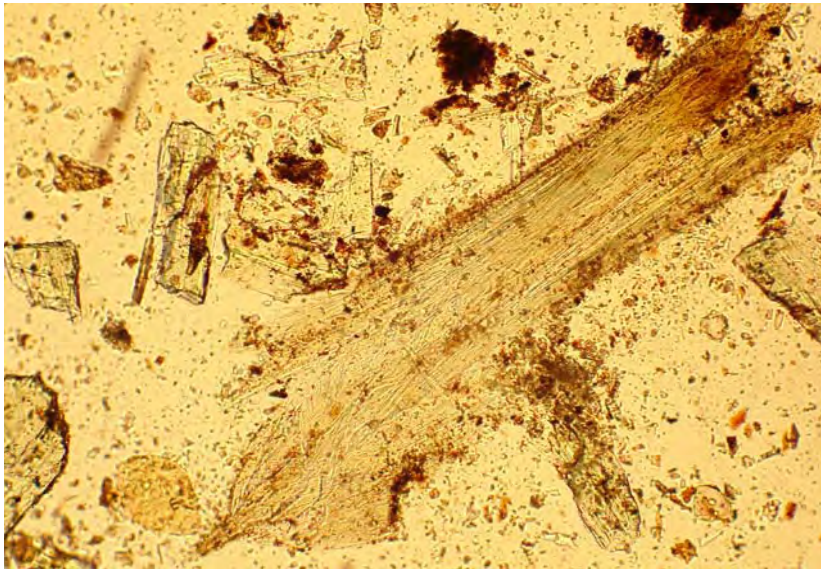
From a Libby Asbestos Superfund Site field sample

From the mine, NIST PE Round M12001 Sample #4

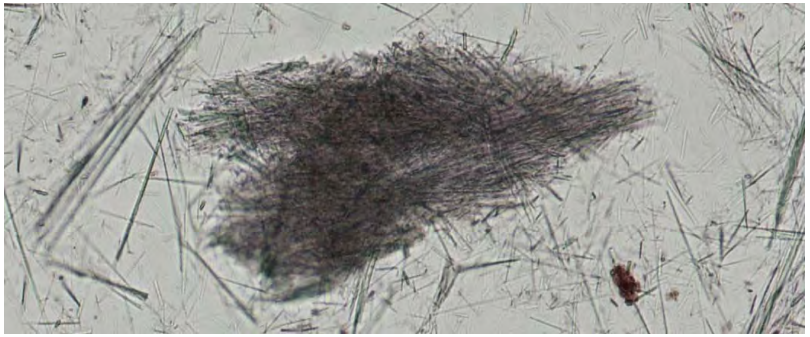
From the mine, NIST PE Round M12001 Sample #4

From a Libby Asbestos Superfund Site field sample

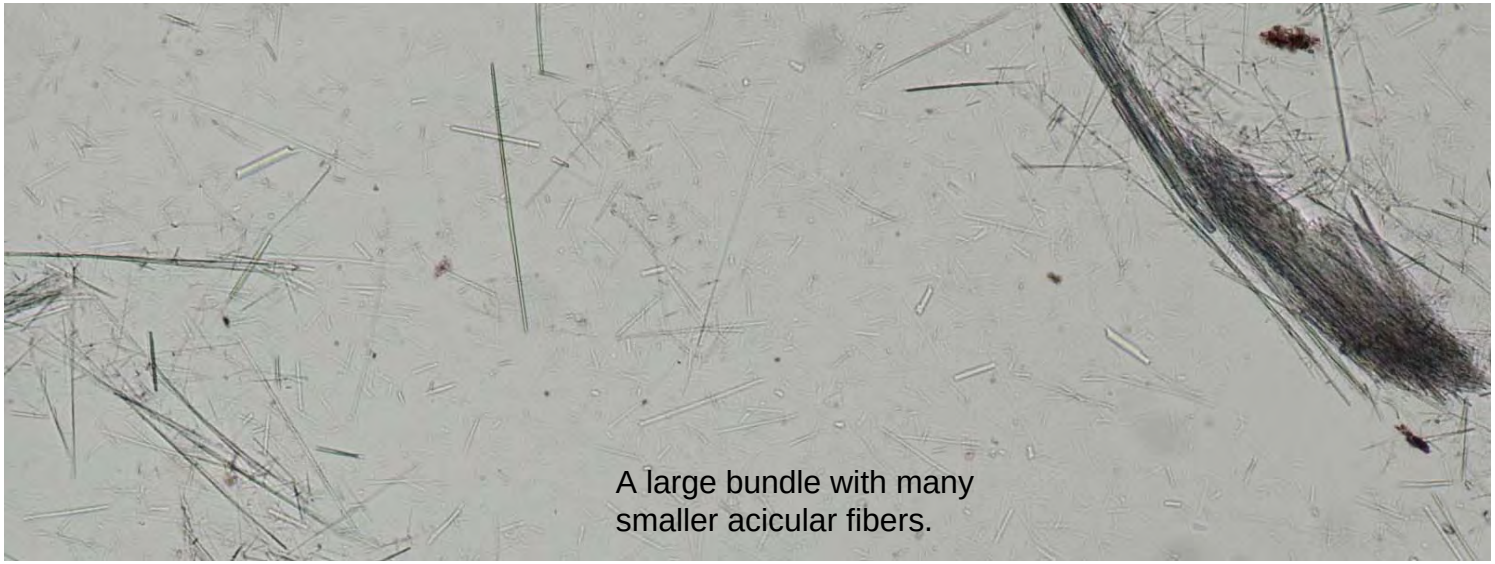
Some Libby Amphibole shows a “matted” or “felted” morphology. The internal structure of these bundles is still fibrous. The green high-relief prismatic crystals in the top right photo are hornblende. The bundles in the two top photos were found in Libby Asbestos Superfund Site field samples. The bundles 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.

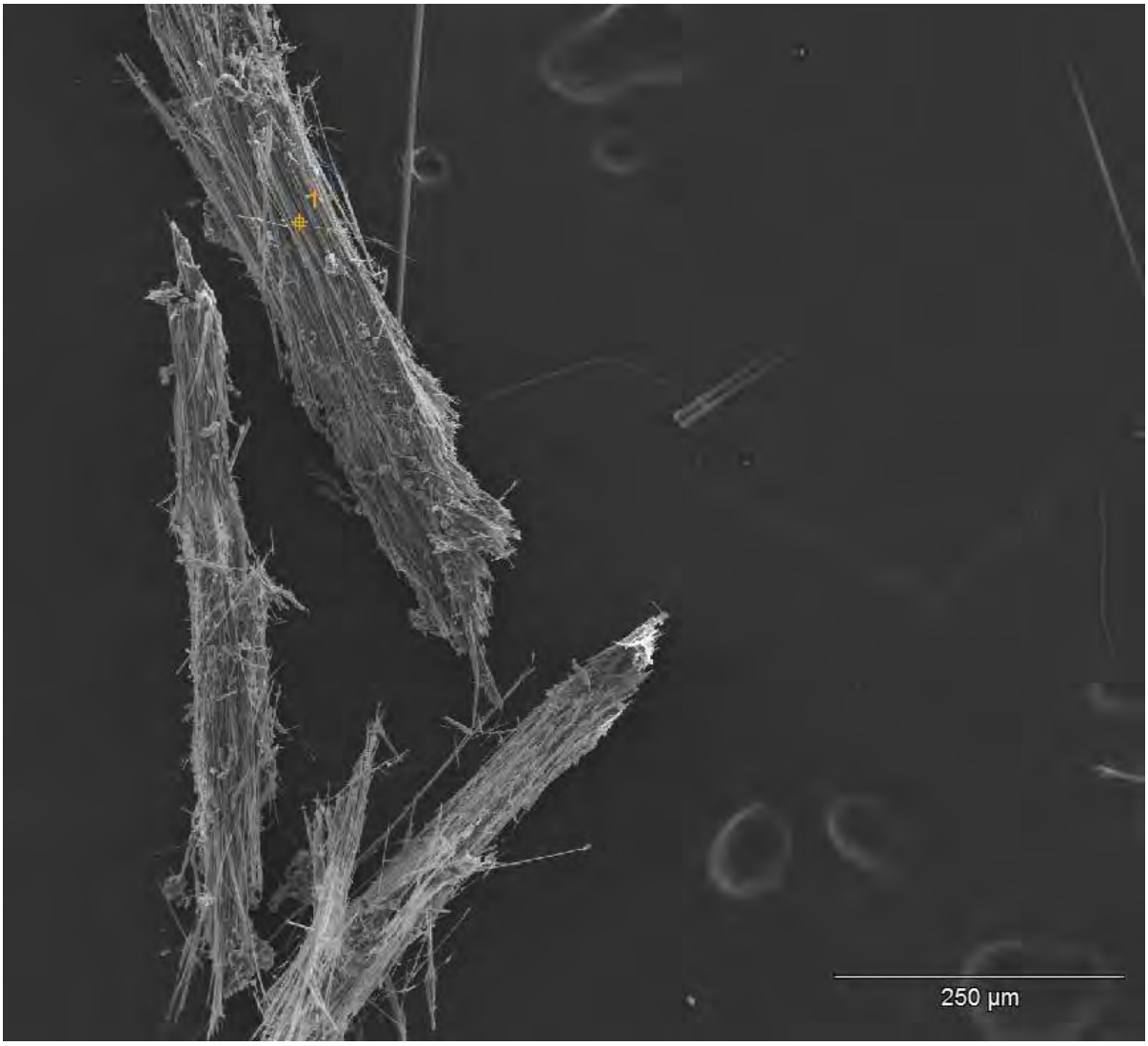
ATTACHMENT 6

SEM Photomicrographs of Representative Examples of Libby Amphibole Morphologies

SEM Photomicrographs of Representative Examples of Libby Amphibole Morphology

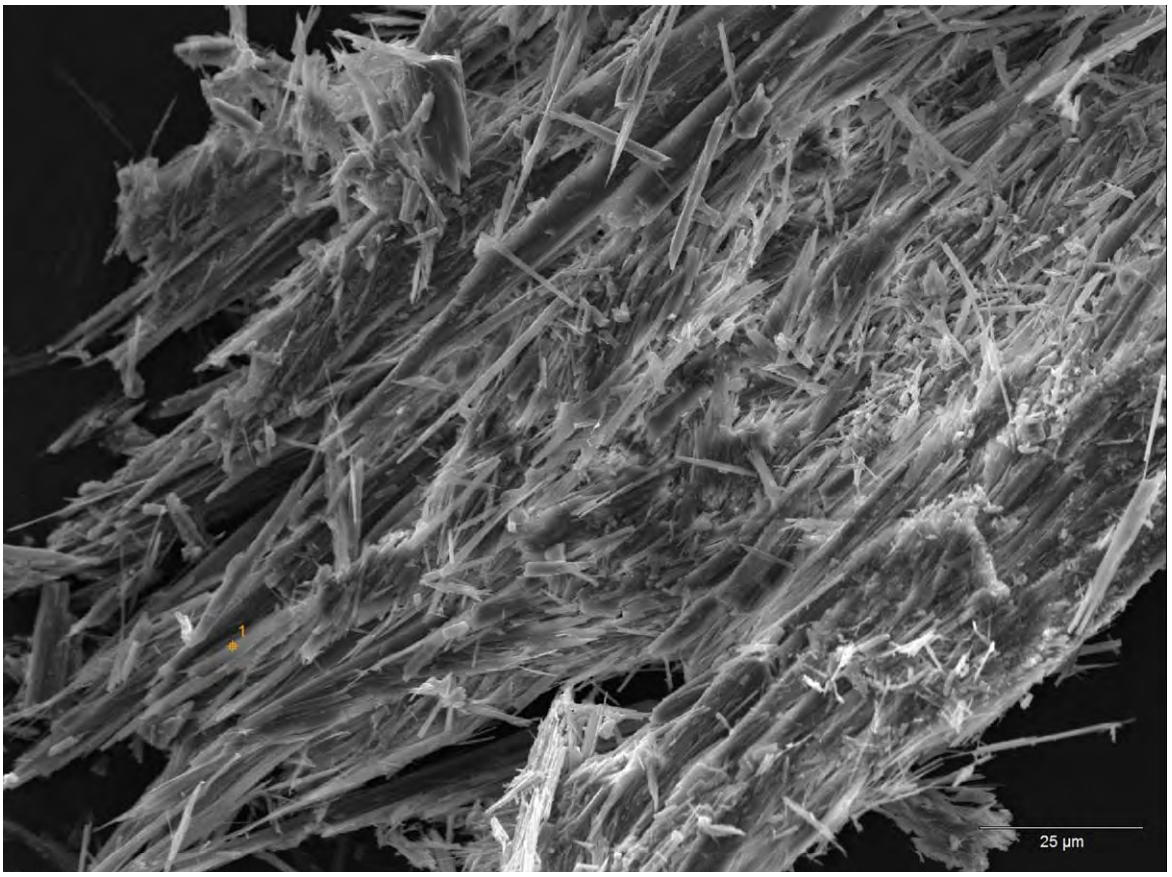
Individual bundles of Libby Amphibole were picked from soil samples at the ESAT Region 8 Laboratory and prepared for analysis by scanning electron microscopy (SEM). Slide mounts of these bundles were initially prepared in a refractive index liquid and the bundles were examined by PLM. Then the refractive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

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 morphologies 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.



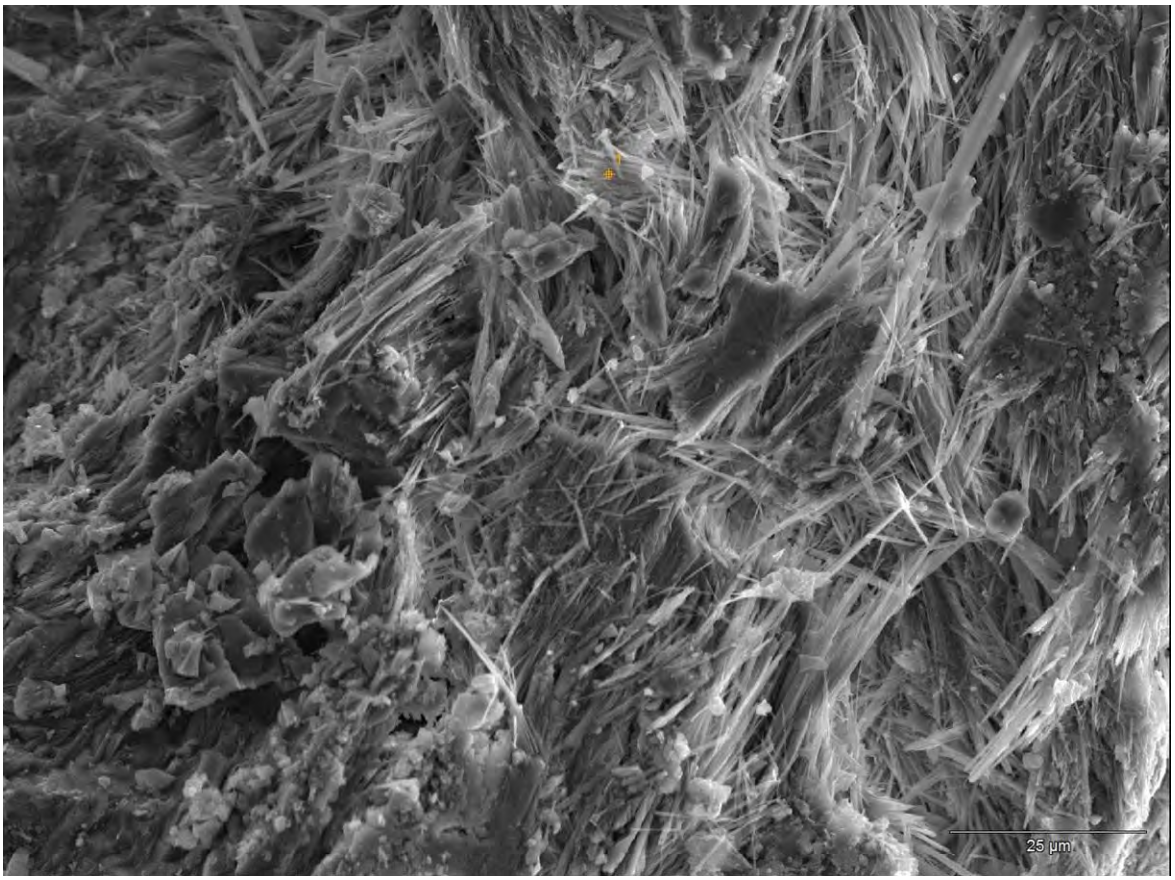
These are typical bundles of Libby Amphibole where the average aspect ratio of the fibers is high and most of the fibers are nearly parallel to one another. Note the scale in microns at the bottom of the photo. These three bundles are all of a size that can be seen with a stereomicroscope and picked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



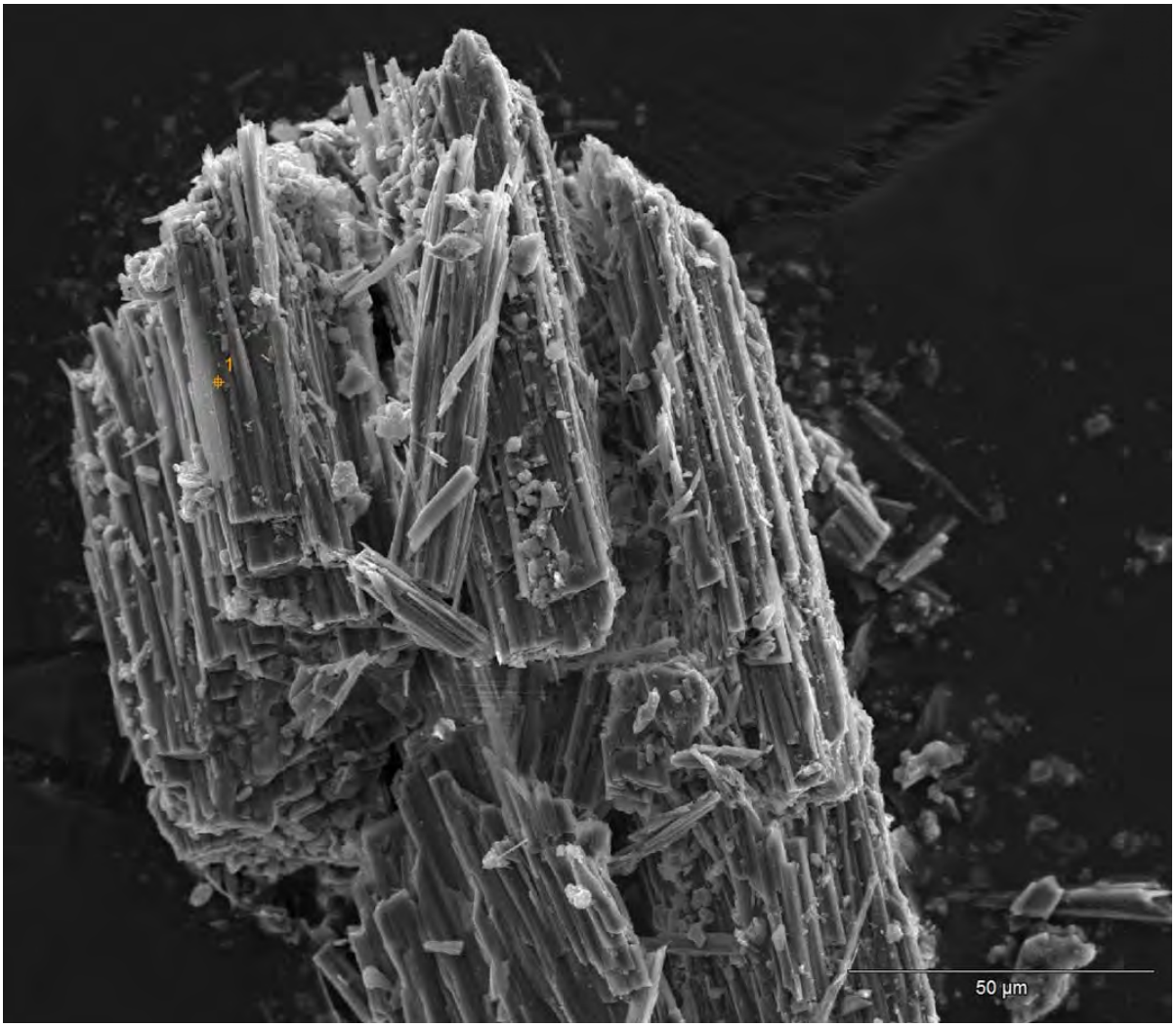
Varying degrees of parallelism can be seen in the fibers that compose bundles of Libby Amphibole. Note that the fibers in this bundle of LA are less parallel than the fibers in the bundles in the previous example.

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



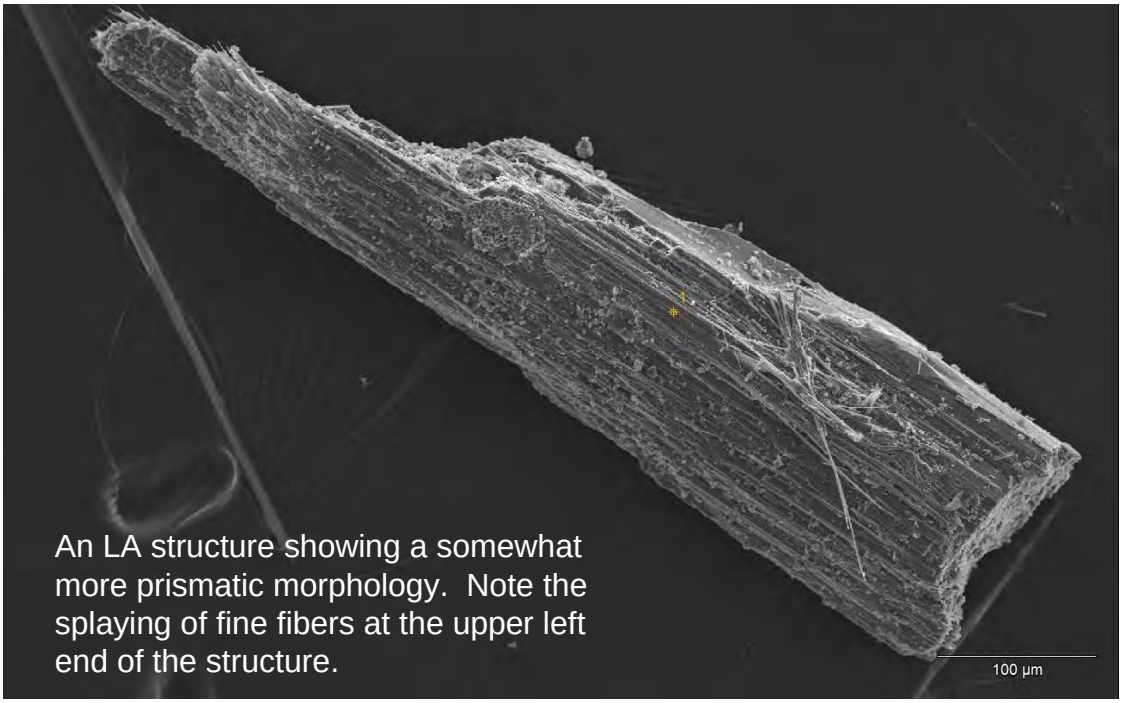
When this bundle of Libby Amphibole was viewed under PLM, its morphology was described as “felted”, or “matted”, with the fibers crossing at high angles to one another. This is how the bundle appeared when it was subsequently viewed by SEM. The fibrous nature of the “felted” or “matted” morphology is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

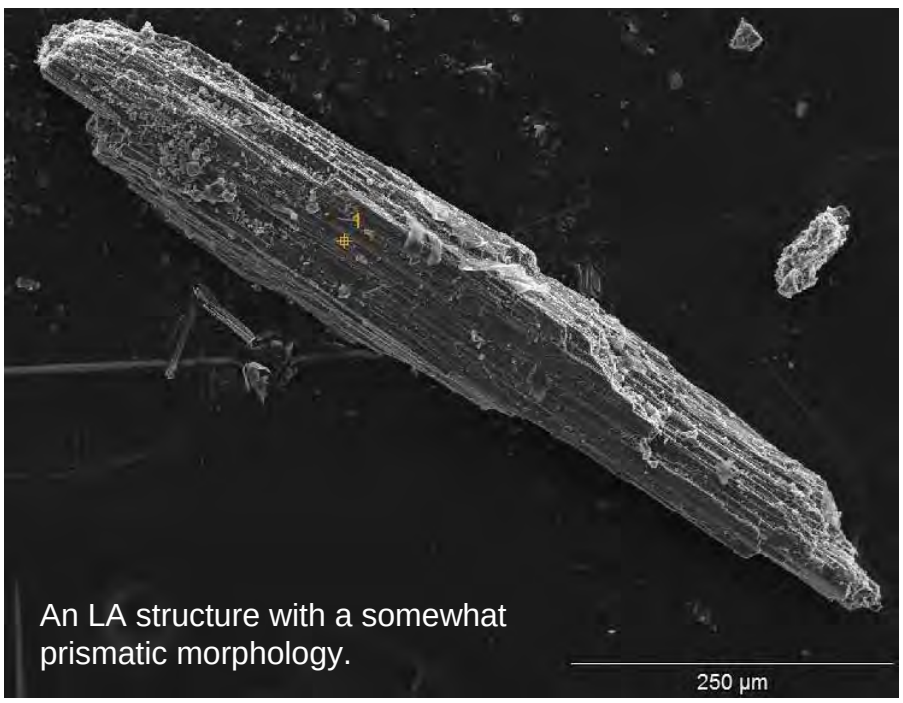


The average aspect ratio of the fibers in this bundle of LA is lower than those of the bundles in the previous examples. However, as seen by SEM, the bundle still splits readily into many small fibers.

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

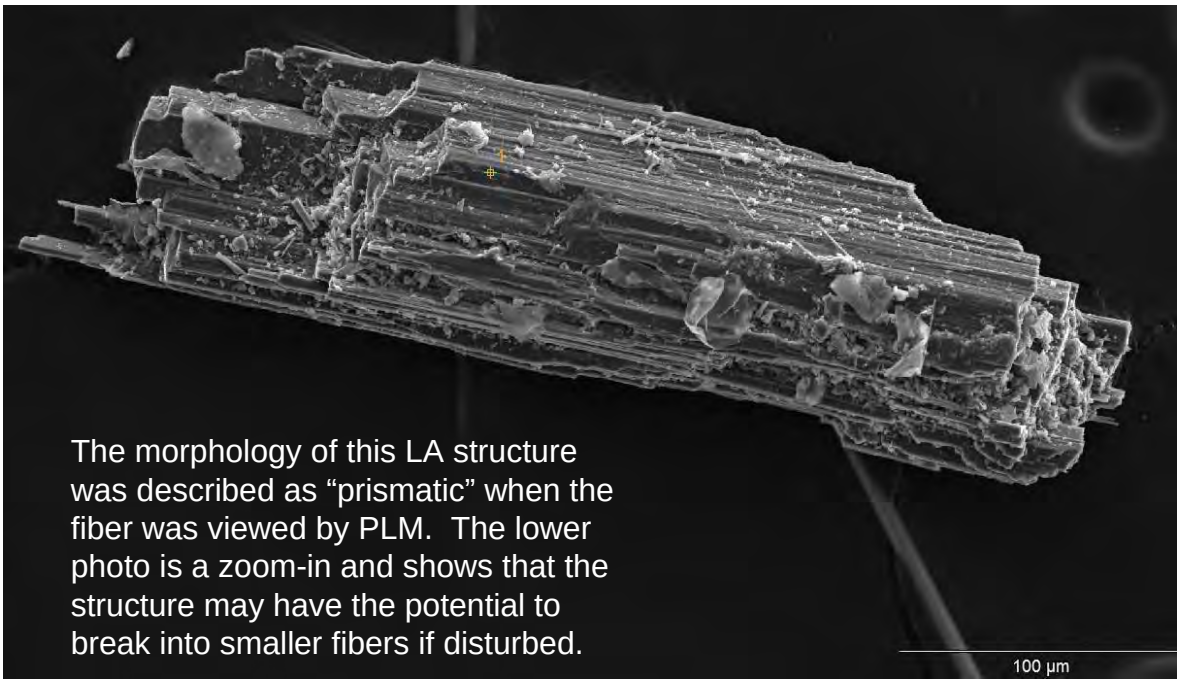


An LA structure showing a somewhat more prismatic morphology. Note the splaying of fine fibers at the upper left end of the structure.

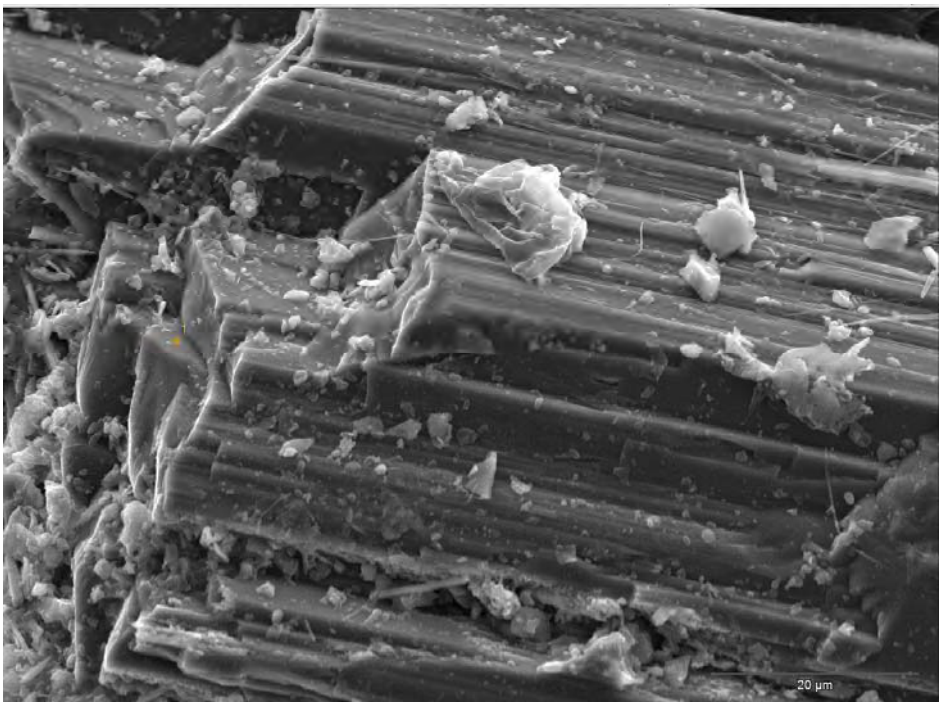


An LA structure with a somewhat prismatic morphology.

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



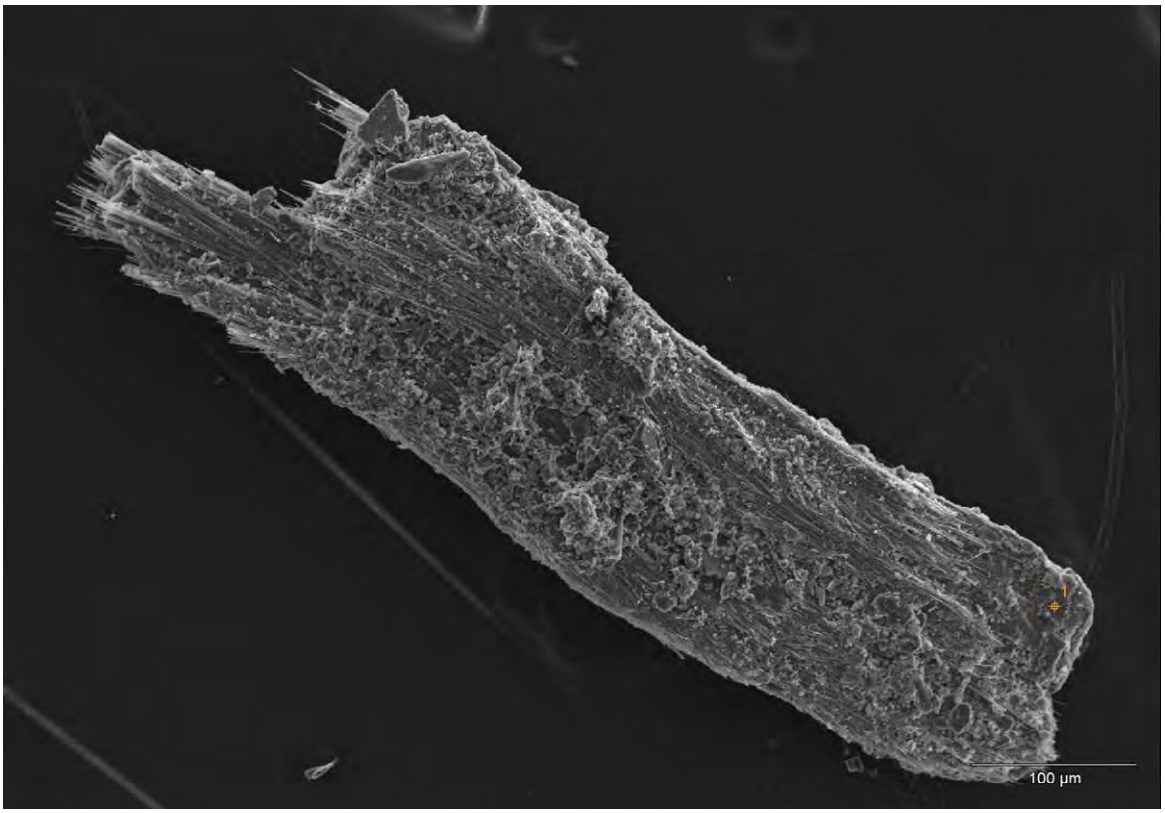
The morphology of this LA structure was described as “prismatic” when the fiber was viewed by PLM. The lower photo is a zoom-in and shows that the structure may have the potential to break into smaller fibers if disturbed.



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. Energy dispersive spectrometry (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 Libby Amphibole.

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 Libby Amphibole.

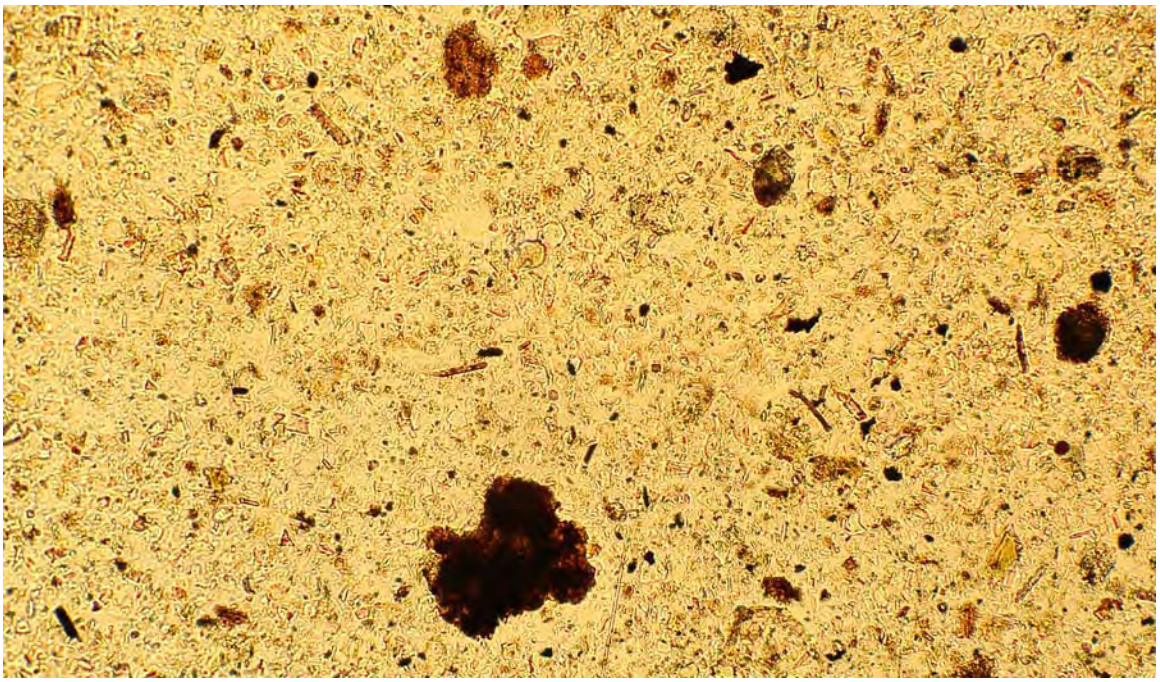
Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.

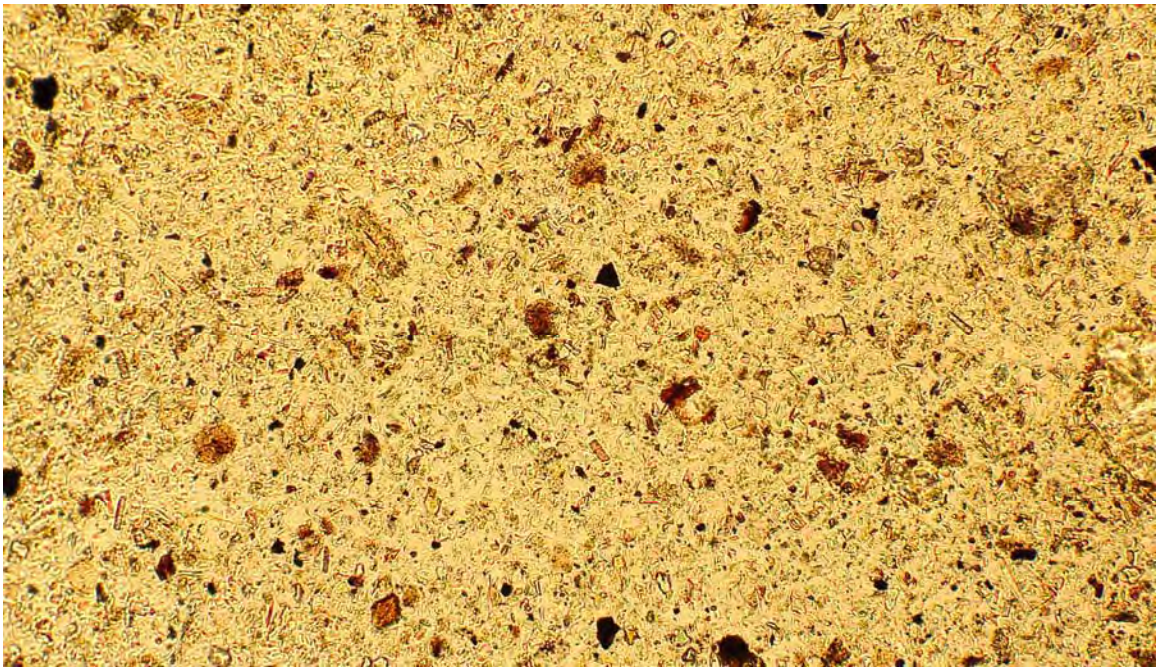
ATTACHMENT 7

Photomicrographs of Representative Fields of View of 0.2% and 1.0% Libby Amphibole Reference Materials

0.2% Libby Amphibole

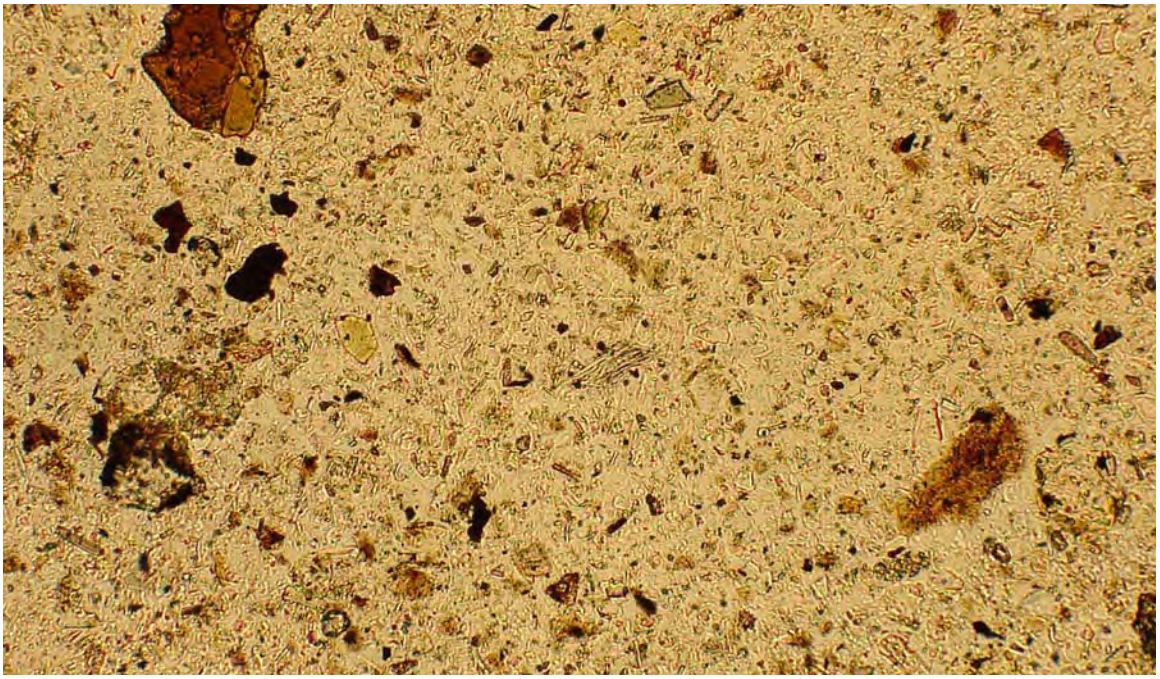
Photomicrographs of representative fields of view of the 0.2% Libby Amphibole by weight Controlled PE Reference Material. All photos taken at 100x, plane light in 1.55 refractive index oil. Width of each picture is approximately 1,500 microns.

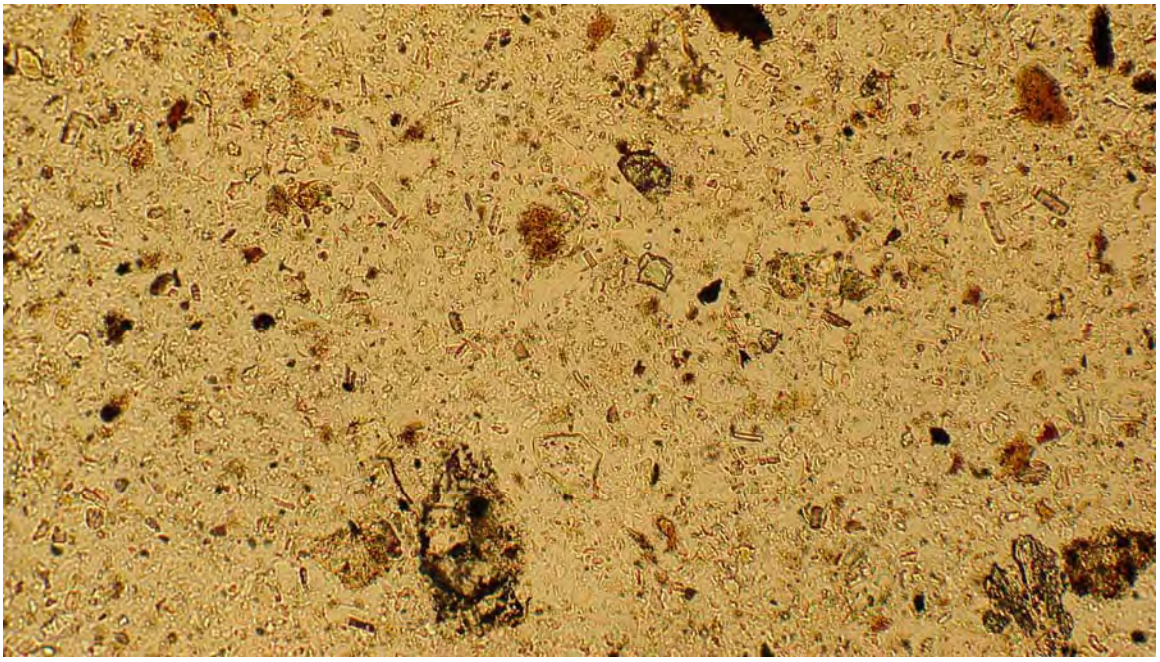




0.2% Libby Amphibole

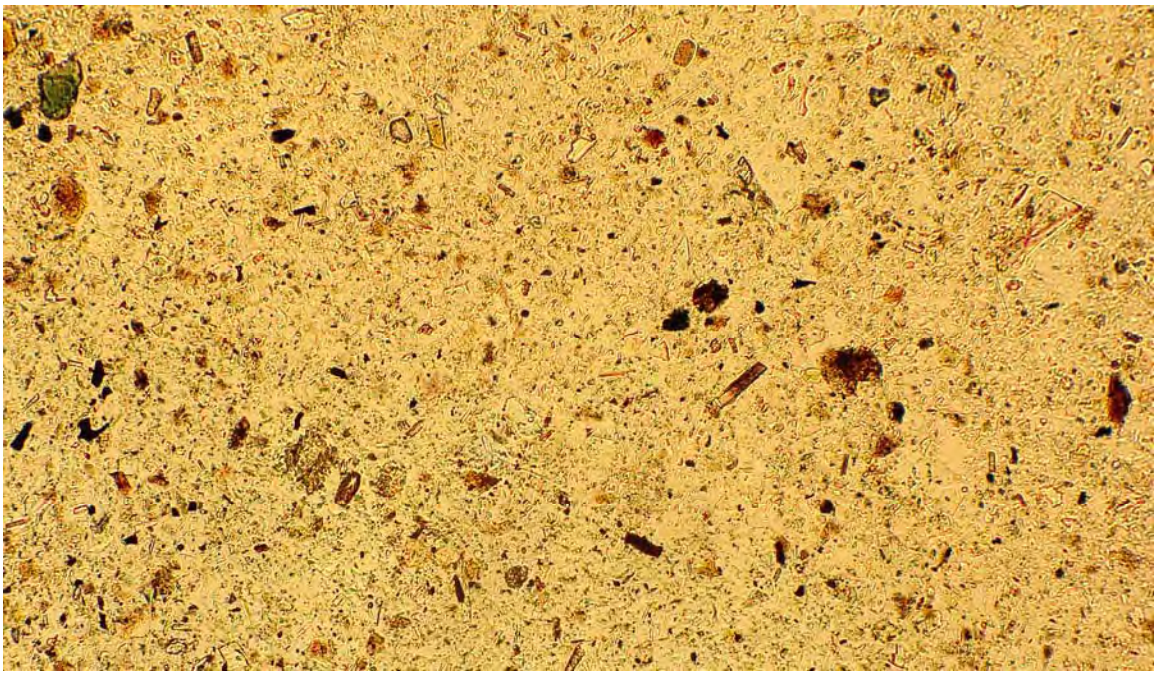
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

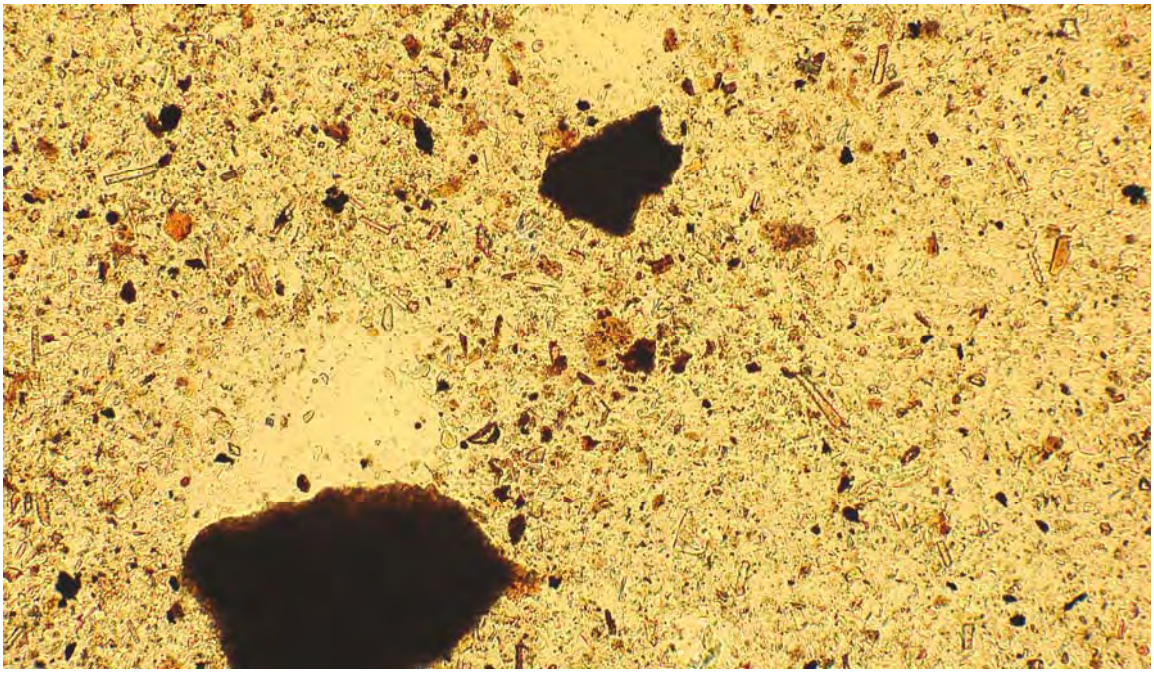




0.2% Libby Amphibole

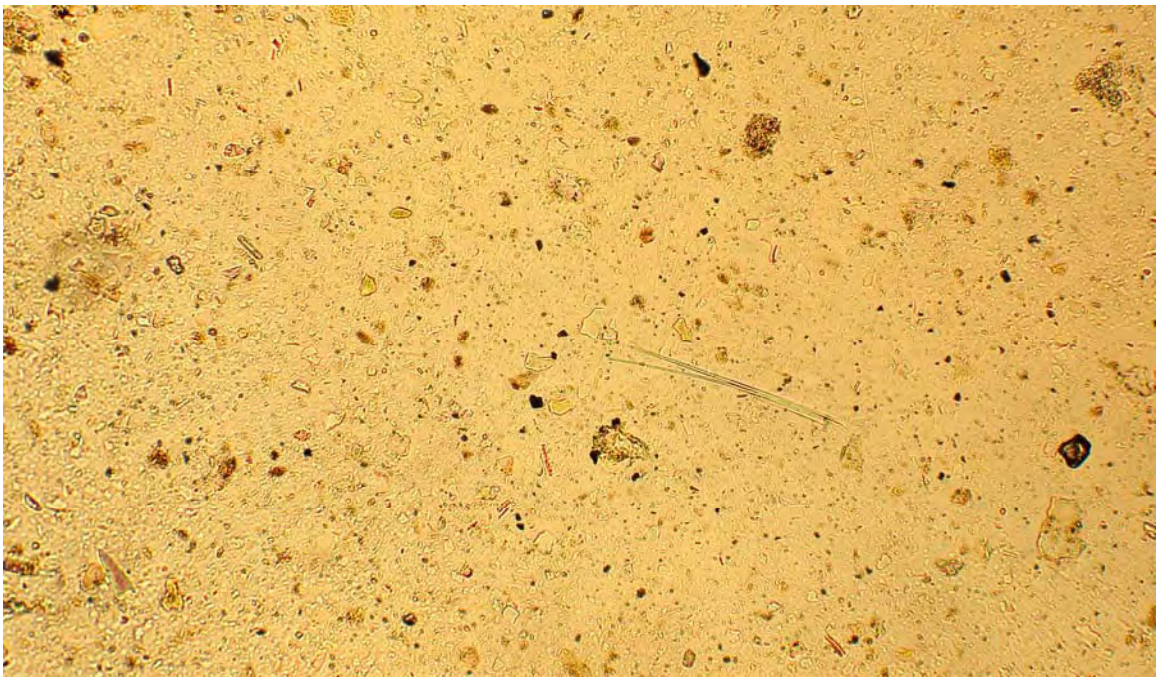
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

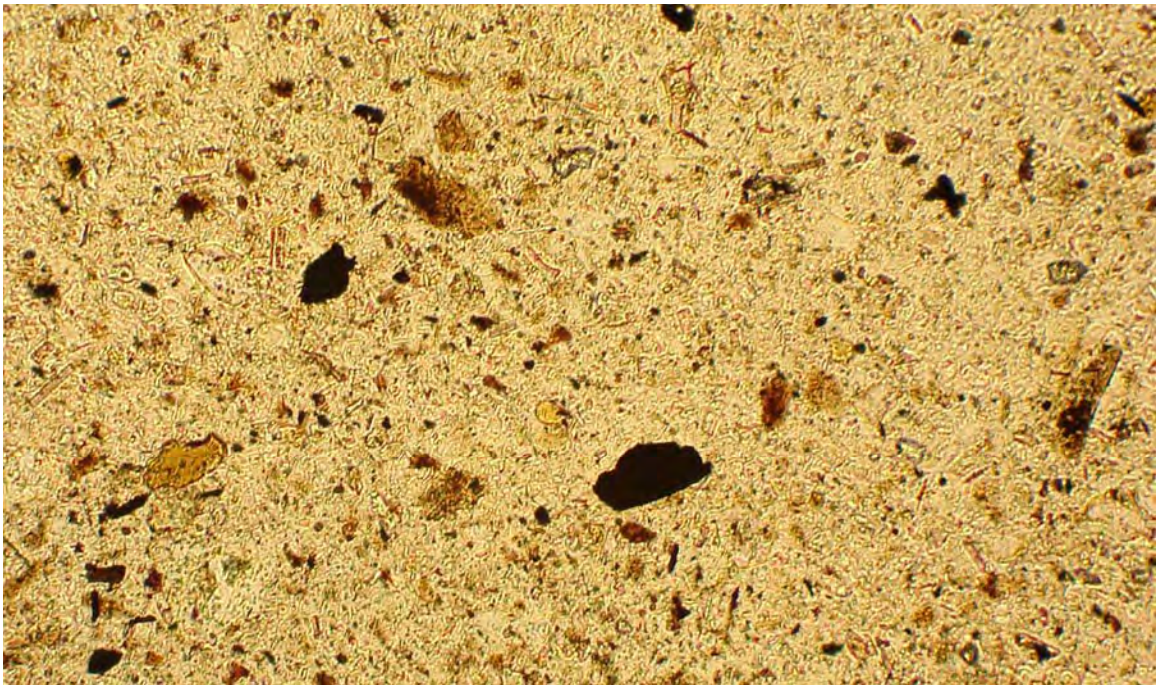




0.2% Libby Amphibole

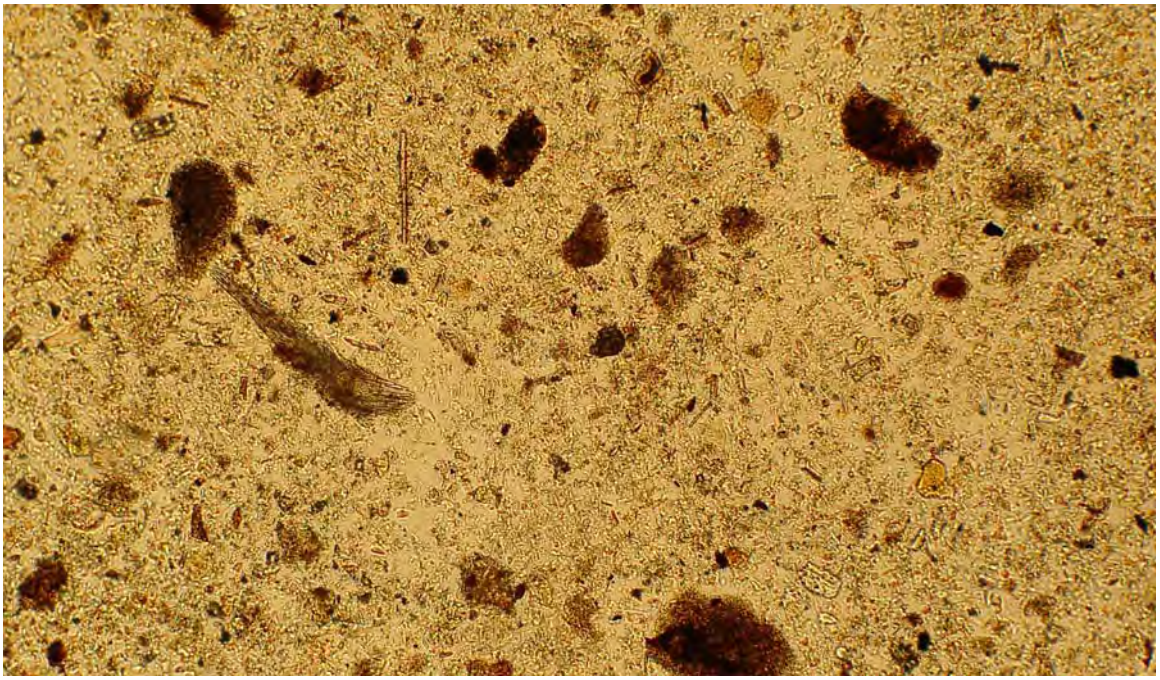
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

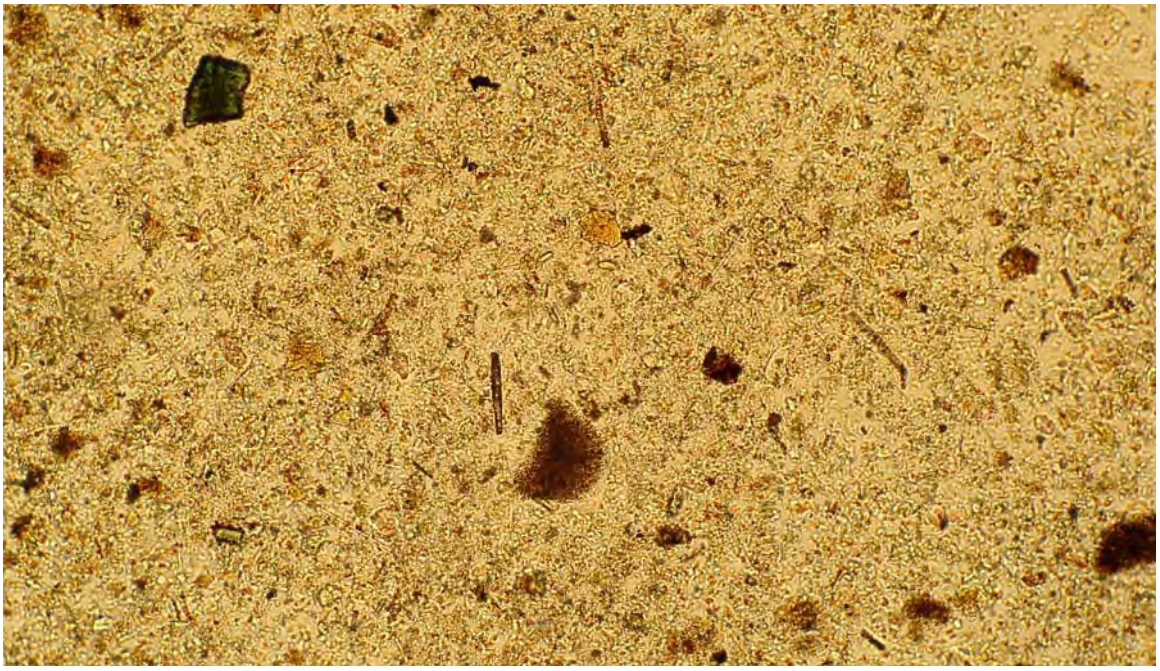




1.0% Libby Amphibole

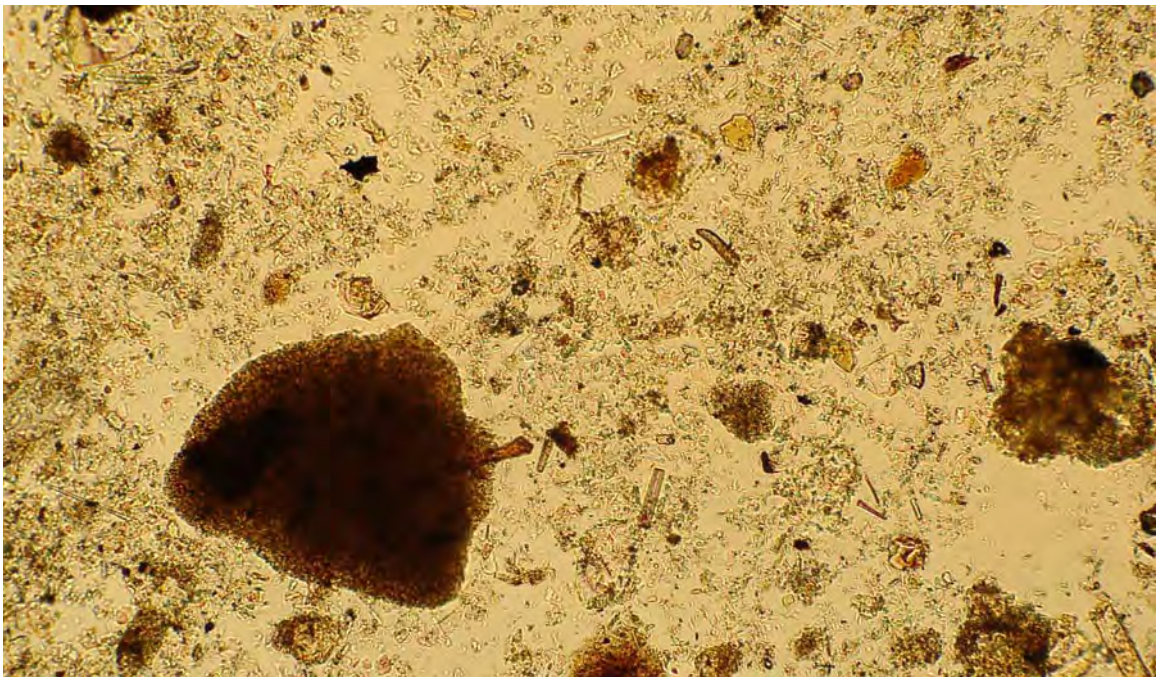
Photomicrographs of representative fields of view of the 1.0% Libby Amphibole by weight Controlled PE Reference Material. All photos taken at 100x, plane light in 1.55 refractive index oil. Width of each picture is approximately 1,500 microns.

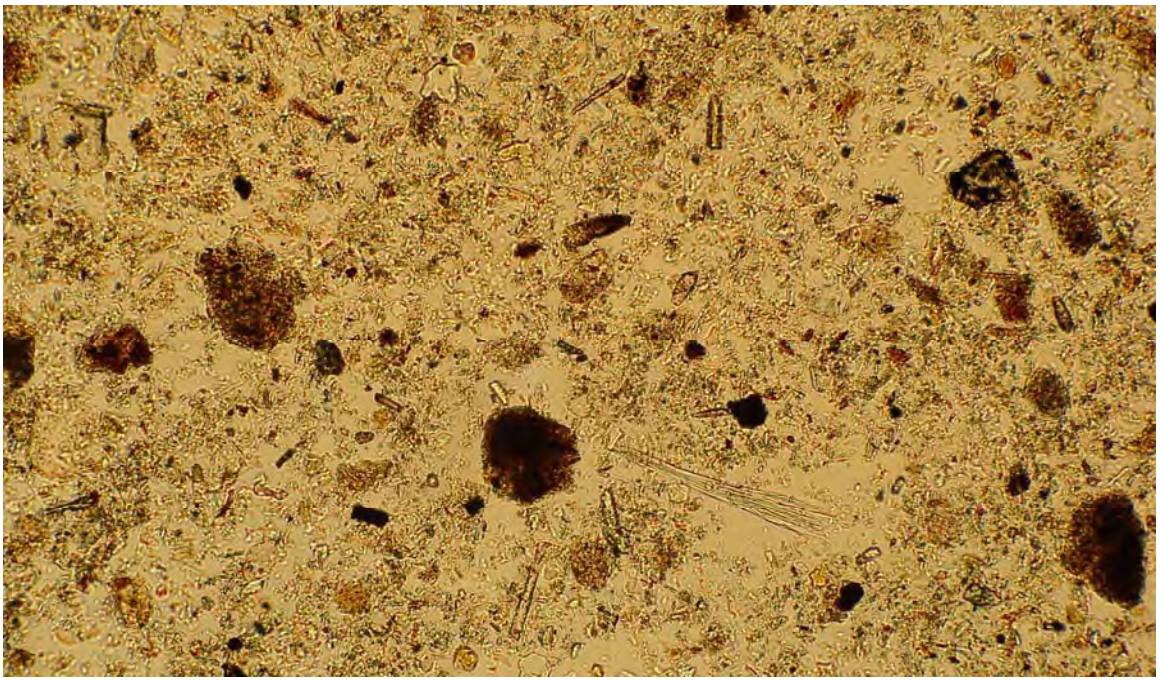




1.0% Libby Amphibole

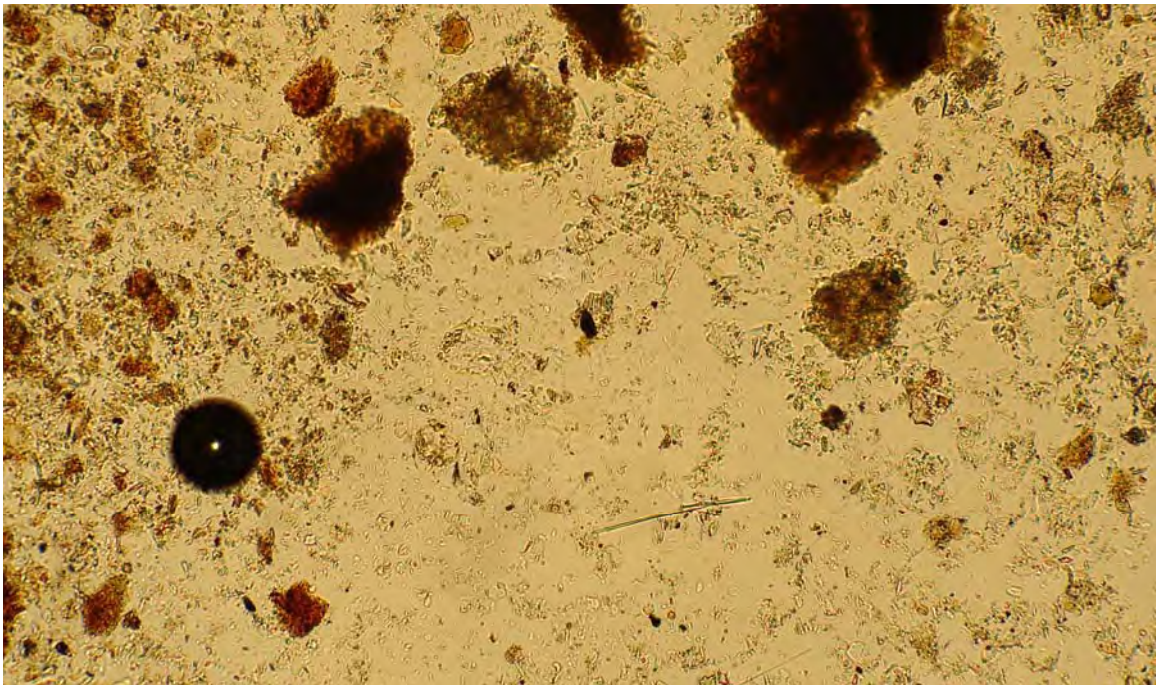
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

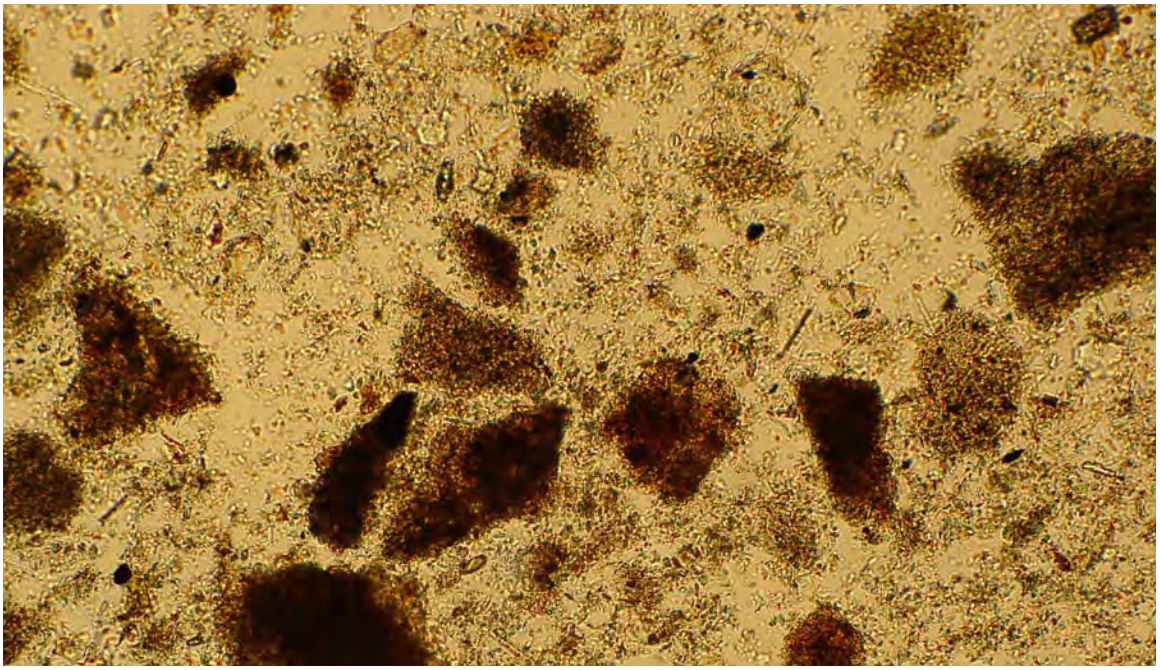




1.0% Libby Amphibole

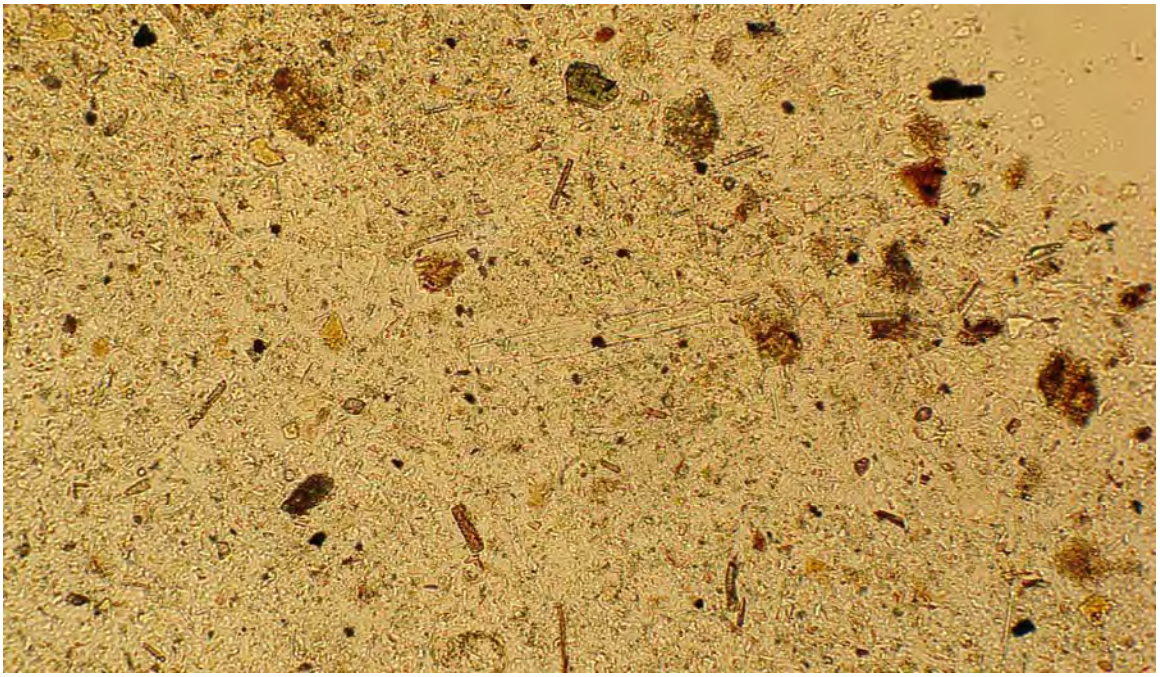
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

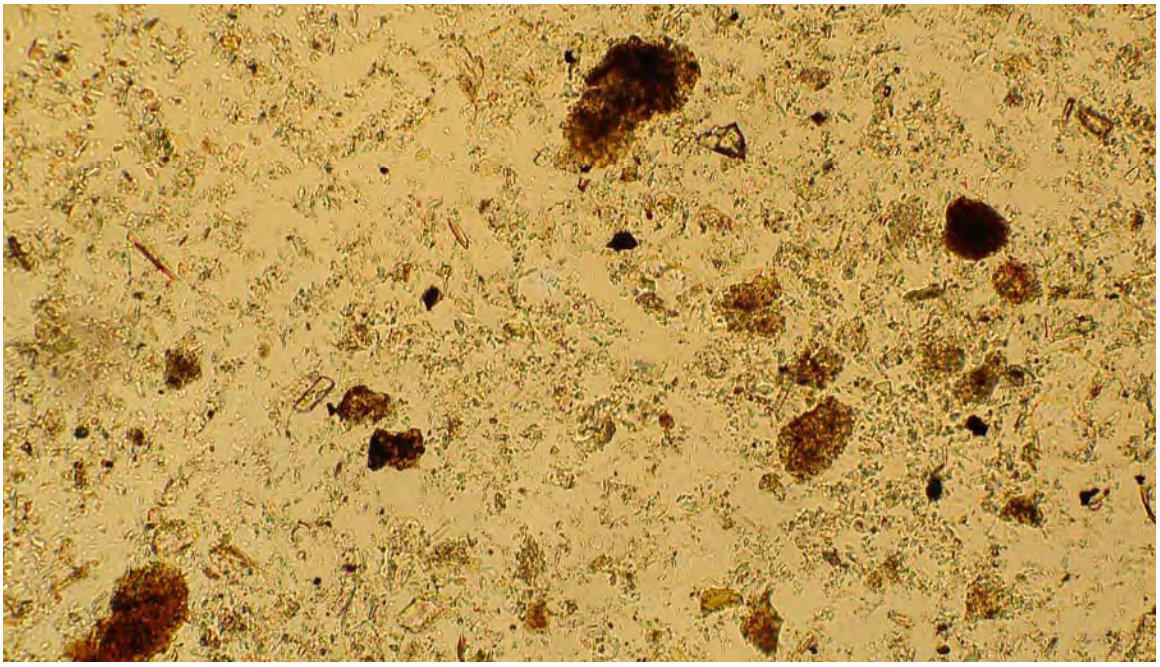




1.0% Libby Amphibole

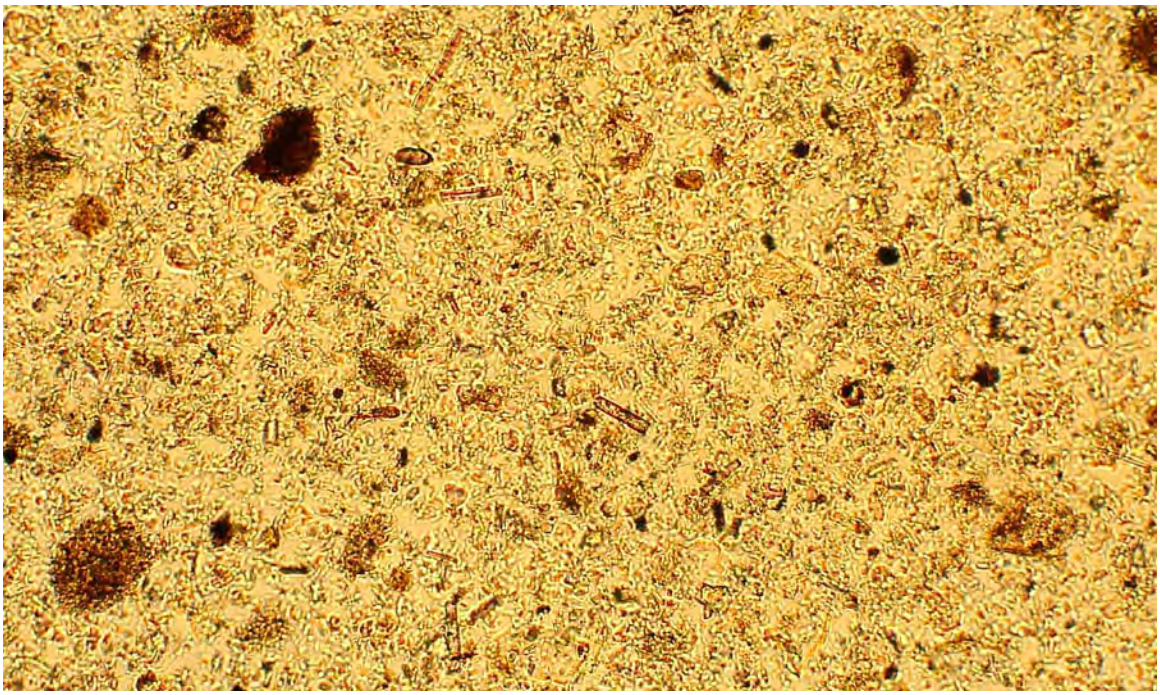
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

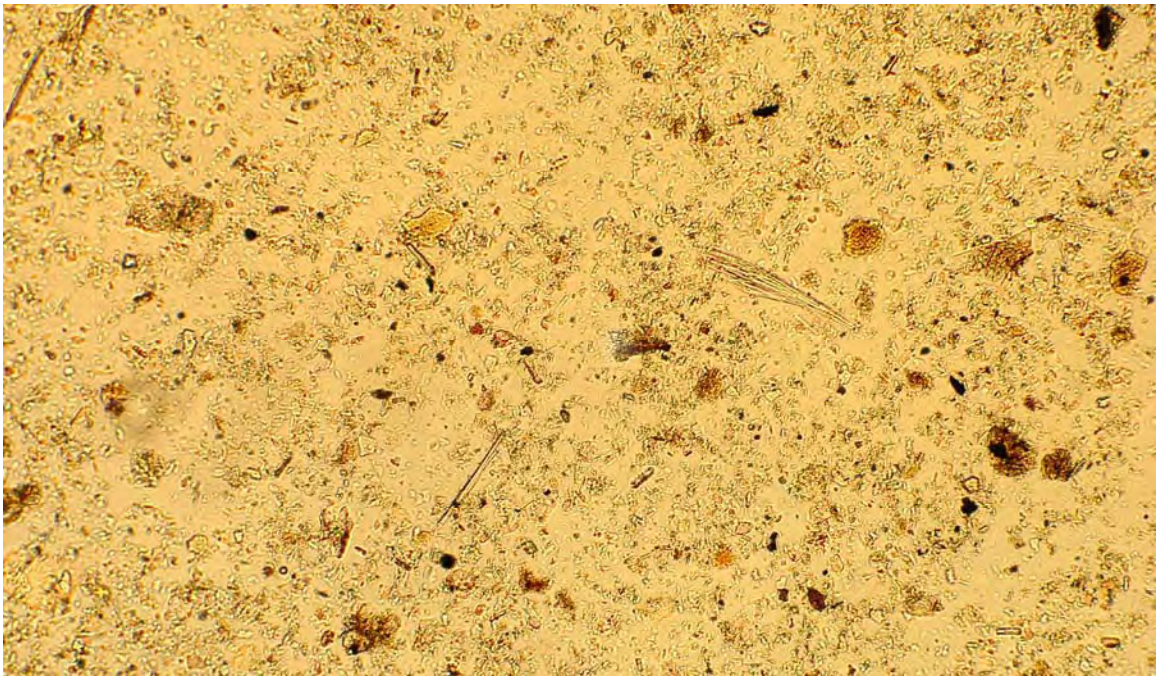




1.0% Libby Amphibole

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

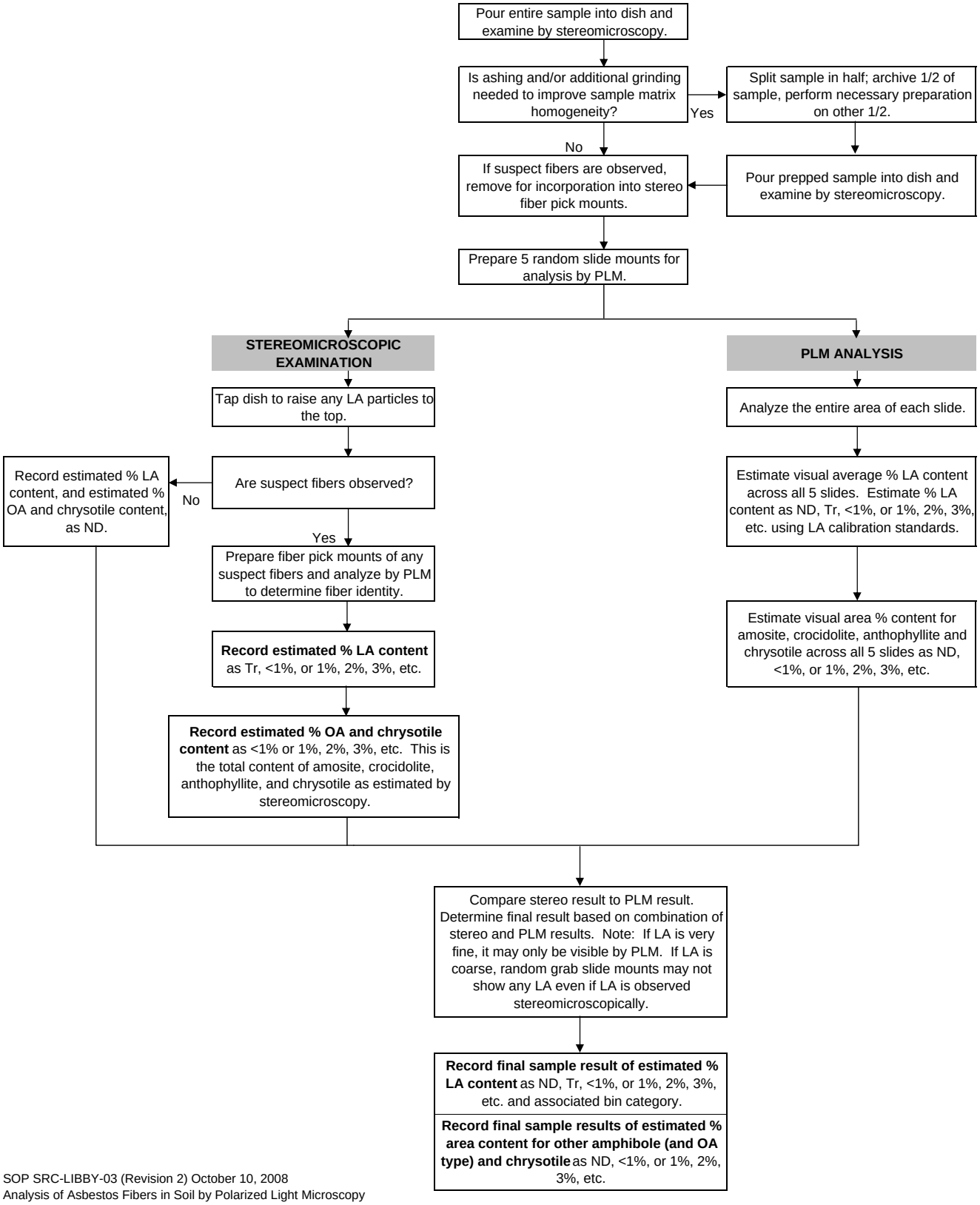




ATTACHMENT 8

Flow Chart for Determining Asbestos Content by Complementary Use of Stereomicroscopic Examination and PLM Visual Estimation

Flow Chart for Determining Asbestos Content by Complementary Use of Stereomicrosopic Examination and PLM Visual Estimation



Date: May 20, 2003 SOP No. SRC-LIBBY-01 (Revision 1)

Title: QUALITATIVE ESTIMATION OF ASBESTOS IN COARSE SOIL BY VISUAL EXAMINATION USING STEREOMICROSCOPY AND POLARIZED LIGHT MICROSCOPY

Author Sally M. L. Gibson Syracuse Research Corporation

SYNOPSIS: A standardized method is described for the examination of the coarse fraction (>1/4") of soil samples using stereomicroscopy and polarized light microscopy (PLM) to identify, segregate, and estimate the mass percent of asbestos in the sample matrix.

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8	[illegible]	5/20/03
Syracuse Research Corp.	W J Brattin	5/20/03

Revision	Date	Reason for Revision
0	11/12/02	--
1	05/20/03	Provided clarification on dealing with very small particles.

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized screening method for the visual examination of the coarse fraction of previously sieved soil samples for evidence of asbestos mineral content using stereomicroscopy with confirmation of asbestos content by polarized light microscopy (PLM). This SOP incorporates salient components of EPA Test Method 600/R-93/116 *Method for Determination of Asbestos in Bulk Building Materials* and National Institute of Occupational Safety and Health (NIOSH) Method 9002 *Asbestos (bulk) by PLM*, Issue 2.

This procedure will be used by employees of contractors/subcontractors supporting USEPA Region 8 projects and tasks for the Libby, Montana, site. Deviations from the procedure outlined in this document must be approved by the USEPA Region 8 Remedial Project Manager or Regional Chemist prior to initiation of sample analysis.

2.0 PREREQUISITE TRAINING

Visual examination will be performed according to this SOP by a laboratory accredited by the National Voluntary Laboratory Accreditation Program (NVLAP) and by analysts proficient either by education or experience in asbestos mineral identification by stereomicroscopy and PLM. Analyst familiarity with the procedural applications prescribed in EPA Test Method 600/R-93/116 and NIOSH Method 9002 is required.

Training as described in the Sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4, (CSS SQAPP [CDM 2002]) will be provided to laboratory personnel or laboratories with less than one year of project-specific experience by “mentors” from either Reservoir Environmental Services, Inc. or EMSL.

3.0 RESPONSIBILITIES

The CDM Laboratory Coordinator (LC) is responsible for overseeing the activities of the CDM Soil Preparation Laboratory and subcontracted laboratories performing sample analysis for the Libby, Montana, project. The LC is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the CSS SQAPP. It

is the responsibility of the LC to communicate with the project personnel and subcontracted laboratory regarding specific analysis objectives and anticipated situations that require any deviation from the CSS SQAPP SOPs. In addition, it is the responsibility of the LC to communicate the need for any deviations from this SOP with the CDM Project Manager, USEPA Region 8 personnel (Remedial Project Manager or Regional Chemist.)

Subcontracted laboratory analysts performing the visual examination are responsible for adhering to the applicable tasks outlined in this SOP and substantiating components of the reference procedures (EPA 1993; NIOSH 1994) with the modifications contained herein.

4. 0 EQUIPMENT

- Analytical balance - accurate to 0.01 g, range of 0.01 g to 1000 g (for weighing total sample)

- Analytical balance - accurate to 1 mg (for weighing asbestos)

- Traceable standards - major asbestos types

- Microscope - binocular stereomicroscope, 5-60X approximate magnification

- Microscope - polarized light, binocular or monocular with a cross hair reticle (or functional equivalent) and magnification of at least 8X
  - 10X, 20X, and 40X objectives
  - 360 degree rotatable stage
  - substage condenser with iris diaphragm
  - polarizer and analyzer which can be placed at 90 degrees to one another and calibrated relative to the cross-line reticle in the ocular
  - port for wave plates and compensators
  - wave retardation plate (Red I Compensator) with ~550 nanometer retardation and known slow and fast vibration directions

- Light Sources - incandescent or fluorescent

- Tweezers, dissecting needles, scalpels, probes, razor knives, etc. - standard sample manipulation instruments/tools

- Microscope slides and cover slips

- Refractive index liquids

- Pre-tared glassine paper, glass plates, weigh boats, petri dishes, watchglasses, etc. - laboratory sample containers

- HEPA-filtered or Class 1 biohazard hood negative pressure

- Three-ring binder book- binders will contain Microscopic Examination Logbook Sheets (Attachment 1)

5.0 METHOD

Soils from the Libby, Montana site will be dried, sieved, and prepared according to the most recent revision of SOP ISSI-LIBBY-01, Soil Sample Preparation. The coarse fraction of the soil sample is defined as that portion of the sample which does not pass through a 1/4" sieve. The coarse fraction will be weighed, placed in a zip-top plastic bag, and labeled as described in Camp, Dresser, and McKee (CDM) SOP 1-3 (with project-specific modifications). The samples will be packaged and shipped by the soil preparation laboratory as described in CDM SOP 2-1 (with project-specific modifications) and transferred to the laboratory via chain-of-custody procedures described in CDM SOP 1-2 (with project-specific modifications).

The following sections describe the stereomicroscopic and PLM examination. Materials tentatively characterized as asbestos by stereomicroscopy will be isolated and subjected to confirmation by PLM. The mass % of Libby amphibole asbestos, other amphibole asbestos, and chrysotile asbestos in the coarse soil fraction will be calculated from the mass of each asbestos type positively identified by PLM and the original sample weight. Figure 1 provides an overview of the process.

5.1 Stereomicroscopic Examination

The laboratory will receive the coarse fraction soil samples from the CDM Soil Preparation Laboratory. The entire sample will be weighed and placed in an appropriate container. The weight of each coarse sample will be recorded, along with the sample identification, on the Microscope Examination Logbook Sheet. The sample will be subject to stereomicroscopic examination and particle segregation as depicted Figure 1. The stereomicroscopic examination to identify and segregate asbestos includes:

- using multiple fields of view over the entire sample
- probing the sample by turning pieces over and breaking clumps where possible
- manipulating the sample using appropriate instruments/tools
- observing homogeneity, texture, friability, color and extent of any observed asbestos in the sample(s)

NOTE: Although the coarse fraction is prepared by sieving with a 1/4" screen, particles smaller than 1/4" may be present in the fraction due to adherence between coarse and fine particles. This may even include some very fine asbestos fibers. Because of the technical difficulty, the analyst should not attempt to physically segregate and weigh particles smaller than about 2-3 mm (1/10 inch). A particle this size is expected to have a mass of about 10-20 mg, which is less than 0.1% of a sample whose total mass is 25 grams. If no particles larger than 2-3 mm are present, this should be noted in the data sheet for each category of asbestos using the following code system:

- ND = No asbestos observed
- Tr = Trace levels of asbestos observed but not quantified

The weight fraction for any asbestos type marked “ND” or “Tr” in a given sample is not calculated and is left blank.

As the sample is examined, the analyst will continue segregation of the sample until the entire coarse soil fraction has been characterized as either “non-asbestos” or “tentatively identified asbestos.” The tentatively identified asbestos particles will be examined by PLM, as described below. The stereomicroscopist will initial and date the Microscopy Examination Logbook Sheet.

5.2 PLM

The coarse material tentatively identified as asbestos by stereomicroscopic examination will be subject to confirmation using PLM, as described in SOP SRC-LIBBY-03 (Revision 0) ("Analysis of Asbestos Fibers in Soil by Polarized Light Microscopy"). The PLM examination will be used to confirm that the particles tentatively classified as asbestos are actually asbestos, and will be assign each particles to one of three categories:

LA = Libby amphibole
OA = Other amphibole
C = Chrysotile

If OA is observed, the type of OA observed should be noted in the data sheet using the following code system:

- AMOS = Amosite
- ANTH = Anthophyllite
- CROC = Crocidolite
- UNK = Unknown

The total weight of each type of positively identified asbestos (LA, OA, C) will be determined and recorded on the Microscopic Examination Logbook Sheet, along with the analyst's initials and the date of the examination.

6.0 QUALITY ASSURANCE

Laboratories performing the examination must be accredited by NVLAP. "Calibration" should be verifiable for each microscopist in terms of project-specific training and the successful analysis of materials of known asbestos content (NVLAP test samples, in-house standards) similar to those anticipated to be observed in Libby, Montana soils. Additionally, references such as photographs of the asbestos minerals illustrating distinguishing properties should be available benchside during characterization.

Quality control samples as described in ISSI-LIBBY-01 (i.e., preparation duplicates) will not submitted for the coarse materials samples. The entire coarse fraction will be subject to examination.

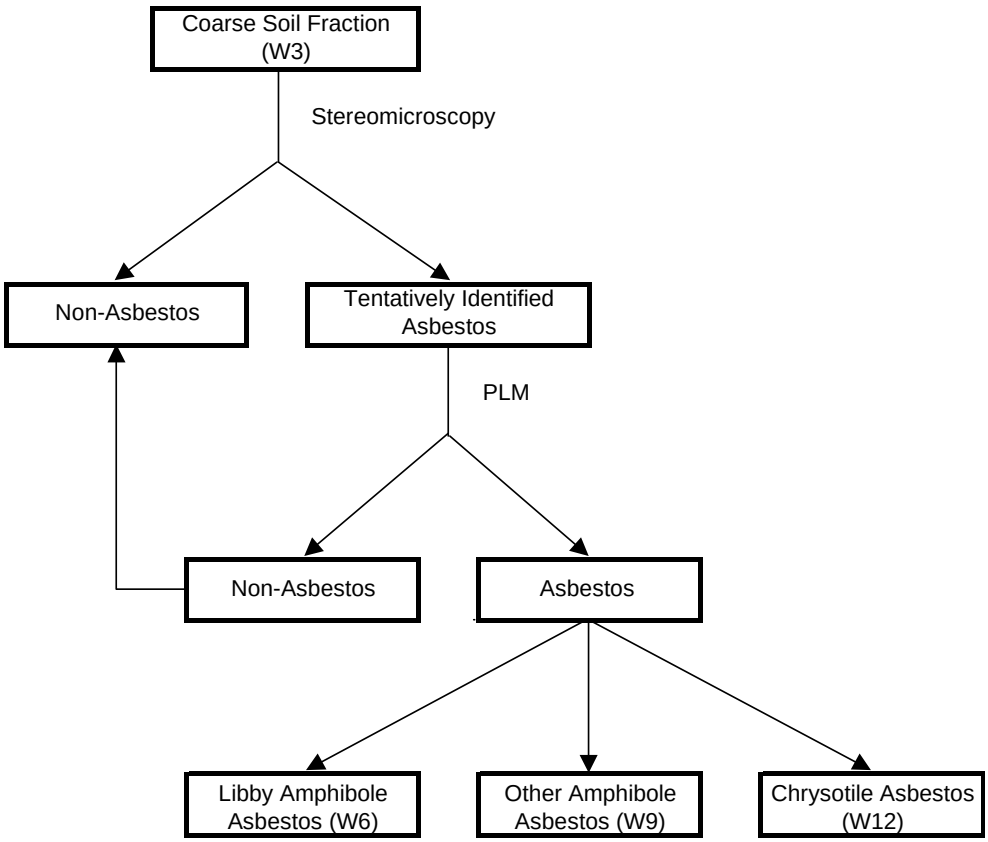
7.0 REFERENCES

CDM 2002. *Sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4.* 3282-116-PP-SAMP-14187. Camp, Dresser and McKee Denver, Colorado. April.

NIOSH 1994. National Institute of Occupational Safety and Health (NIOSH) Method 9002 *Asbestos (bulk) by PLM*, Issue 2.

USEPA 1993. *Method for Determination of Asbestos in Bulk Building Materials*. 600/R-93/116.

Figure 1. Overview of Sample Examination Process



W3 = Original coarse soil fraction mass (g)
W6 = If present in measurable quantities, mass (mg) of Libby amphibole
W9 = If present in measurable quantities, mass (mg) of other amphibole
W12 = If present in measurable quantities, mass (mg) of chrysotile

Codes used in the illustration (e.g., W3) correspond to Data Log Sheet

ATTACHMENT 1

MICROSCOPIC EXAMINATION LOGBOOK SHEET

See attached electronic file "SRC-LIBBY-01 Data sheet and EDD v6a.xls"

Example hard copy of data entry sheet shown on next page (for illustration purposes only).

Data Log Sheet v6 for SOP SRC-LIBBY-01

Stereomicroscopic and Gravimetric Analysis of Coarse Soil

Lab Name:	
SOP Version:	
Lab Job No.	

Page _____ of _____

Calculated automatically in the "Electronic Data Entry" form. Do not enter data here.

EPA Index ID	Index Suffix	Lab Job-Sample No.	Status	QA Type	Total Sample Weight (g)			Analysis Details		Mass of Asbestos Particles (mg)																Comments (see Notes below)
										Libby Amphibole (LA) Asbestos				Other Amphibole (OA) Asbestos					Chrysotile (C) Asbestos							
			1 = Analyzed 2 = Missing 3 = Contam 4 = Cancelled	(Not QA)	Tare Weight (g) Empty Container	Mass of Sample + Container (g)	Mass of Sample (g)	Analyst Initials	Analysis Date	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, UNK)	Tare Weight - Container (mg)	Mass of OA + Container (mg)	Mass (mg) OA	C Qual* (ND, Tr)	Tare Weight - Container (mg)	Mass of C + Container (mg)	Mass (mg) C	% LA	% OA	% C	

Notes:

*Qualifier codes: ND = No asbestos observed.
Tr = Trace levels observed but not quantified.

**OA Type codes: AMOS = Amosite
ANTH = Anthophyllite
CROC = Crocidolite
UNK = Unknown

Comment Codes (user-defined):

Date: March 5, 2008 SOP No. EPA-LIBBY-09 (rev 1)

Title: STANDARD OPERATING PROCEDURE FOR TEM DATA REVIEW AND DATA ENTRY VERIFICATION

Author Lynn Woodbury, Syracuse Research Corporation (SRC)

SYNOPSIS: This standard operating procedure provides a standardized method for review of raw TEM data and verification of entry of TEM results into the Libby2 Database. Steps included in this SOP are: a) selection of TEM analyses for review and verification, b) review of the original laboratory TEM bench sheets, and c) verification of the transfer of results from the bench sheets into the Libby2 Database. This method is applicable for use only at the Libby Superfund Site.

APPROVALS:

TEAM MEMBER	**SIGNATURE/TITLE**	**DATE**
EPA, Region 8		
SRC		

Revision	Date	Reason for Revision
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. ▪ Modify SOP to include a check of samples with errors to ensure that corrections were made properly. ▪ Change review time period from monthly to quarterly. ▪ Add consistency review of data entered in accord with LB-000066. ▪ Refer to LB-000016 (ISO) and LB-000031 (AHERA/ASTM) for appropriate aspect ratio recording rules.

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a standardized method for review of raw transmission electron microscopy (TEM) data and verification of entry of TEM results into the Libby2 Database. Steps included in this SOP are: a) selection of TEM analyses that will undergo a data consistency review and verification, b) performing a consistency review of the original laboratory TEM bench sheets to verify that TEM analysts working on the Libby project are performing analyses in accord with project-specific recording rules, and c) verifying the correct transfer of results from the bench sheets into the Libby2 Database.

2.0 PERSONNEL QUALIFICATIONS

Personnel performing data review and verification under this SOP must be skilled and/or trained in interpretation of raw data sheets and electronic data files in support of TEM analysis for the Libby Superfund Site. Personnel must be well-versed in TEM counting rules and Libby project-specific counting and recording rules in order to perform the required consistency reviews.

3.0 APPLICABILITY

A representative portion of TEM data, analyzed for the Libby Superfund Site, will be selected for review and verification to ensure consistency in data collection and data entry. The frequency of samples selected for review is discussed in subsequent sections.

4.0 SELECTION OF TEM RECORDS FOR REVIEW

The goals for selecting a representative subset of TEM results for review and verification are provided below. Selections should be made to ensure representation across several areas: 1) the fraction of total samples analyzed by TEM; 2) the types of programs (SAPs, QAPPs, etc.) carried out at the Site; 3) the laboratories performing TEM analysis.

> Total Samples. Over the course of the Libby project (that begins with the date of this approved SOP), a minimum of ten percent (10%) of all TEM analyses should be selected for review and verification. Samples will be selected in a manner that ensures representation across the different types of programs and the laboratories performing the TEM analysis.
>
> Types of Programs. If there are important differences in sampling and analysis protocols between sampling programs, data reviews and verifications will be stratified by program. At the request of EPA, the frequency of data review may be increased for specific programs of interest (i.e., investigative samples associated with ambient air monitoring, activity-based sampling, and cleanup efficacy evaluations). Of specific interest is ensuring reviews are stratified across programs that reflect differences in structure recording and/or counting rules.
>
> Laboratories performing TEM analysis. Data reviews and verifications will be performed for each laboratory participating in TEM analysis in support of the Site sampling programs.

Specific details for selecting TEM records for review are outlined below.

1. Interlab samples will be selected on a quarterly basis – 1st Quarter = January 1 - March 31, 2nd Quarter = April 1 - June 30, 3rd Quarter = July 1 - September 30, 4th Quarter = October 1 - December 31. At the beginning of each quarterly review period, compile a list of all TEM ISO 10312 and all TEM AHERA/ASTM samples for which new results were uploaded into the Libby2 Database in the preceding quarter (e.g., for the 1st

Quarter, specify a date range of January 1 - March 31). Samples will be selected for review separately for TEM ISO 10312 and AHERA/ASTM.

The Libby2 Database query will be based on the analysis upload date rather than the analysis date to ensure that analyses with an upload in a different quarter as the analysis date are not excluded. For example, consider the case where the TEM ISO 10312 analysis for sample X-12345 was performed on September 22 (in the 3rd Quarter) and the results were uploaded on October 3 (in the 4th Quarter). The selection query performed on October 1 for the 3rd Quarter results, if limited to all results analyzed from July 1 - September 30, would not capture the results for X-12345 because they had not yet been uploaded. The selection query performed on January 1 for the 4th Quarter results, if limited to all results analyzed from October 1 - December 31, would also not capture the results for sample X-12345 because the analysis date is outside of the specified range.

However, use of the analysis upload date has the potential to include both new analyses and corrections to older analyses resulting from earlier validation efforts. To avoid having to re-review analyses that have already been validated and corrected, only new analyses should be selected for review. To do this, the list of candidate samples will be compared to a running list of all previously validated samples[a]. Any samples that have been validated previously will be excluded from selection. In addition, samples that will be validated under other review efforts associated with specific investigations (e.g., ambient air) will also be excluded from selection.

2. A minimum of 10% of all TEM ISO 10312 and TEM AHERA/ASTM analyses will be selected for review each quarter. To the extent practical, these will be first stratified by analyst, with the number of samples from each analyst being in proportion to the total number of samples analyzed by each analyst. If there are important differences between sampling programs (e.g., differences in counting and/or recording protocols), samples will also be stratified by program. In addition, samples will be stratified according to detect/non-detect, with approximately 50% of the samples selected being detects, and 50% being non-detects. The following table illustrates the selection process:

	Analyzed			Selected		
Analyst	Detect	ND	Total	Detect	ND	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

	Goal	Actual
Total	58	59
Detect	29	29
Non-detect	29	30

In this example, there are a total of 581 new TEM ISO 10312 analyses available for the quarter (36 detects + 545 non-detects), analyzed by four analysts. Thus, the total number of TEM ISO 10312 analyses to be selected for review is 10% · 581 = 58.1 (rounded to 58). This total is to be split evenly between detects (29) and non-detects

[a] This running list of all validated samples will include validation efforts associated with specific investigations (e.g., 2006 demolition investigation, ambient air investigation, SQAPP sampling), as well as TEM validation efforts from preceding quarters.

(29). The number of detects and non-detects selected per analysis 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 = 29 · (14/36) = 11.3 (rounded to 11)
Number of non-detects = 29 · (112/545) = 5.9 (rounded to 6)

If an analyst has analyzed at least one sample in a category (detect or non-detect), the minimum number of samples 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 non-detects, the number to be selected (computed using the approach above) is:

Number of non-detects = 29 · (8/545) = 0.4 (rounded to 0)

In this case, the number selected 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 may tend to be higher in samples with one or more detected structures than in samples with no detected structures.

3. Stratify the list of newly uploaded samples according to program (if applicable), analyst, and detection status (detect, non-detect), and select the appropriate number of samples for each category at random.

4. Based on the samples selected for review, create a list of all the unique analytical laboratory jobs which will be needed to review the selected analyses. Submit the list of analytical laboratory jobs to EPA's project file manager (Volpe).

5. Volpe will provide SRC with electronic copies (as Adobe Acrobat PDFs) of the requested analytical laboratory jobs via CD, an FTP site, or another electronic transfer mechanism.

5.0 CONSISTENCY REVIEW OF LABORATORY BENCH SHEETS

The purpose of the consistency review is to inspect data entered on the laboratory bench sheets in order to identify the occurrence of any data omissions, apparent inconsistencies, or potential errors in structure.

5.1 Consistency Review Procedure for TEM ISO 10312

1. For each TEM ISO 10312 analysis to be reviewed, locate the original hand-written laboratory bench sheet(s) within the appropriate laboratory job.

2. Review the original hand-written laboratory bench sheets to determine if the raw structure data are recorded in accord with ISO 10312 counting rules (as modified in Libby Laboratory Modification LB-000016). The types of information that will be reviewed include:

   - The recorded structure types are consistent with the counting rules. Valid structure types include F, B, CC, CD, CF, CR, MC, MD, MF, and MR.

   - Disperse complex structures are broken down in accord with ISO 10312 counting rules and compact complex structures are not broken down. For example, a CD43 should provide 4 secondary structures, with 3 secondary structures greater than 5 um. In this example, the structure type for each of the recorded secondary structures should begin with the "C" prefix (e.g., CF, CB, CR).

- The primary and total columns have been populated with non-zero numbers for all countable structures and a zero for all non-countable structures.

- If recorded, all non-asbestos mineral (NAM) structures are identified as non-countable structures.

- All recorded fibers (F, CF, and MF) meet the appropriate aspect ratio requirement. [See Libby Laboratory Modification LB-000016 for aspect ratio recording rules for ISO 10312.]

- The mineral class is populated for all structures.

- If Libby Laboratory Modification LB-000066 is applicable, 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., < 3:1) are supported by recorded data.

- The stored values in the Libby2 Database for primary, total, structure type, length, width, and mineral class match the original bench sheet.

5.2 Consistency Review Procedure for TEM AHERA/ASTM

1. For each TEM AHERA/ASTM analysis to be reviewed, locate the original hand-written laboratory bench sheet(s) within the appropriate laboratory job.

2. Review the original hand-written laboratory bench sheets to determine if the raw structure data are recorded in accord with AHERA/ASTM counting rules (as modified in Libby Laboratory Modification LB-000031). The types of information that will 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.

    - 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 Libby Laboratory Modification LB-000031 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, 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.

- The stored values in the Libby 2 Database for primary, total, structure type, length, width, and mineral class match the original bench sheet.

5.3 Corrective Action

The data reviewer will prepare a list of any apparent inconsistencies, omissions, or other suspected errors. This list will be provided to EPA and to the Libby laboratory coordinator (CDM), who will forward the list to the appropriate laboratories and analysts for review and response.

At the laboratory, the analyst that performed the analysis and the Quality Assurance (QA) personnel that signed off on the TEM electronic data deliverable (EDD) will review the issues identified and determine which of the issues identified are authentic errors that require correction. All errors will be corrected and a revised TEM EDD and/or hard copy bench sheet will be submitted to the Libby laboratory coordinator (CDM). Each laboratory will provide re-training for analysts and QA reviewers, as needed, to minimize the occurrence of errors at the level of the bench sheet and EDD.

6.0 VERIFICATION OF DATA TRANSFER FROM BENCH SHEET TO DATABASE

6.1 Verification Procedure

The purpose of verification is to ensure that the data from the bench sheet have been transferred into the Libby 2 Database without error or omission. The following steps will be performed as part of the data verification procedure.

1. Compare the analysis-specific information provided in the Libby2 Database to the original lab job documentation (e.g., internal laboratory chain of custody, preparation logs, etc.). [Note: Whenever possible, verification will be performed against hand-written notations, NOT internal laboratory summary tables prepared from hand-written notes. Every attempt should be made to obtain the original hand-written notes. If laboratory summary tables are used instead of hand-written notes, this should be documented and specific rationale should be provided.] The following fields will be verified:

    - Analysis Method (TEM-ISO10312, TEM-AHERA, ASTM)
    - Analysis Date
    - Lab Name
    - Lab Job Number
    - Lab Sample Number
    - Preparation Method (Direct, Indirect, or Indirect with Ashing)
    - Filter Status (Analyzed, Overloaded, Damaged, Missing, Cancelled)
    - Primary Effective Filter Area (EFA, mm^2)
    - Secondary EFA (mm^2) [For indirect preparations only]
    - Grid Opening Area (Ago, mm^2) [b]
    - F-factor [For indirect preparations only, direct prep F-factor = 1]
    - Air Volume (L) or Sample Area (cm^2) [c]
    - Analysis Comments

[b] If the grid opening area is not within the expected range (0.005 - 0.015 mm^2), the value should be confirmed with the laboratory.
[c] To account for potential rounding issues, if the reported analysis air volume or sample area different from the value reported for the sample but is within 0.5% this will be noted in the summary report, but the value will be considered to be correct.

2. Verify the calculation of the F-factor for indirect preparations as follows:

   F-factor = Fraction of primary filter used · Volume of resuspension fluid applied to secondary filter / Total resuspension volume

3. Verify the amphibole sensitivity recorded in the Libby2 Database as follows:

   Air Sensitivity = EFA / (GOx · Ago · V · 1000 · F-factor)
   Dust Sensitivity = EFA / (GOx · Ago · SA · F-factor)

   where: EFA = Effective Filter Area (mm^2) [d]
   GOx = Grid Openings Counted for Libby amphibole
   Ago = Area of a Grid Opening (mm^2)
   V = Air Volume (L)
   SA = Dust Sample Area (cm^2)
   F-factor = indirect preparation dilution factor

4. Count the total number of unique grid openings evaluated in the original hand-written laboratory bench sheets, and compare to the number in the field titled "AnalysisGOCounted" in the Libby2 Database. [Note: If more than one analysis has been performed for the same sample, determine if the grid openings recorded in the second analysis were inclusive or exclusive of the grid openings in the first analysis. This check helps identify cases where an updated or revised EDD is added to the database as a new file rather than replacing (overwriting) an old file, thereby resulting in the duplication of some data.]

5. Using the original hand-written laboratory bench sheets, count the total number of "countable" Libby amphibole (LA) structures across all grid openings evaluated, and compare this number with the "binned" LA values stored in the Libby2 Database.

   - For ISO 10312 analyses, LA counts will be compared to Bin G for LA, which is equal to the total number of countable LA.

   - For AHERA/ASTM, LA counts will be compared to the "S<5um" and "S>5um" bins for LA.

6.2 Corrective Action

For each sample where an issue has been identified, the data reviewer will obtain a hard copy of the laboratory bench sheet. Based on a review of the bench sheet, each issue will be classified as either a) an omission or data entry error at the level of the EDD, or b) an error at the level of the data upload from the EDD into the Libby2 Database.

The data reviewer will prepare a list of any noted discrepancies or omissions for each sample, along with the apparent type of error. This list will be provided to EPA and to the Libby laboratory coordinator (CDM) for review and response.

In cases of apparent data omission or error at the level of the EDD preparation, the laboratory coordinator will contact the laboratory and identify the apparent error(s). At the laboratory, the individual responsible for data entry from the bench sheet into the EDD and the QA personnel that signed off on the EDD will review the issue and make corrections to the EDD as needed. If corrections are made, a revised EDD will be submitted to EPA's database manager for re-entry into the Libby 2 Database. Re-training of data entry and QA review personnel may be implemented, as needed.

[d] For direct preparations this will be the primary EFA. For indirect preparations, this will be the secondary EFA.

If the error is due to a database upload error, EPA's database manager (Volpe) will be contacted and notified of the issue. At Volpe, the TEM upload procedure will be reviewed to identify the source of the issue and modified to ensure that future TEM EDDs will be uploaded correctly. Depending on the nature of the issue, it may be necessary to identify other TEM analyses in the Libby 2 Database that would have been similarly impacted. Any potentially impacted TEM analyses should be removed from the Libby2 Database and re-uploaded after the upload procedure has been corrected.

7.0 CHECKING CORRECTIONS

Each quarter, the data reviewer will review the Libby2 Database and the lab job documentation to ensure that the appropriate corrections have been made for all analyses where one or more issues were identified during previous verification efforts. In cases where a revised EDD was uploaded into the database, the data reviewer will ensure that the incorrect EDD has been removed. A comprehensive summary of all issues and their status will be maintained by the data reviewer. As needed, this summary will be provided to EPA's database manager and the Libby laboratory coordinator for follow-up.

8.0 REPORTING

The data reviewer will prepare a report which summarizes the results of the consistency review and data verification for the sample set and identifies areas for improvement. Attachment A provides an example of this report. As seen, this report includes a detailed summary of the consistency review and data verification findings, and includes a summary of the potential implications of the review and verification findings on the data quality and use of the TEM analyses in the Libby2 Database. This report will also provide copies of all electronic spreadsheets generated which track any identified discrepancies and the resolution status of each issue.

Based on the results of the review and verification, EPA may choose to modify (either increase or decrease) the frequency of TEM samples selected for review and verification and/or the selection/review/verification process.

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). 2003. Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations. ASTM D 5755-03. American Society for Testing and Materials. October 2003.

International Organization for Standardization (ISO). 1995. Ambient Air – Determination of asbestos fibres – Direct-transfer transmission electron microscopy method. ISO 10312:1995(E).

ATTACHMENT A

EXAMPLE OF TEM CONSISTENCY REVIEW AND DATA TRANSFER VERIFICATION REPORT

TEM CONSISTENCY REVIEW AND DATA TRANSFER VERIFICATION REPORT

Date: _____ Prepared by: _____

Reporting Date Range: _____

SUMMARY OF FINDINGS AND DATA QUALITY IMPLICATIONS

_____

_____

_____

_____

_____

_____

_____

Recommendations for future review and verification: _____

_____

_____

_____

_____

TEM-ISO 10312 SELECTION AND CONSISTENCY REVIEW RESULTS

Summary of available analyses for date range specified –

Analyst, Lab	Number of TEM-ISO 10312 Analyses			Number of 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 guidance

_____ 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 aspect ratio criteria (LB-000016)

_____ Mineral class designation is missing or inconsistent

_____ Structure comments are inconsistent with LB-000066

_____ Structure comments are inconsistent with recorded data

_____ Structure attributes in the database do not match the bench sheet

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

Summary of available analyses for date range specified –

Analyst, Lab	Number of TEM-AHERA/ASTM Analyses			Number of 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 AHERA/ASTM guidance

_____ Total column is not populated correctly

_____ NAM structures are recorded and not identified as non-countable

_____ Fibers recorded as countable do not meet aspect ratio criteria (LB-000031)

_____ Recorded dimensions for matrices are matrix dimensions not protrusion dimensions

_____ Mineral class designation is missing or inconsistent

_____ Structure comments are inconsistent with LB-000066

_____ Structure comments are inconsistent with recorded data

_____ Structure attributes in the database do not match the bench sheet

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)

Types of data transfer issues identified:

_____ Incorrect/missing information on analysis details (e.g., lab job number, analysis date, filter status)

_____ F-factor calculation is incorrect or inputs are missing

_____ Air volume or dust area 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 LA 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: _____

_____

_____

_____

ISSUE RESOLUTION AND STATUS

_____

_____

_____

_____

_____

_____

[1] Only those analyses that have passed the bench sheet consistency review are included in the data transfer verification.

Polarized Light Microscopy (PLM) Validation
Standard Operating Procedure

1. The validator will locate the appropriate laboratory job package to be validated on the FTP site. Laboratory jobs will be organized by laboratory name and job number (e.g., "RESI_123569.pdf").

2. Each validator will create a hard copy of the data validation spreadsheets provided by SRC for the sample Index IDs to be validated within the laboratory job. As necessary, expand the column width and/or allow the text to wrap for description and comment fields.

3. The validator will verify the analysis-specific information provided in the spreadsheet against the original lab job documentation (e.g., internal laboratory chain of custody, preparation logs, etc.). [Note: Whenever possible, validation 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 Errata Summary Sheets (provided as Attachment 1).] The following fields will need to be validated:
    - Analysis Method (should be PLM NIOSH 9002)
    - Analysis Date
    - Lab Name
    - Lab Job Number
    - Lab Sample Number
    - Index ID Suffix (if applicable)
    - Analysis Comments[1]

4. Based on the original laboratory results, the validator will compare the reported PLM results for Libby amphibole (LA), reported as Tremolite/Actinolite, with the column titled "9002 TREM-ACTN (%)" in the data validation spreadsheet. For PLM NIOSH 9002, results should be reported as either non-detect (ND), Trace (Tr or <1%), or detect (detected at % level indicated).

5. For each field to be validated, if the data in the spreadsheet matches the information in the laboratory job documentation, mark the appropriate spreadsheet field with a check mark in red. If the spreadsheet does not match the laboratory job documentation, circle and highlight in yellow the incorrect entry on the spreadsheet, then note the Index ID and the specific change to be implemented on the Errata Summary Sheet (see Attachment 1). For example, "1-12345" will go in the Index ID column and "Change from ND to <1%" will go in the Description column. Place the appropriate Errata # next to the highlighted field. [Note: To avoid duplication of Errata #s within a laboratory job, one Errata Summary should be used for each analytical job number. If the validation process takes more than one day, the validator should continue to add to the Errata Summary Sheet until the job is complete.]

6. At the end of each day (or more frequently, as necessary), the Errata Summary Sheet should be clipped to the hard copy data validation spreadsheet for those samples which were validated. If only a portion of the laboratory job was completed, the validator should make a copy of the data validation spreadsheet and Errata Summary Sheet(s), to allow for continued validation on the next day. This data validation package will be sent via courier to the SRC data validation coordinator (DVC) for review.

[1] The analysis comments field is to be reviewed for any applicable information regarding analysis status, laboratory quality control (QC) information, etc.

7. At the beginning of each day (or more frequently, as necessary), the SRC DVC will review the data validation packages for correctness and clarity. All Errata Summary Sheet entries will be reviewed and initialed by the SRC DVC. If any deficiencies are noted, the SRC DVC will re-train validation personnel as necessary. If the SRC DVC disagrees with the discrepancy noted on the Errata Summary Sheet, a diagonal line will be placed through the entry on the Errata Summary Sheet along with the date and the SRC DVC initials.

8. In addition to verifying all issues noted in the Errata Summary Sheet, the SRC DVC will perform a check of every 5th Index ID for each validator to ensure that any potential issues have been identified correctly. If any deficiencies are noted, the SRC DVC will re-train validation personnel as necessary. At the discretion of the SRC DVC, the frequency of this verification may be reduced over time if no deficiencies are noted.

9. As needed, the SRC DVC will provide copies of the Errata Summary Sheets to the Volpe Center DVC so that daily corrections may be made to the Libby 2 Database. The date the appropriate correction has been made in the database will be noted on the Errata Summary Sheet by the Volpe Center DVC.

10. After the validation has been completed, all hard copy data validation spreadsheets and Errata Summary Sheets will be scanned and provided to EPA as an electronic deliverable. Hard copies of all original data validation spreadsheets and Errata Summary Sheets will be provided to the Volpe Center for storage in the project file.

INTERNATIONAL STANDARD

ISO 10312

First edition
1995-05-01

Ambient air — Determination of asbestos fibres — Direct-transfer transmission electron microscopy method

Air ambiant — Détermination des fibres d'amiante — Méthode de microscopie électronique à transmission directe

Reference number
ISO 10312:1995(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10312 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 3, *Ambient atmospheres*.

Annexes A, B, C, D, E and F form an integral part of this International Standard. Annexes G, H and J are for information only.

Introduction

This International Standard is applicable to the determination of airborne asbestos in a wide range of ambient air situations, including the interior atmospheres of buildings, and for detailed evaluation of any atmosphere in which asbestos structures are likely to be present. Because the best available medical evidence indicates that the numerical fibre concentration and the fibre sizes are the relevant parameters for evaluation of the inhalation hazards, a fibre counting technique is the only logical approach. Most fibres in ambient atmospheres are not asbestos, and therefore there is a requirement for fibres to be identified. Many airborne asbestos fibres in ambient atmospheres have diameters below the resolution limit of the optical microscope. This International Standard is based on transmission electron microscopy, which has adequate resolution to allow detection of small fibres and is currently the only technique capable of unequivocal identification of the majority of individual fibres of asbestos. Asbestos is often found, not as single fibres, but as very complex, aggregated structures which may or may not be also aggregated with other particles. The fibres found suspended in an ambient atmosphere can often be identified unequivocally, if a sufficient measurement effort is expended. However, if each fibre were to be identified in this way, the analysis would become prohibitively expensive. Because of instrumental deficiencies or because of the nature of the particulate, some fibres cannot be positively identified as asbestos, even though the measurements all indicate that they could be asbestos. Subjective factors therefore contribute to this measurement, and consequently a very precise definition of the procedure for identification and enumeration of asbestos fibres is required. The method specified in this International Standard is designed to provide the best description possible of the nature, numerical concentration, and sizes of asbestos-containing particles found in an air sample. This International Standard is necessarily complex, because the instrumental techniques used are complex, and also because a very detailed and logical procedure must be specified to reduce the subjective aspects of the measurement. The method of data recording specified in this International Standard is designed to allow re-evaluation of the structure counting data as new medical evidence becomes available. All of the feasible specimen preparation techniques result in some modification of the airborne particulate. Even the collection of particles from a three-dimensional airborne dispersion onto a two-dimensional filter surface can be considered a modification of the particulate, and some of the particles in most samples are modified by the specimen preparation procedures. However, the procedures specified in this International Standard are designed to minimize the disturbance of the collected particulate material, and the effect of those disturbances which do occur can be evaluated.

This International Standard describes the method of analysis for a single air filter. However, one of the largest potential errors in characterizing asbestos in ambient atmospheres is associated with the variability between filter samples. For this reason, it is necessary to design a replicate sampling scheme in order to determine this International Standard's accuracy and precision.

Ambient air — Determination of asbestos fibres — Direct-transfer transmission electron microscopy method

1 Scope

1.1 Substance determined

This International Standard specifies a reference method using transmission electron microscopy for the determination of the concentration of asbestos structures in ambient atmospheres and includes measurement of the lengths, widths and aspect ratios of the asbestos structures. The method allows determination of the type(s) of asbestos fibres present. The method cannot discriminate between individual fibres of the asbestos and non-asbestos analogues of the same amphibole mineral.

1.2 Type of sample

The method is defined for polycarbonate capillary-pore filters or cellulose ester (either mixed esters of cellulose or cellulose nitrate) filters through which a known volume of air has been drawn. The method is suitable for determination of asbestos in both exterior and building atmospheres.

1.3 Measuring range

The range of concentration which can be determined is 50 structures/mm^2 to 7 000 structures/mm^2 on the filter. The air concentrations represented by these values are a function of the volume of air sampled. There is no lower limit to the dimensions of asbestos fibres which can be detected. In practice, microscopists vary in their ability to detect very small asbestos fibres. Therefore, a minimum length of 0,5 µm has been defined as the shortest fibre to be incorporated in the reported results.

1.4 Limit of detection

The limit of detection theoretically can be lowered indefinitely by filtration of progressively larger volumes of air and by extending the examination of the specimens in the electron microscope. In practice, the lowest achievable limit of detection for a particular area of TEM specimen examined is controlled by the total suspended particulate concentration.

For total suspended particulate concentrations of approximately 10 µg/m^3, corresponding to clean, rural atmospheres, and assuming filtration of 4 000 litres of air, an analytical sensitivity of 0,5 structure/l can be obtained, equivalent to a limit of detection of 1,8 structure/l, if an area of 0,195 mm^2 of the TEM specimens is examined. If higher total suspended particulate concentrations are present, the volume of air filtered must be reduced in order to maintain an acceptable particulate loading on the filter, leading to a proportionate increase in the analytical sensitivity.

Where this is the case, lower limits of detection can be achieved by increasing the area of the TEM specimens that is examined. In order to achieve lower limits of detection for fibres and bundles longer than 5 µm, and for PCM equivalent fibres, lower magnifications are specified which permit more rapid examination of larger areas of the TEM specimens when the examination is limited to these dimensions of fibre. The direct analytical method cannot be used if the general particulate loading of the sample collection filter exceeds approximately 10 µg/cm^2 of filter surface, which corresponds to approximately 10 % coverage of the collection filter by particulate. If the total suspended particulate is largely organic material, the limit of detection can be lowered significantly by using an indirect preparation method.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4225:1994, *Air quality — General aspects — Vocabulary.*

ISO 4226:1993, *Air quality — General aspects — Units of measurement.*

ISO Standard Handbook No. 2:1993, *Quantities and units.*

ISO Standard Handbook No. 3:1989, *Statistical Methods.*

3 Definitions

For the purposes of this International Standard, the following definitions apply (see also ISO 4225).

3.1 acicular: The shape of an extremely slender crystal with cross-sectional dimensions which are small relative to its length, i.e. needle-like.

3.2 amphibole: A group of rock-forming ferromagnesium silicate minerals, closely related in crystal form and composition, with the nominal formula:

$$A_0 \text{ or } {}_1B_2C_5T_8O_{22}(OH,F,Cl)_2$$

where

A = K, Na

B = Fe^{2+}, Mn, Mg, Ca, Na

C = Al, Cr, Ti, Fe^{3+}, Mg, Fe^{2+}

T = Si, Al, Cr, Fe^{3+}, Ti

In some varieties of amphibole, these elements can be partially substituted by Li, Pb or Zn. Amphibole is characterized by a cross-linked double chain of Si-O tetrahedra with a silicon:oxygen ratio of 4:11, by columnar or fibrous prismatic crystals and by good prismatic cleavage in two directions parallel to the crystal faces and intersecting at angles of about 56° and 124°.

3.3 amphibole asbestos: Amphibole in an asbestiform habit.

3.4 analytical sensitivity: The calculated airborne asbestos structure concentration in asbestos structures/litre, equivalent to counting of one asbestos structure in the analysis. The method in this International Standard does not specify an analytical sensitivity.

3.5 asbestiform: A specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility.

3.6 asbestos: A term applied to a group of silicate minerals belonging to the serpentine and amphibole groups which have crystallized in the asbestiform habit, causing them to be easily separated into long, thin, strong fibres when crushed or processed. The Chemical Abstracts Service Registry Numbers of the most common asbestos varieties are: chrysotile (12001-29-5), crocidolite (12001-28-4), grünerite asbestos (amosite) (12172-73-5), anthophyllite asbestos (77536-67-5), tremolite asbestos (77536-68-6) and actinolite asbestos (77536-66-4).

3.7 asbestos structure: A term applied to any connected or overlapping grouping of asbestos fibres or bundles, with or without other particles.

3.8 aspect ratio: The ratio of length to width of a particle.

3.9 blank: A structure count made on TEM specimens prepared from an unused filter, to determine the background measurement.

3.10 camera length: The equivalent projection length between the specimen and its electron diffraction pattern, in the absence of lens action.

3.11 chrysotile: A fibrous mineral of the serpentine group which has the nominal composition

$$Mg_3Si_2O_5(OH)_4$$

Most natural chrysotile deviates little from this nominal composition. In some varieties of chrysotile, minor substitution of silicon by Al^{3+} may occur. Minor substitution of magnesium by Al^{3+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Mn^{2+} and Co^{2+} may also be present. Chrysotile is the most prevalent type of asbestos.

3.12 cleavage: The breaking of a mineral along one of its crystallographic directions.

3.13 cleavage fragment: A fragment of a crystal that is bounded by cleavage faces.

3.14 cluster: A structure in which two or more fibres, or fibre bundles, are randomly oriented in a connected grouping.

3.15 d-spacing: The distance between identical adjacent and parallel planes of atoms in a crystal.

3.16 electron diffraction: A technique in electron microscopy by which the crystal structure of a specimen is examined.

3.17 electron scattering power: The extent to which a thin layer of substance scatters electrons from their original directions.

3.18 energy dispersive X-ray analysis: Measurement of the energies and intensities of X-rays by use of a solid state detector and multichannel analyser system.

3.19 eucentric: The condition when the area of interest of an object is placed on a tilting axis at the intersection of the electron beam with that axis and is in the plane of focus.

3.20 field blank: A filter cassette which has been taken to the sampling site, opened, and then closed. Such a filter is used to determine the background structure count for the measurement.

3.21 fibril: A single fibre of asbestos, which cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearances.

3.22 fibre: An elongated particle which has parallel or stepped sides. For the purposes of this International Standard, a fibre is defined to have an aspect ratio equal to or greater than 5:1 and a minimum length of 0,5 µm.

3.23 fibre bundle: A structure composed of parallel, smaller diameter fibres attached along their lengths. A fibre bundle may exhibit diverging fibres at one or both ends.

3.24 fibrous structure: A fibre, or connected grouping of fibres, with or without other particles.

3.25 habit: The characteristic crystal growth form, (or combination of these forms), of a mineral, including characteristic irregularities.

3.26 limit of detection: The calculated airborne asbestos structure concentration in structures per litre, equivalent to counting 2,99 asbestos structures in the analysis.

3.27 matrix: A structure in which one or more fibres, or fibre bundles, touch, are attached to, or partially concealed by, a single particle or connected group of nonfibrous particles.

3.28 Miller index: A set of either three or four integer numbers used to specify the orientation of a crystallographic plane in relation to the crystal axes.

3.29 PCM equivalent fibre: A fibre of aspect ratio greater than or equal to 3:1, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm.

3.30 PCM equivalent structure: A fibrous structure of aspect ratio greater than or equal to 3:1, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm.

3.31 primary structure: A fibrous structure that is a separate entity in the TEM image.

3.32 replication: A procedure in electron microscopy specimen preparation in which a thin copy, or replica, of a surface is made.

3.33 selected area electron diffraction: A technique in electron microscopy in which the crystal structure of a small area of a sample is examined.

3.34 serpentine: A group of common rock-forming minerals having the nominal formula

$$Mg_3Si_2O_5(OH)_4$$

3.35 structure: A single fibre, fibre bundle, cluster or matrix.

3.36 twinning: The occurrence of crystals of the same species joined together at a particular mutual orientation, such that the relative orientations are related by a definite law.

3.37 unopened fibre: An asbestos fibre bundle of large diameter which has not been separated into its constituent fibrils or fibres.

3.38 zone-axis: The line or crystallographic direction through the centre of a crystal which is parallel to the intersection edges of the crystal faces defining the crystal zone.

4 Principle

A sample of airborne particulate is collected by drawing a measured volume of air through either a

capillary-pore polycarbonate membrane filter of maximum pore size 0,4 µm or a cellulose ester (either mixed esters of cellulose or cellulose nitrate) membrane filter of maximum pore size 0,45 µm by means of a battery-powered or mains-powered pump. TEM specimens are prepared from polycarbonate filters by applying a thin film of carbon to the filter surface by vacuum evaporation. Small areas are cut from the carbon-coated filter, supported on TEM specimen grids, and the filter medium is dissolved away by a solvent extraction procedure. This procedure leaves a thin film of carbon which bridges the openings in the TEM specimen grid, and which supports each particle from the original filter in its original position. Cellulose ester filters are chemically treated to collapse the pore structure of the filter, and the surface of the collapsed filter is then etched in an oxygen plasma to ensure that all particles are exposed. A thin film of carbon is evaporated onto the filter surface and small areas are cut from the filter. These sections are supported on TEM specimen grids and the filter medium is dissolved away by a solvent extraction procedure.

The TEM specimen grids from either preparation method are examined at both low and high magnifications to check that they are suitable for analysis before carrying out a quantitative structure count on randomly-selected grid openings. In the TEM analysis, electron diffraction (ED) is used to examine the crystal structure of a fibre, and its elemental composition is determined by energy dispersive X-ray analysis (EDXA). For a number of reasons, it is not possible to identify each fibre unequivocally, and fibres are classified according to the techniques which have been used to identify them. A simple code is used to record, for each fibre, the manner in which it was classified. The fibre classification procedure is based on successive inspection of the morphology, the electron diffraction pattern for a selected area, and the qualitative and quantitative energy dispersive X-ray analyses. Confirmation of the identification of chrysotile is done only by quantitative ED, and confirmation of amphibole is done only by quantitative EDXA and quantitative zone axis ED.

In addition to isolated fibres, ambient air samples often contain more complex aggregates of fibres, with or without other particles. Some particles are composites of asbestos fibres with other materials. Individual fibres and structures that are more complex are referred to as "asbestos structures". A coding system is used to record the type of fibrous structure, and to provide the optimum description of each of these complex structures. The two codes remove the requirement to interpret the structure counting data from the microscopist, and allow this evaluation to be made later without the requirement for re-examination of the TEM specimens. Several levels of analysis are specified, the higher levels providing more rigorous approach to the identification of fibres. The procedure permits a minimum required fibre identification criterion to be defined on the basis of previous knowledge, or lack of it, about the particular sample. Attempts are then made to achieve this minimum criterion for each fibre, and the degree of success is recorded for each fibre. The lengths and widths of all classified structures and fibres are recorded. The number of asbestos structures found on a known area of the microscope sample, together with the equivalent volume of air filtered through this area, is used to calculate the airborne concentration in asbestos structures/litre of air.

5 Symbols of units and abbreviations

5.1 *Symbols of units* (*see also ISO 4226 and* ISO No. 2)

eV = electron volt

kV = kilovolt

l/min = litres per minute

µg = microgram (10^{-6} gram)

µm = micrometre (10^{-6} metre)

nm = nanometre (10^{-9} metre)

W = watt

5.2 Abbreviations

DMF	Dimethylformamide
DE	Electron diffraction
EDXA	Energy dispersive X-ray analysis
FWHM	Full width, half maximum
HEPA	High efficiency particle absolute
MEC	Mixed esters of cellulose
PC	Polycarbonate
PCM	Phase contrast optical microscopy
SAED	Selected area electron diffraction
SEM	Scanning electron microscope
STEM	Scanning transmission electron microscop
TEM	Transmission electron microscope

UICC Union Internationale Contre le Cancer

6 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and water (6.1).

WARNING — Use the reagents in accordance with the appropriate health and safety regulations.

6.1 water, fibre-free.

A supply of freshly distilled, fibre-free water, or another source of fibre-free, pyrogen-free water shall be used.

6.2 Chloroform, analytical grade, distilled in glass, preserved with 1 % (*V/V*) ethanol.

6.3 1-Methyl-2-pyrrolidone.

6.4 Dimethylformamide.

6.5 Glacial acetic acid.

6.6 Acetone.

7 Apparatus

7.1 Air sampling — Equipment and consumable supplies

7.1.1 Filter cassette

Field monitors, comprising 25 mm to 50 mm diameter three-piece cassettes, with cowls which project less than 2 cm in front of the filter surface shall be used for sample collection. The cassette shall be loaded with either a capillary pore polycarbonate filter of maximum pore size 0,4 µm or an MEC or cellulose nitrate filter of maximum pore size 0,45 µm. Either type of filter shall be backed by a 5 µm pore size MEC or cellulose nitrate filter, and supported by a cellulose back-up pad. When the filters are in position, an elastic cellulose band or adhesive tape shall be applied to prevent air leakage. Suitable precautions shall be taken to ensure that the filters are tightly clamped in the assembly, so that significant air leakage around the filter cannot occur.

Representative filters from the filter lot shall be analysed as specified in 9.7 for the presence of asbestos structures before any are used for air sample collection.

7.1.2 Sampling pump

The sampling pump shall be capable of a flow-rate sufficient to achieve the desired analytical sensitivity. The face velocity through the filter shall be between 4,0 cm/s and 25,0 cm/s. The sampling pump used shall provide a non-fluctuating airflow through the filter, and shall maintain the initial volume flow-rate to within ± 10 % throughout the sampling period. A constant flow or critical orifice controlled pump meets these requirements. Flexible tubing shall be used to connect the filter cassette to the sampling pump. A means for calibration of the flow-rate of each pump is also required.

7.1.3 Stand

A stand shall be used to hold the filter cassette at the desired height for sampling, and shall be isolated from the vibrations of the pump (7.1.2).

7.1.4 Variable area flowmeter

A calibrated variable are a flowmeter with a range of approximately 1 l/min to 10 l/min is required for calibration of the air sampling system.

The variable area flowmeter shall be cleaned before use to avoid transfer of asbestos contamination from the flowmeter to the sample being collected.

7.2 Specimen preparation laboratory

Asbestos, particularly chrysotile, is present in varying quantities in many laboratory reagents. Many building materials also contain significant amounts of asbestos or other mineral fibres which may interfere with the analysis if they are inadvertently introduced during preparation of specimens. It is most important to ensure that, during preparation, contamination of TEM specimens by any extraneous asbestos fibres is minimized. All specimen preparation steps shall therefore be performed in an environment where contamination of the sample is minimized. The primary requirement of the sample preparation laboratory is that a blank determination shall yield a result which will meet the requirements specified in 9.7. A minimum facility considered suitable for preparation of TEM specimens is a laminar flow hood with positive pressure. However, it has been established that work practices in specimen preparation appear to be more important than the tape of clean handling facilities in use. Preparation of samples shall be carried out only after acceptable blank values have been demonstrated.

NOTE 1 It is recommended that activities involving manipulation of bulk asbestos samples not be performed in the

same area as TEM specimen preparation, because of the possibilities of contaminating the TEM specimens.

7.3 Equipment for analysis

7.3.1 Transmission electron microscope

A TEM operating at an accelerating potential of 80 kV to 120 kV, with a resolution better than 1,0 nm, and a magnification range of approximately × 300 to × 100 000 shall be used. The ability to obtain a direct screen magnification of about × 100 000 is necessary for inspection of fibre morphology; this magnification may be obtained by supplementary optical enlargement of the screen image by use of a binocular if it cannot be obtained directly. It is also required that the viewing screen of the microscope be calibrated such that the lengths and widths of fibre images down to 1 mm width can be measured in increments of 1 mm, regardless of image orientation. This requirement is often fulfilled through the use of a fluorescent screen with calibrated gradations in the form of circles, as shown in figure 1.

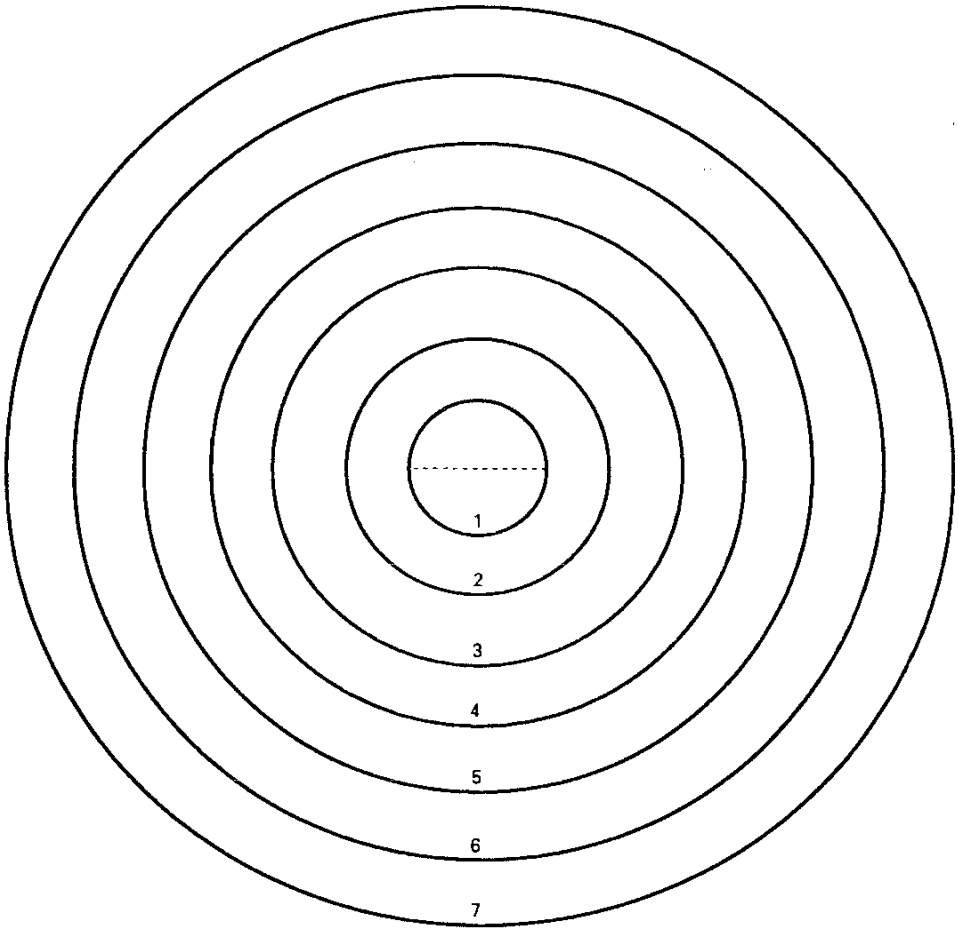


Figure 1 — Example of calibration markings on TEM viewing screen

For Bragg angles less than 0,01 rad, the TEM shall be capable of performing ED from an area of 0,6 μm^2 or less, selected from an in-focus image at a screen magnification of × 20 000. This performance requirement defines the minimum separation between particles at which independent ED patterns can be obtained from each particle. If SAED is used, the performance of a particular instrument may normally be calculated using the following equation

$$A = 0{,}785\,4 \times \left(\frac{D}{M} + 2\,000 C_s \theta^3\right)^2$$

where

- A is the effective SAED area, in square micrometres;
- D is the diameter, in micrometres, of the SAED aperture;
- M is the magnification of the objective lens;
- C_s is the spherical aberration coefficient, in millimetres, of the objective lens;
- θ is the maximum required Bragg angle, in radians.

It is not possible to reduce the effective SAED area indefinitely by the use of progressively smaller SAED apertures, because there is a fundamental limitation imposed by the spherical aberration coefficient of the objective lens.

If zone-axis ED analyses are to be performed, the TEM shall incorporate a goniometer stage which permits the TEM specimen to be either

a) rotated through 360°, combined with tilting through at least + 30° to − 30° about an axis in the plane of the specimen;

b) tilted through at least + 30° to − 30° about two perpendicular axes in the plane of the specimen.

The analysis is greatly facilitated if the goniometer permits eucentric tilting, although this is not essential. If EDXA and zone-axis ED are required on the same fibre, the goniometer shall be of a type which permits tilting of the specimen and acquisition of EDXA spectra without changing the specimen holder.

The TEM shall have an illumination and condenser lens system capable of forming an electron probe of diameter less than 250 nm.

NOTE 2 Use of an anti-contamination trap around the specimen is recommended if the required instrumental performance is to be obtained.

7.3.2 Energy dispersive X-ray analyser

The TEM shall be equipped with an energy dispersive X-ray analyser capable of achieving a resolution better than 180 eV (FWHM) on the MnKα. Since the performance of individual combinations of TEM and EDXA equipment is dependent on a number of geometrical factors, the required performance of the combination of the TEM and X-ray analyser is specified in terms of the measured X-ray intensity obtained from a fibre of small diameter, using a known electron beam diameter. Solid state X-ray detectors are least sensitive in the low energy region, and so measurement of sodium in crocidolite shall be the performance criterion. The combination of electron microscope and X-ray analyser shall yield, under routine analytical conditions, a background-subtracted NaKα integrated peak count rate of more than 1 count per second (cps) from a fibre of UICC crocidolite, 50 nm in diameter or smaller, when irradiated by an electron probe of 250 nm diameter or smaller at an accelerating potential of 80 kV. The peak/background ratio for this performance test shall exceed 1,0.

The EDXA unit shall provide the means for subtraction of the background, identification of elemental peaks, and calculation of background-subtracted peak areas.

7.3.3 Computer

Many repetitive numerical calculations are necessary, and these may be performed conveniently by relatively simple computer programmes. For analyses of zone-axis ED pattern measurements, a computer with adequate memory is required to accommodate the more complex programmes involved.

7.3.4 Plasma asher

For preparation of TEM specimens from MEC filters, a plasma asher, with a radio frequency power rating of 50 W or higher, shall be used to etch the surface of collapsed MEC filters. The asher shall be supplied with a controlled oxygen flow, and shall be modified, if necessary, to provide a valve to control the speed of air admission so that rapid air admission does not disturb particulates from the surface of the filter after the etching step.

NOTE 3 It is recommended that filters be fitted to the oxygen supply and the air admission line.

7.3.5 Vacuum coating unit

A vacuum coating unit capable of producing a vacuum better than 0,013 Pa shall be used for vacuum deposition of carbon on the membrane filters. A sample

holder is required which will allow a glass microscope slide to be continuously rotated during the coating procedure.

NOTE 4 A mechanism which also allows the rotating slide to be tilted through an angle of approximately 45° during the coating procedure is recommended. A liquid nitrogen cold trap above the diffusion pump may be used to minimize the possibility of contamination of the filter surfaces by oil from the pumping system. The vacuum coating unit may also be used for deposition of the thin film of gold, or other calibration material, when it is required on TEM specimens as an internal calibration of ED patterns.

7.3.6 Sputter coater

A sputter coater with a gold target may be used for deposition of gold onto TEM specimens as an integral calibration of ED patterns. Other calibration materials are acceptable. Experience has shown that a sputter coater allows better control of the thickness of the calibration material.

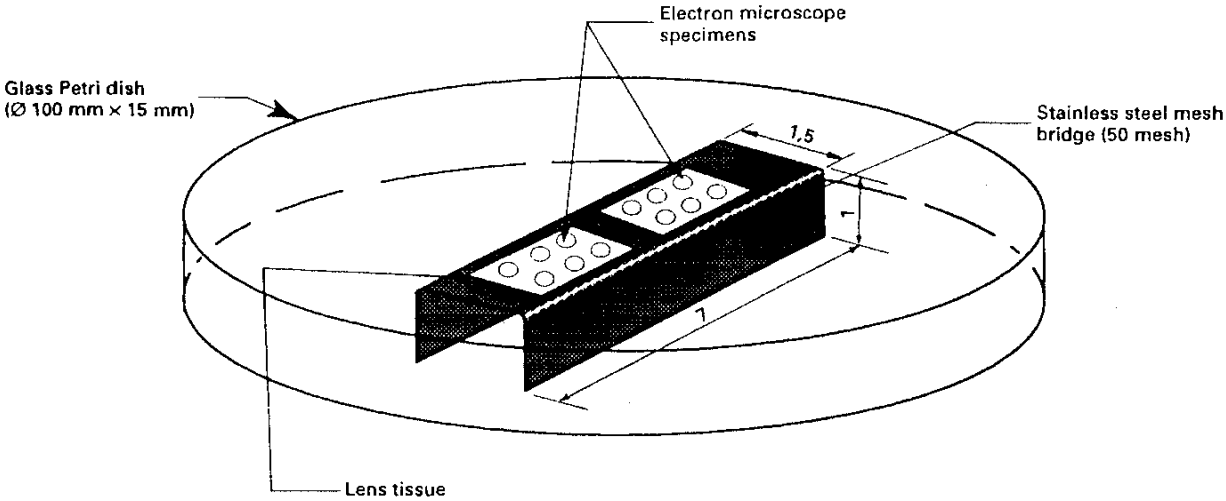
7.3.7 Solvent washer (Jaffe washer)

The purpose of the Jaffe washer is to allow dissolution of the filter polymer while leaving an intact evaporated carbon film supporting the fibres and other particles from the filter surface. One design of a washer which has been found satisfactory for various solvents and filter media is shown in figure 2. In general, either chloroform or 1-methyl-2-pyrrolidone has been used for dissolving polycarbonate filters and dimethylformamide or acetone has been used for dissolving MEC or cellulose nitrate filters. The higher evaporation rates of chloroform and acetone require that a reservoir of 10 ml to 50 ml of solvent be used, which may need replenishment during the procedure. Dimethylformamide and 1-methyl-2-pyrrolidone have lower vapour pressures and much smaller volumes of solvent may be used. It is recommended that all washers be used in a fume hood, and when specimens are not being inserted or removed, the Petri dish lid shall be in place during the solvent dissolution. The washer shall be cleaned before it is used for each batch of specimens.

7.3.8 Condensation washer

For more rapid dissolution of the filter polymer, or if difficulties are experienced in dissolving the filter polymer, use a condensation washer, consisting of a flask, condenser and cold finger assembly, with a heating mantle and means for controlling the temperature. A suitable assembly is shown in figure 3, using either acetone or chloroform as the solvent, depending on the type of filter.

Dimensions in centimetres



NOTE — Solvent is added until the meniscus contacts the underside of the stainless steel mesh bridge.

Figure 2 — Example of design of solvent washer (Jaffe washer)

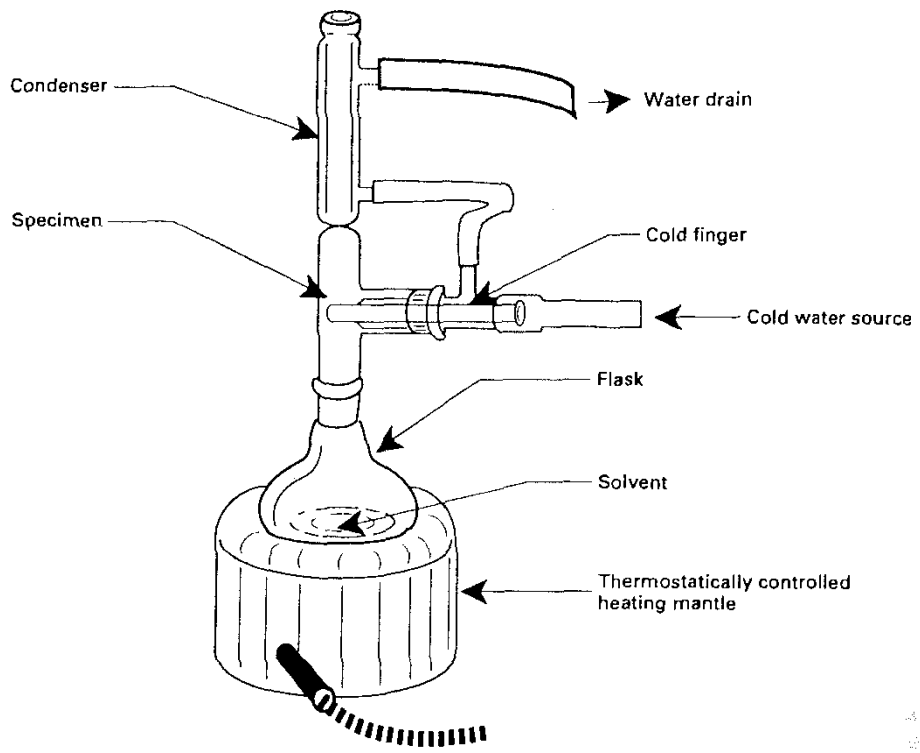


Figure 3 — Example of design of condensation washer

7.3.9 Slide warmer or oven

Use either a slide warmer or an oven for heating slides during the preparation of TEM specimens from MEC or cellulose nitrate filters. It is required to maintain a temperature of 65 °C to 70 °C.

7.3.10 Ultrasonic bath

An ultrasonic bath is necessary for cleaning the apparatus used for TEM specimen preparation.

7.3.11 Carbon grating replica

A carbon grating replica with about 2 000 parallel lines per millimetre shall be used to calibrate the magnification of the TEM.

7.3.12 Calibration specimen grids for EDXA

TEM specimen grids prepared from dispersions of calibration minerals are required for calibration of the EDXA system. Some suitable calibration minerals are riebeckite, chrysotile, halloysite, phlogopite, wollastonite and bustamite. The mineral used for calibration of the EDXA system for sodium shall be prepared using a gold TEM grid.

7.3.13 Carbon rod sharpener

The use of necked carbon rods, or equivalent, allows the carbon to be evaporated onto the filters with a minimum of heating.

7.3.14 Disposable tip micropipettes

A disposable tip micropipette, capable of transferring a volume of approximately 30 µl, is necessary for the preparation of TEM specimen grids from MEC filters.

7.4 Consumable supplies

7.4.1 Copper electron microscope grids

Copper TEM grids with 200 mesh are recommended. Grids which have grid openings of uniform size such that they meet the requirement specified in 9.6.2 shall be chosen. To facilitate the relocation of individual grid openings for quality assurance purposes, the use of grids with numerical or alphabetical indexing of individual grid openings is recommended.

7.4.2 Gold electron microscope grids

Gold TEM grids with 200 mesh are recommended to mount TEM specimens when sodium measurements are required in the fibre identification procedure. Grids which have grid openings of uniform size such that they meet the requirement specified in 9.6.2 shall be chosen. To facilitate the relocation of individual grid openings for quality assurance purposes, the use of grids with numerical or alphabetical indexing of individual grid openings is recommended.

7.4.3 Carbon rod electrodes

Spectrochemically pure carbon rods, shall be used in the vacuum evaporator (7.3.5) during carbon coating of filters.

7.4.4 Routine electron microscopy tools and supplies

Fine-point tweezers, scalpel holders and blades, microscope slides, double-coated adhesive tape, lens tissue, gold wire, tungsten filaments and other routine supplies are required.

7.4.5 Reference asbestos samples

Asbestos samples, shall be for preparation of reference TEM specimens of the primary asbestos minerals. The UICC set of minerals is suitable for this purpose.

8 Air sample collection

The desired analytical sensitivity is a parameter that shall be established for the analysis prior to sample collection. It is defined as the structure concentration corresponding to the detection of one structure in the analysis. For direct transfer methods of TEM specimen preparation, the analytical sensitivity is a function of the volume of air sampled, the active area of the collection filter, and the area of the TEM specimen over which structures are counted. If total airborne dust levels are high, it may be necessary to terminate sampling before the required volume has been sampled. If this happens, the analytical sensitivity required can be achieved only by counting structures on more grid openings, or by selective concentration of asbestos structures using an indirect TEM specimen preparation technique. Select the sampling rate and the period of sampling to yield the required analytical sensitivity, as detailed in table 1. Before air samples are collected, unused filters shall be analysed as described in 9.7 to determine the mean asbestos structure count for blank filters.

Air samples shall be collected using filter cassettes (7.1.1). During sampling, the cassette shall be supported on a stand (7.1.3) which is isolated from the vibrations of the pump (7.1.2). The cassette shall be held facing vertically downwards at a height of approximately 1,5 m to 2,0 m above ground/floor level, and shall be connected to the pump with a flexible tube.

Measure the sampling flow-rate at the front end of the cassette, both at the beginning and end of the sampling period, using a calibrated variable area flowmeter (7.1.4) temporarily attached to the inlet of the cassette. The mean value of these two measurements shall be used to calculate the total air volume sampled.

Basic strategies for monitoring environmental sources of airborne asbestos are described in annex G. After sampling, a cap shall be placed over the open end of the cassette, and the cassette packed with the filter face-upwards for return to the laboratory. Field blank filters shall also be included, as specified in 9.7, and submitted to the remaining analytical procedures along with the samples.

NOTES

5 In table 1 a collection filter area of 385 mm^2 is assumed, and the TEM grid openings are assumed to be 85 μm^2 square. The limit of detection is defined as the upper 95 % confidence limit of the Poisson distribution for a count of 0 structures. In the absence of background, this is equal to 2,99 times the analytical sensitivity. Backgrounds that are different from 0 observed during analysis of blank filters will degrade the limit of detection.

6 The analytical sensitivity S, expressed in number of structures per litre, is calculated using the following equation:

$$S = \frac{A_f}{kA_g V}$$

where

A_f is the active area, in square millimetres, of sample collection filter;

A_g is the mean area, in square millimetres, of grid openings examined;

k is the number of grid openings examined;

V is the volume of air sampled, in litres.

Table 1 — Examples of the minimum number of grid openings required to achieve a particular analytical sensitivity and limit of detection

Analytical sensitivity structures/l	Limit of detection structures/l	Volume of air sampled (litres) 500	1 000	2 000	3 000	4 000	5 000
0,1	0,30	1 066	533	267	178	134	107
0,2	0,60	533	267	134	89	67	54
0,3	0,90	356	178	89	60	45	36
0,4	1,2	267	134	67	45	34	27
0,5	1,5	214	107	54	36	27	22
0,7	2,1	153	77	39	26	20	16
1,0	3,0	107	54	27	18	14	11
2,0	6,0	54	27	14	9	7	6
3,0	9,0	36	18	9	6	5	4
4,0	12	27	14	7	5	4	4
5,0	15	22	11	6	4	4	4
7,0	21	16	8	4	4	4	4
10	30	11	6	4	4	4	4

9 Procedure for analysis

9.1 General

The techniques used to prepare TEM specimens are different for polycarbonate and cellulose ester filters. The preparation method to be used shall be either 9.3 or 9.4, depending on the type of membrane filter used for air sampling. Cleaning of the sample cassettes before they are opened, preparation of the carbon evaporator, criteria for acceptable specimen grids, and the requirement for blank determinations are identical for the two preparation techniques. TEM examination, structure counting, fibre identification and reporting of results are independent of the type of filter or preparation technique used.

The ability to meet the blank sample criteria is dependent on the cleanliness of equipment and supplies. Consider all supplies such as microscope slides and glassware as potential sources of asbestos contamination. It is necessary to wash all glassware before it is used. Wash any tools or glassware which come into contact with the air sampling filters or TEM specimen preparations both before use and between handling of individual samples. Where possible, disposable supplies should be used.

9.2 Cleaning of sample cassettes

Asbestos fibres can adher to the exterior surfaces of air sampling cassettes, and these fibres can be inadvertently transferred to the sample during handling. To prevent this possibility of contamination, and after ensuring that the cassette is tightly sealed, wipe the exterior surfaces of each sampling cassette before it is placed in the clean facility or laminar flow hood.

9.3 Direct preparation of TEM specimens from polycarbonate filters

9.3.1 Selection of filter area for carbon coating

Use a cleaned microscope slide to support representative portions of polycarbonate filter during the carbon evaporation. Double-coated adhesive tape is used to attach the filter portions to the glass slide. Take care not to stretch the polycarbonate filters during handling. Using freshly cleaned tweezers, remove the polycarbonate filter from the sampling cassette, and place it on to a second cleaned glass microscope slide which is used as a cutting surface. Using a freshly cleaned curved scalpel blade, cut the filter by rocking the blade from the point, pressing it into contact with the filter. Repeat the process as necessary. Several such portions may be mounted on the same microscope slide. The scalpel blade and tweezers shall be washed and dried between the handling of each filter. Identify the filter portions by writing on the glass slide.

9.3.2 Carbon coating of filter portions

Place the glass slide holding the filter portions on the rotation-tilting device, approximately 10 cm to 12 cm

from the evaporation source, and evacuate the evaporator chamber (7.3.5) to a vacuum better than 0,013 Pa. The evaporation of carbon shall be performed in very short bursts, separated by a few seconds to allow the electrodes to cool. If evaporation of carbon is too rapid, the strips of polycarbonate filter will begin to curl, and cross-linking of the surface will occur. This cross-linking procedures a layer of polymer which is relatively insoluble in organic solvents, and it will not be possible to prepare satisfactory TEM specimens. The thickness of carbon required is dependent on the size of particles on the filter, and approximately 30 nm to 50 nm has been found to be satisfactory. If the carbon film is too thin, large particles will break out of the film during the later stages of preparation, and there will be few complete and undamaged grid openings on the specimen. Too thick a carbon film will lead to a TEM image which is lacking in contrast, and the ability to obtain ED patterns will be compromised. The carbon film thickness should be the minimum possible, while retaining most of the grid openings of the TEM specimen intact.

9.3.3 Preparation of the Jaffe washer

Place several pieces of lens tissue, as shown in figure 2, on the stainless steel bridge (7.1.3) and fill the washer (see 7.3.7) with chloroform (6.2) or 1-methyl-2-pyrrolidone (6.3) to a level where the meniscus contacts the underside of the mesh, resulting in saturation of the lens tissue.

9.3.4 Placing of specimens in the Jaffe washer

Using a curved scalpel blade, cut three 3 mm square pieces of carbon-coated polycarbonate filter form the carbon-coated filter portion. Select three squares to represent the centre and the periphery of the active surface of the filter. Place each square of filter, carbon side up, on a TEM specimen grid, and place the grid and filter on the saturated lens tissue in the Jaffe washer. Place the three specimen grids from one sample on the same piece of lens tissue. Any number of separate pieces of lens tissue may be placed in the same Jaffe washer. Cover the Jaffe washer with the lid, and allow the washer to stand for at least 8 h.

NOTE 7 It has been found that some polycarbonate filters will not completely dissolve in the Jaffe washer, even after exposure to chloroform for as long as 3 d. This problem is more severe if the surface of the filter was overheated during the carbon evaporation. It has been found that the problem of residual undissolved filter polymer can be overcome in several ways:

a) condensation washing of the grids, using chloroform as the solvent, after the initial Jaffe washer treatment, can often remove much of the residual filter medium in a period of approximately 30 min. To carry out this procedure, transfer the piece of lens tissue supporting the specimen grids to the cold finger of the condensation washer (7.3.8), which has achieved stable operating conditions. Operate the washer for approximately 30 min after inserting the grids;

b) used in a Jaffe washer, 1-methyl-2-pyrrolidone has been found to be a more effective solvent than chloroform for polycarbonate filters. This solvent is more effective if the lens paper is not used and grids are placed directly on the stainless steel mesh of the Jaffe washer. A dissolution period of 2 h to 6 h has been found to be satisfactory. After dissolution is complete, remove the stainless steel mesh from the Jaffe washer and allow the grids to dry. 1-methyl-2-pyrrolidone evaporates very slowly. If it is required to dry the grids more rapidly, transfer the stainless steel bridge into another Petri dish, and add water (6.1) until the meniscus contacts the underside of the mesh. After approximately 15 min, remove the mesh and allow the grids to dry. If it is desired to retain water-soluble particle species on the TEM grids, ethanol may be used instead of water (6.1) for the second wash;

c) a mixture of 20 % 1,2-diaminoethane [ethylenediamine] and 80 % 1-methyl-2-pyrrolidone, used in a Jaffe washer, completely dissolves polycarbonate filters in 15 min, even if the surface of the filter has been overheated. To use this solvent place the grids directly on the stainless steel mesh of the Jaffe washer, do not use the lens paper. After a period of 15 min, transfer the stainless steel bridge into another Petri dish, and add water (6.1) until the meniscus contacts the underside of the mesh. After approximately 15 min, remove the mesh and allow the grids to dry. If it is desired to retain water-soluble particle species on the TEM grids, ethanol may be used instead of water (6.1) for the second wash.

9.3.5 Rapid preparation of TEM specimens from PC filters

TEM specimens can be prepared rapidly from PC filters, if desired, by washing for approximately 1 h in a Jaffe washer, followed by washing for 30 min in a condensation washer using chloroform as the solvent. The alternative filter dissolution procedures described in note 7 may also be used.

9.4 Direct preparation of TEM specimens from cellulose ester filters

9.4.1 Selection of area of filter for preparation

Using clean tweezers, remove the filter from the filter cassette, and place it on a cleaned microscope slide. Using a clean, curved scalpel blade, cut out a portion of the filter.

9.4.2 Preparation of solution for collapsing cellulose ester filters

Mix 35 ml of dimethylformamide (6.4), and 15 ml of glacial acetic acid (6.5) with 50 ml of water (6.1). Store this mixture in a clean bottle, The mixture is stable and suitable for use for up to 3 months after preparation.

9.4.3 Filter collapsing procedure

Using a micropipette with a disposable tip (7.3.14), place 15 $\mu l/cm^2$ to 25 $\mu l/cm^2$ of the solution prepared in 9.4.2 on a cleaned microscope slide, and using the end of the pipette tip, spread the liquid over the area to be occupied by the filter portion. Place the filter portion, active surface upwards, on top of the solution, lowering the edge of the filter at an angle of about 20° so that air bubbles are not created. Remove any solution not absorbed by the filter by allowing a paper tissue to contact the liquid at the edge of the filter. More than one filter portion may be placed on one slide. Place the slide either on a thermostatically controlled slide warmer (7.3.9) at a temperature of 65 °C to 70 °C, or in an oven (7.3.9) at this temperature, for 10 min. The filter collapses slowly to about 15 % of its original thickness. The procedure leaves a thin, transparent polymer film, with particles and fibres embedded in the upper surface.

9.4.4 Plasma etching of the filter surface

The optimum conditions and time for plasma etching (see 7.3.4) have been determined experimentally from the recovery of fine chrysotile fibrils on 0,8 µm pore size MEC filters. The conditions required in a particular plasma asher shall be established using the procedure specified in annex A. Place the microscope slide holding the collapsed filter portions in the plasma asher, and etch for the time and under the conditions determined. Take care to ensure that the correct conditions are respected. After etching, admit air slowly to the chamber and remove the microscope slide.

Adjust the air admission valve of the plasma asher such that the time taken for the chamber to reach atmospheric pressure exceeds 2 min. Rapid air admission may disturb particulates on the surface of the etched filter.

9.4.5 Carbon coating

Coat the microscope slide holding the collapsed filter portions with carbon as specified in 9.3.2.

9.4.6 Preparation of the Jaffe washer

Place several pieces of lens tissue on the stainless steel bridge, and fill the washer with dimethylformamide (6.4) or acetone (6.6) to a level where the meniscus contacts the underside of the mesh, resulting in saturation of the lens tissue.

9.4.7 Placing of specimens in the Jaffe washer

Place the specimens in the Jaffe washer as specified in 9.3.4. Specimens are normally cleared after approximately 4 h.

9.4.8 Rapid preparation of TEM specimens from cellulose ester filters

An alternative washing procedure may be used to prepare TEM specimens from cellulose ester filters more rapidly than can be achieved by the Jaffe washing procedure. After the specimens have been washed in a Jaffe washer for approximately 1 h, transfer the piece of lens tissue supporting the specimens to the cold finger of a condensation washer (7.3.8) operating with acetone as the solvent because dimethylformamide shall not be used in a condensation washer. Operate the condensation washer for approximately 30 min. This treatment removes all the remaining filter polymer.

9.5 Criteria for acceptable TEM specimen grids

Valid data cannot be obtained unless the TEM specimens meet specified quality criteria. Examine the TEM specimen grid in the electron microscope at a sufficiently low magnification (× 300 to × 1 000) for complete grid openings to be inspected. Reject the grid if

a) the TEM specimen has not been cleared of filter medium by the filter dissolution step. If the TEM specimen exhibits areas of undissolved filter medium, and if at least two of the three specimen grids are not cleared, either additional washing with solvent shall be carried out, or new specimens shall be prepared from the filter;

b) the sample is overloaded with particulate. If the specimen grid exhibits more than approximately 10 % obscuration on the majority of the grid openings, the specimen shall be designated as overloaded. This filter cannot be alanysed satisfactorily using the direct preparation methods because the grid is too heavily loaded with debris to allow separate examination of individual particles by ED and EDXA, and obscuration of fibres by

other particulates may lead to underestimation of the asbestos structure count;

c) the particulate deposits on the specimen are not uniformly distributed from one grid opening to the next. If the particulate deposits on the specimen are obviously not uniform from one grid opening to the next, the specimen shall be designated as non-uniform. This condition is a function either of the air sampling conditions or of the fundamental nature of the airborne particulate. Satisfactory analysis of this filter may not be possible unless a large number of grid openings is examined;

d) the TEM grid is too heavily loaded with fibrous structures to make an accurate count. Accurate counts cannot be made if the grid has more than approximately 7 000 structures/mm^2; or

e) more than approximately 25 % of the grid openings have broken carbon film over the whole grid opening. Since the breakage of carbon film is usually more frequent in areas of heavy deposit, counting of the intact openings can lead to an underestimate of the asbestos structure count.

NOTE 8 If the specimens are rejected because unacceptable numbers of grid openings exhibit broken carbon replica, an additional carbon coating may be applied to the carbon coated filter, and new specimen grids prepared. The larger particles can often be supported by using a thicker carbon film. If this action does not produce acceptable specimen grids, this filter cannot be analysed using the direct preparation methods.

If one or more of the conditions described in b), c), d) or e) exists, it may not be possible to analyse the sample by this method.

9.6 Procedure for structure counting by TEM

9.6.1 General

The examination consists of a count of asbestos structures which are present on a specified number of grid openings. Fibres are classified into groups on the basis of morphological observations, ED patterns and EDXA spectra. The total number of structures to be counted depends on the statistical precision desired. In the absence of asbestos structures, the area of the TEM specimen grids which must be examined depends on the analytical sensitivity required. The precision of the structure count depends not only on the total number of structures counted, but also on their uniformity from one grid opening to the next. Additional structure counting will be necessary if greater precision is required.

In order that the estimate of the structure density on the sampling filter shall not be based on the small area represented by one specimen grid, grid openings shall be examined on two of the three specimen grids prepared. Then combine the results in the calculation of the structure density. Structure counts shall be made at a magnification of approximately × 20 000, and shall be terminated at the end of the examination of the grid opening on which the 100th asbestos structure is observed, except that the count shall be continued until a minimum of 4 grid openings have been examined. Otherwise, the structure count shall continue to that number of grid openings at which the specified analytical sensitivity has been achieved.

NOTE 9 The normal range for the number of grid openings which should be examined is from 4 to 20. If insufficient air has been sampled through the filter, the calculation in 9.6.4 can indicate that an impractically large number of grid openings should be examined. When this situation occurs, a larger value of analytical sensitivity may have to be accepted.

9.6.2 Measurement of mean grid opening area

The mean grid opening area shall be measured for the type of TEM specimen grids in use. The standard deviation of the mean of 10 openings selected from 10 grids should be less than 5 %. As an optional procedure, or if the 5 % standard deviation criterion cannot be demonstrated, the dimensions of each grid opening examined in the TEM shall be measured at a calibrated magnification.

9.6.3 TEM alignment and calibration procedures

Before structure counting is performed, align the TEM according to instrumental specifications. Calibrate the TEM and EDXA system according to the procedures specified in annex B.

9.6.4 Determination of stopping point

Before structure counting is begun, calculate the area of specimen to be examined in order to achieve the selected analytical sensitivity. Calculate the maximum number of grid openings to be examined using the following equation:

$$k = \frac{A_f}{A_g V S}$$

where

k is the number of grid openings to be examined, rounded upwards to the next highest integer;

A_f is the area, in square millimetres, of sample filter;

A_g is the area, in square millimetres, of TEM specimen grid opening;

V is the volume of air sampled, in litres;

S is the required analytical sensitivity, expressed in number of structures per litre.

9.6.5 General procedure for structure counting and size analysis

Use at least two specimen grids prepared from the filter in the structure count. Select at random several grid openings from each grid, and combine the data in the calculation of the results.

Use a form similar to that shown in figure 4 to record the data. Insert the first specimen grid into the TEM.

NOTE 10 In order to facilitate quality assurance measurements which require re-examination of the same grid opening by different microscopists, the grid should be inserted into the specimen holder in a standard orientation with the grid bars parallel and perpendicular to the axis of the specimen holder. This will provide scan directions parallel to the edges of the grid opening. It should be ensured that all microscopists begin scanning at the same starting point on the grid opening, and that they use similar scan patterns. This procedure permits rapid relocation of fibrous structures for further examination if necessary.

Select a typical grid opening and set the screen magnification to the calibrated value (approximately × 20 000). Adjust the sample height until the features in the centre of the TEM viewing screen are at the eucentric point. Set the goniometer tilt angle to zero. In column 1 of the data recording form, record the number or letter used to identify the grid. In column 2, record the identification of the particular grid opening. Position the specimen so that the grid opening is positioned with one corner visible on the screen. Move the image by adjustment of only one translation control, carefully examining the sample for fibres, until the opposite side of the grid opening is encountered. Move the image by a predetermined distance less than one screen diameter, using the other translation control, and scan the image in the reverse direction. Continue the procedure in this manner until the entire grid opening has been inspected in a pattern similar to that shown in figure 5. When a fibrous structure is detected, assign a sequential number to the primary structure in column 3, perform the identification procedures required as detailed in annex E, and enter the appropriate compositional classification on the structure counting form in column 5. Assign a morphological classification to the structure according to the procedures specified in annex D, and record this in column 6. Measure on the TEM viewing screen the length and width of the image of the primary structure, in millimetres, and record these measurements in columns 7 and 8. For a disperse cluster or matrix, assign a compositional classification and a morphological classification to each structure component, measure the length and width, and enter the data in columns 4 to 8. Use column 4 of the data recording form to tabulate the sequential number of total structures taking into account structure components. if non-asbestos fibres are observed, note their presence and type, if known. After a fibrous structure has been examined and measured, relocate the original field of view accurately before continuing scanning of the specimen. Failure to do this may cause structures to be overlooked or counted twice. Continue the examination until the completion of the grid opening on which the 100th asbestos structure has been recorded, or until the number of grid openings required to achieve the specified analytical sensitivity, calculated according to 9.6.4, have been examined whichever occurs first. The data shall be drawn approximately equally from a minimum of two grids. Regardless of the value calculated according to 9.6.4, fibrous structures on a minimum of four openings shall be counted.

9.6.6 Measurement of concentration for asbestos fibres and bundles longer than 5 µm

Consider improving the statistical validity for measurement of asbestos fibres and bundles longer than 5 µm by additional examination at a lower magnification, taking account only of the longer fibres and bundles. Perform this extended examination for fibres and bundles longer than 5 µm in accordance with the procedures specified in annex E. Use a magnification of approximately × 10 000 for counting all asbestos fibres and bundles longer than 5 µm, or approximately × 5 000 if only fibres and bundles within the diameter range 0,2 µm to 3,0 µm are to be counted. Continue the count until completion of the grid opening on which 100 fibres and bundles have been recorded, or until a sufficient area of the specimen has been examined to achieve the desired analytical sensitivity. Only those structures which are identified as, or are suspected to be, either chrysotile or one of the amphibole minerals will be reported in either the original or the extended TEM examination. Other materials, such as gypsum, cellulose fibres, and filter artifacts such as undissolved filter strands, will not be included in the fibre count. This restriction is intended to ensure that the best statistical validity is obtained for the materials of interest.

TEM asbestos structure count (page of)

Report number: .....
Sample number: .....
File name: .....
Sample description: .....
.....
.....
Preparation date: ..... By: .....
Analysis date: ..... By: .....
Computer entry date: ..... By: .....

Air volume: ..... litres
Sample filter area: ..... mm^2
Magnification: .....
Grid opening dimension: ..... µm
Level of analysis (C): .....
(A): .....

Grid	Grid opening	Number of structures		Class	Type of structure	Length mm	Width mm	Comments
		primary	total					

Figure 4 — Example of structure counting form

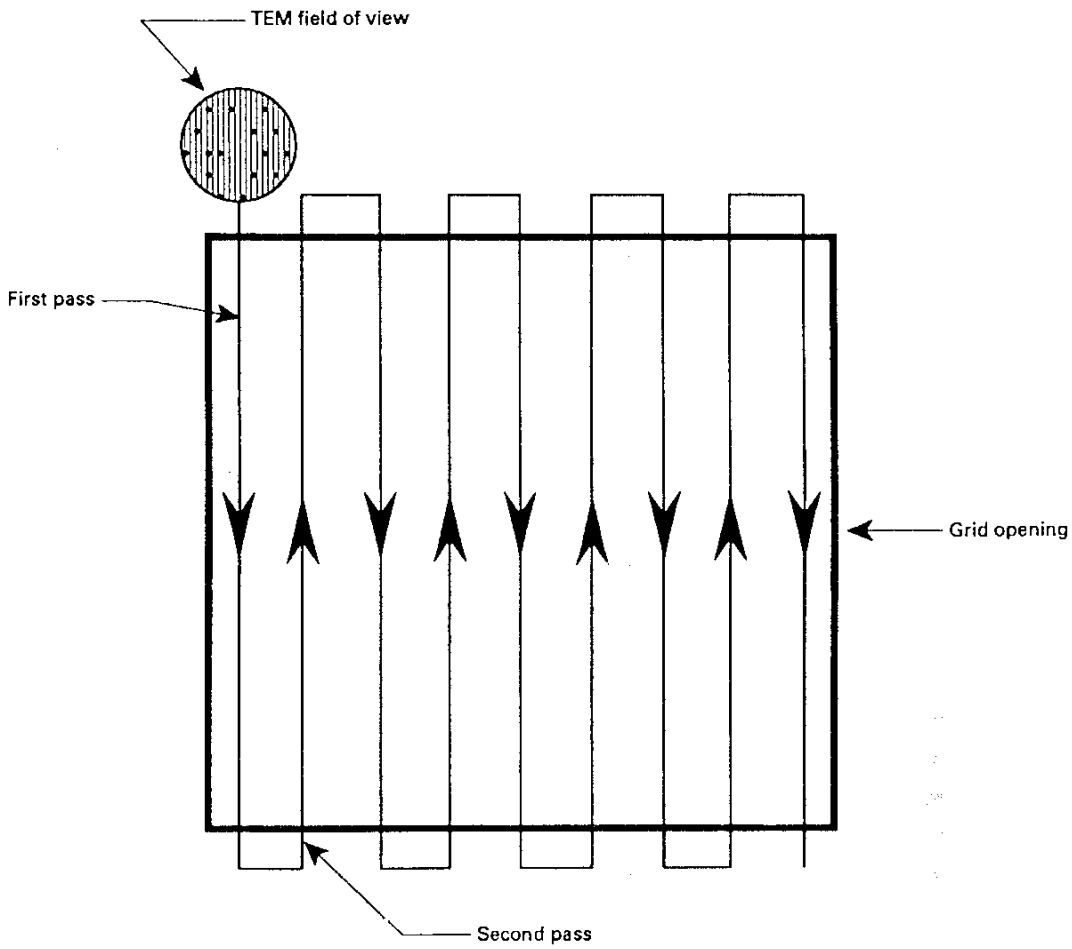


Figure 5 — Example of scanning procedure for TEM specimen examination

9.7 Blank and quality control determinations

Before air samples are collected, a minimum of two unused filters from each filter lot of 100 filters shall be analysed to determine the mean asbestos structure count. If the mean count for all types of asbestos structures is found to be more than 10 structures/mm^2, or if the mean fibre count for asbestos fibres and bundles longer than 5 µm is more than 0,1 fibre/mm^2, reject the filter lot.

To ensure that contamination by extraneous asbestos fibres during specimen preparation is insignificant compared with the results reported on samples, establish a continuous programme of blank measurements. At least one field blank shall be processed along with each batch of samples. In addition, at least one unused filter shall be included with every group of samples prepared on one microscope slide.

Initially, and also at intervals afterwards, ensure that samples of known asbestos concentrations can be analysed satisfactorily. Since there is a subjective component in the structure counting procedure, it is necessary that recounts of some specimens be made by different microscopists, in order to minimize the subjective effects. Such recounts provide a means of maintaining comparability between counts made by different microscopists. Variability between and within microscopists and between laboratories shall be characterized. These quality assurance measurements shall constitute approximately 10 % of the analyses. Repeat results should not differ at the 5 % significance level.

9.8 Calculation of results

Calculate the results using the procedures detailed in annex F. Prior to the TEM examination of the specimens, the level of analysis was specified. Before the results are calculated, the compositional and morphological classifications to be included in the result shall be specified. The chi-squared uniformity test shall be conducted using the number of primary asbestos structures found on each grid opening, prior to the application of the cluster and matrix counting criteria. The concentration result shall be calculated using the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

10 Performance characteristics

10.1 General

It is important to use this analytical method in conjunction with a continuous quality control programme. The quality control programme should include use of standard samples, blank samples, and both interlaboratory and intralaboratory analyses.

10.2 Interferences and limitations of fibre identification

Unequivocal identification of every chrysotile fibre is not possible, due to both instrumental limitations and the nature of some of the fibres. The requirement for a calibrated ED pattern eliminates the possibility of an incorrect identification of the fibre selected. However, there is a possibility of misidentification of fibres for which both the morphologies and the ED patterns are reported on the basis of visual inspection only. The only significant possibilities of misidentification occur with halloysite, vermiculite scrolls or palygorskite, all of which can be discriminated from chrysotile by the use of EDXA and by observation of the 0,73 nm (002) reflection of chrysotile in the ED pattern.

As in the case of chrysotile fibres, complete identification of every amphibole fibre is not possible due to instrumental limitations and the nature of some of the fibres. Moreover, complete identification of every amphibole fibre is not practical due to the limitations of both time and cost. Particles of a number of other minerals with compositions similar to those of some amphiboles could be erroneously classified as amphibole when the classification criteria do not include zone-axis ED techniques. However, the requirement for quantitative EDXA measurements on all fibres as support for the random orientation ED technique makes misidentification very unlikely, particularly when other similar fibres in the same sample have been identified as amphibole by zone-axis methods. The possibility of misidentification is further reduced with increasing aspect ratio, since it is rare for the minerals with which amphibole may be confused to display an asbestiform habit.

10.3 Precision and accuracy (see ISO Standard Handbook No. 3)

10.3.1 Precision

The analytical precision that can be obtained is dependent upon the number of structures counted, and also on the uniformity of the particulate deposit on the original filter. Assuming that the structures are randomly deposited on the filter, if 100 structures are counted and the loading is at least 3,5 structures/grid opening, computer modelling of the counting procedure shows that a coefficient of variation of about 10 % can be expected. As the number of structures counted decreases, the precision will also decrease approximately as $\sqrt{N}$, where N is the number of structures counted. In practice, particulate deposits obtained by filtration of ambient air samples are rarely ideally distributed, and it is found that the precision is correspondingly reduced. Degradation of precision is a consequence of several factors, such as:

a) non-uniformity of the filtered particulate deposit;

b) distorsion of the fibre distribution by application of the structure counting criteria;

c) variation between microscopists in their interpretation of the fibrous structures;

d) variation between microscopists in their ability to detect and identify fibres.

The 95 % confidence interval about the mean for a single structure concentration measurement using this analytical method should be approximately ± 25 % when 100 structures are counted over 10 grid openings.

10.3.2 Accuracy

There is no independent method available to determine the accuracy.

NOTE 11 It has been demonstrated that, after polycarbonate membrane filters have been coated with carbon, particulate material is transferred to the TEM specimens without measurable losses. However, if the filters are heavily loaded by particulate material, some of this may be lost before they are coated with carbon. Good comparability between the capillary-pore polycarbonate pro-

cedure and the cellulose ester filter procedure has been demonstrated for laboratory-generated aerosols of chrysotile asbestos.

10.3.3 Interlaboratory and intralaboratory analyses

Interlaboratory and intralaboratory analyses are required in order to monitor systematic errors that may develop among microscopists when using this method. These analyses should be designed to test both the overall method and the performance of individual microscopists. Repeating preparation of TEM grids from different sectors of a filter, followed by examination of the grids by a different microscopist, is a test for the reproducibility of the whole method. However, non-uniformity of the particulate deposit on the filter may lead to differences which are not related to the performance of the microscopists. Verified fibre counting (counting of asbestos structures on the same grid opening of a TEM grid by two or more operators, followed by resolution of any discrepancies) may be used both as a training aid and to determine the performance of different microscopists. The use of indexed TEM grids as described in 7.4.1 and 7.4.2 is recommended in order to facilitate relocation of specific grid openings.

10.4 Limit of detection

The limit of detection of the method can be varied by choice of the area of the collection filter, the volume of air sampled and the area of the specimen examined in the TEM. It is also a function of the background of asbestos structures on unused filters. A limit of detection shall be quoted for each sample analysis.

NOTE 12 In practice, the lowest limit of detection is frequently determined by the total suspended particulate concentration, since each particle on the filter must be separated from adjacent ones by a distance large enough for the particle to be identified without interference. Particulate loadings on sampling filters greater than 25 $\mu g/cm^2$ usually preclude preparation of TEM specimens by the direct methods. If the analysis is to be performed with an acceptable expenditure of time, the area of the specimen examined in the TEM for structures of all sizes is limited in most cases to between 10 and 20 grid openings. In typical ambient or building atmospheres, it has been found that an analytical sensitivity of 1 structure/l can be achieved. In some circumstances, where the atmosphere is exceptionally clean, this can be reduced to 0,1 structure/l or lower. For fibres and bundles longer than 5 µm, the reduced magnifications specified permit larger areas of the TEM specimens to be examined with an acceptable expenditure of time, resulting in proportionately lower limits of detection. If no structures are found in the analysis, the upper 95 % confidence limit can be quoted as the upper boundary of the concentration, corresponding to 2,99 times the analytical sensitivity if a Poisson distribution of structures on the filter is assumed. This 95 % confidence limit for 0 structures counted is taken as the detection limit. Since there is sometimes contamination of unused samples filters by asbestos structures, this should also be taken into account in the discussion of limits of detection.

11 Test report

The test report shall include at least the following information:

a) reference to this International Standard;

b) identification of the sample;

c) the date and time of sampling, and all necessary sampling data;

d) the date of the analysis;

e) the identity of the analyst;

f) any procedure used that is not specified in this International Standard or regarded as optional;

g) a complete listing of the structure counting data (the following data should be included: grid opening number, structure number, identification category, structure type, length and width of the structure in micrometres, and any comments concerning the structure);

h) a statement of the minimum acceptable identification category and the maximum identification category attempted (refer to tables D.1 and D.2);

i) a statement specifying which identification and structure categories have been used to calculate the concentration values;

j) separate concentration values for chrysotile and amphibole structures, expressed in number of asbestos structures per litre;

k) the 95 % confidence interval limits for the concentration values, expressed in number of asbestos structures per litre;

l) the analytical sensitivity, expressed in number of asbestos structures per litre;

m) the limit of detection, expressed in number of asbestos structures per litre;

n) compositional data for the principal varieties of amphibole, if present;

o) items g) to m) for asbestos fibres and bundles longer than 5 μm;

p) items g) to m) for PCM equivalent asbestos fibres and bundles.

An example of a suitable format for the structure counting data is shown in figures 6 and 7.

Sample analysis information (page 1)

Laboratory name	**Report number**	**Date**

Sample: 456 Queen Street
Ashby de la Zouch
Exterior sample 1991-09-09

Air volume:	2 150,0 litres
Area of collection filter:	385,0 mm^2
Level of analysis (chrysotile):	CD or CMQ
Level of analysis (amphibole):	ADQ
Magnification used for fibre counting:	× 20 500
Aspect ratio for fibre definition:	5/1
Mean dimension of grid openings:	95,4 μm
Initials of analyst:	JMW
Number of grid openings examined:	10
Analytical sensitivity:	1,968 structures/l
Number of primary asbestos structures:	13
Number of asbestos structures counted:	26
Number of asbestos structures > 5 μm :	7
Number of asbestos fibres and bundles > 5 μm :	10
Number of PCM equivalent asbestos structures:	3
Number of PCM equivalent asbestos fibres:	5

Figure 6 — Example of format for reporting sample and preparation data

Sample analysis information (pages 2 and following)

Laboratoriy name **Report number** **Date**

Sample: 456 Queen Street
Ashby de la Zouch
Exterior sample 1991-09-09

TEM asbestos structure count — Raw data

Grid	Grid opening	Number of structures primary	Number of structures total	Identification[1]	Structure type	Length µm	Width µm	Comments
A	F4-4	1	1	CD	F	1,7	0,045	
		2	2	CMQ	B	2,6	0,09	
		3	3	ADQ	F	4,0	0,15	Crocidolite
	E3-6	4	4	CD	MC+0	3,5	1,3	
	E5-1	5		CD	MD43	7,5	5,0	
			5	CD	MB	7,7	0,30	
			6	CMQ	MF	5,6	0,045	
			7	CD	MB	5,1	0,30	
			8	CD	MF	1,7	0,045	
B	F4-1	6		CD	CD+0	6,5	3,0	
			9	CD	CB	3,5	0,15	
			10	CD	CF	3,5	0,045	
			11	CMQ	CR+0	2,6	1,9	
	G5-1	7		CD	CD31	6,1	3,2	
			12	CD	CB	5,6	0,3	
			13	CMQ	CF	4,0	0,045	
			14	CMQ	CB	3,2	0,090	
	E4-4	8	15	CD	B	1,5	0,23	
		9	16	AD	F	8,7	0,15	
C	G4-4	10		CMQ	CD42	25	5,6	
			17	CMQ	CB	15	0,15	
			18	CMQ	CF	9,4	0,045	
			19	ADQ	CF	3,6	0,30	Tremolite
			20	CM	CF	4,2	0,045	
	E4-4			No fibres				
	E5-6	11		ADQ	CD+3	9,4	2,5	
			21	ADQ	CF	7,1	0,30	Amosite
			22	ADQ	CF	6,2	0,10	Crocidolite
			23	CM	CB	5,1	0,2	
			24	CM	CR+0	3,3	1,8	
	F4-1	12	25	CMQ	MC10	3,7	2,1	
		13	26	CD	CC+0	7,4	0,5	

1) Identification codes listed in tables D.1 and D.2.

Figure 7 — Example of format for reporting structure counting data

Annex A
(normative)

Determination of operating conditions for plasma asher

A.1 General

During the preparation of TEM specimens from an MEC or cellulose nitrate filter, the spongy structure of the filter is collapsed into a thinner film of polymer by the action of a solvent. Some of the particles on the surface of the original filter become completely buried in the polymer, and the specimen preparation procedure incorporates a plasma etching step to oxidize the surface layer of the polymer. Particles buried by the filter collapsing step are then exposed so that they can become subsequently affixed to the evaporated carbon film without altering their position on the original filter. The amount of etching is critical, and individual ashers vary in performance. Therefore, the plasma asher (7.3.4) shall be calibrated to give a known amount of etching of the surface of the collapsed filter. This is carried out by adjusting the radio-frequency power output and the oxygen flow-rate, and measuring the time taken to completely oxidize an uncollapsed cellulose ester filter with 25 mm diameter of the same type and pore size as those used in the analysis.

A.2 Procedure

Place an unused cellulose ester filter, with 25 mm diameter, of the same type as that being used, in the centre of a glass microscope slide. Position the slide approximately in the centre of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 ml/min to 20 ml/min. Adjust the tuning of the system so that the intensity of the plasma in maximized. Measure the time required for complete oxidation of the filter. Determine operating parameters which result in complete oxydation of the filter in a period of approximately 15 min. For etching of collapsed filters, these operating parameters shall be used for a period of 8 min.

NOTE 13 Plasma oxidation at high radio-frequency powers will cause the filter to shrink and curl, followed by sudden violent ignition. At lower powers, the filter will remain in position and will slowly become thinner until it is nearly transparent. It is recommended that a radio-frequency power be used such that violent ignition does not occur. When multiple filters are etched, the rate of etching is reduced, and the system should be calibrated accordingly.

Annex B
(normative)

Calibration procedures

B.1 Calibration of the TEM

B.1.1 Calibration of TEM screen magnification

The electron microscope should be aligned according to the specifications of the manufacturer. Initially, and at regular intervals, calibrate the magnifications used for the analysis using a diffraction grating replica (7.3.11). Adjust the specimen height to the eucentric position before carrying out the calibration. Measure the distance on the fluorescent viewing screen occupied by a convenient number of repeat distances of the grating image, and calculate the magnification. Always repeat the calibration after any instrumental maintenance or change of operating conditions. The magnification of the image on the viewing screen is not the same as that obtained on photographic plates or film. The ratio between these is a constant value for the particular model of TEM.

B.1.2 Calibration of ED camera constant

Calibrate the camera constant of the TEM when used in ED mode. Use a specimen grid supporting a carbon film on which a thin film of gold has been evaporated or sputtered. Form an image of the gold film with the specimen adjusted to the eucentric position and select ED conditions. Adjust the objective lens current to optimize the pattern obtained, and measure the diameters of the innermost two rings either on the fluorescent viewing screen or on a recorded image. Calculate the radius-based camera constant, λL, for both the fluorescent screen and the photographic plate or film, using the following equation:

$$\lambda L = \frac{aD}{2{,}0\sqrt{h^2 + k^2 + l^2}}$$

where

λ is the wavelength, in nanometres, of the incident electrons;

L is the camera length, in millimetres;

a is the unit cell dimension of gold, in nanometres (= 0,407 86 nm);

D is the diameter, in millimetres, of the (hkl) diffraction ring.

Using gold as the calibration material, the radius-based camera constant is given by

$\lambda L = 0{,}117\ 74D$ mm·nm (smallest ring)

$\lambda L = 0{,}101\ 97D$ mm·nm (second ring)

B.2 Calibration of the EDXA system

Energy calibration of the EDXA system for a low energy and high energy peak shall be performed regularly. Calibration of the intensity scale of the EDXA system permits quantitative composition data, at an accuracy of about 10 % of the elemental concentration, to be obtained from EDXA spectra of reference silicate minerals involving the elements Na, Mg, Al, Si, K, Ca, Mn and Fe, and applicable certified reference materials. If quantitative determinations are required for minerals containing other elements, reference standards other than those referred to below will be required. Well-characterized mineral standards permit calibration of any TEM-EDXA combination which meets the instrumental specifications of 7.3.1 and 7.3.2, so that EDXA data from different instruments can be compared. Reference minerals are required for the calibration; the criteria for selection being that they should be silicate minerals with matrices as close as possible to those of the amphiboles or serpentine, and that small individual fragments of the minerals are homogeneous in composition within a few percent.

Determine the compositions of these standards by electron microprobe analysis or chemicals methods. Crush fragments of the same selected mineral standards and prepare filters by dispersal of the crushed material in water and immediate filtration of the suspensions. Prepare TEM specimens from these filters according to the procedures specified in clause 9. These TEM specimens can then be used to calibrate any TEM-EDXA system so that comparable composi-

tional results can be obtained from different instruments.

NOTES

14 The microprobe analysis of the mineral standards are carried out by conventional techniques which can be found in annex J. The mineral is first embedded in a mount of poly(methyl methacrylate) or epoxy resin. The mount is then ground and polished to achieve a flat, polished surface of the mineral fragment. This surface is then analysed, using suitable reference standards, preferably oxide standards of the individual elements wherever these are available. It is necessary to take into account the water concentration in the minerals, which in the case of chrysotile amounts to 13 % by mass. This water content may vary due to losses in the vacuum system.

15 Aqueous suspensions of mineral standards should be filtered immediately after preparation, since alkali and alkali earth metals may be partially leached from minerals containing these elements.

Express the results of the electron microprobe analyses as atomic or mass percentage ratios relative to silicon. X-ray peak ratios of the same elements relative to silicon, obtained from the EDXA system, can then be used to calculate the relationship between peak area ratio and atomic or mass percentage ratio. The technique was described by Cliff and Lorimer (see annex J, reference [8]).

The X-rays generated in a thin specimen by an incident electron beam have a low probability of interacting with the specimen. Thus, mass absorption and fluorescence effects are negligible. In a silicate mineral specimen containing element i, the following equation can be used to perform quantitative analyses in the TEM:

$$\frac{C_i}{C_{Si}} = k_i \times \frac{A_i}{A_{Si}}$$

where

C_i is the concentration or atomic percentage of element i;

C_{Si} is the concentration or atomic percentage of silicon;

A_i is the elemental integrated peak area for element i;

A_{Si} is the elemental integrated peak area for silicon;

k_i is the k-ratio for element i relative to silicon.

For a particular instrumental configuration and a particular particle size, the value of k_i is constant.

To incorporate correction for the particle size effect on peak area ratios (see annex J, references [35] and [36], extend the Cliff and Lorimer technique by obtaining separate values of the constant k_i for different ranges of fibre diameter. It is recommended that 20 EDXA measurements be made for each range of fibre diameters. Suitable ranges of fibre diameter are:

< 0,25 µm; 0,25 µm to 0,5 µm; 0,5 µm to 1,0 µm; > 1,0 µm.

Insert the TEM grid into the transmission electron microscope, obtain an image at the calibrated higher magnification of about × 20 000, and adjust the specimen height to the eucentric point. If the X-ray detector is a side-entry variety, tilt the specimen towards the X-ray detector. Select an isolated fibre or particle less than 0,5 µm in width, and accumulate an EDXA spectrum using an electron probe of suitable diameter. When a well-defined spectrum has been obtained, perform a background subtraction and calculate the background-corrected peak areas for each element listed, using energy windows centred on the peaks. Calculate the ratio of the peak area for each specified element relative to the peak area for silicon. All background-subtracted peak areas used for calibration shall exceed 400 counts.

Repeat this procedure for 20 particles of each mineral standard. Reject analyses of any obviously foreign particles. Calculate the arithmetic mean concentration to peak area ratio, k_i (k-ratio), for each specified element of each mineral standard and for each of the fibre diameter ranges. Periodic routine checks shall be carried out to ensure that there has been no degradation of the detector performance. These k-ratios are used to calculate the elemental concentrations of unknown fibres, using the Cliff and Lorimer relationship.

Annex C
(normative)

Structure counting criteria

C.1 General

In addition to isolated fibres, other assemblages of particles and fibres frequently occur in air samples. Groupings of asbestos fibres and particles, referred to as "asbestos structures", are defined as fibre bundles, clusters and matrices. The numerical result of a TEM examination depends largely on whether the analyst assigns such an assemblage of fibres as a single entity, or as the estimated number of individual fibres which form the assemblage. It is therefore important that a logical system of counting criteria be defined, so that the interpretation of these complex structures is the same for all analysts, and so that the numerical result is meaningful. Imposition of specific structure-counting criteria generally requires that some interpretation, partially based on uncertain information on health effects, be made of each asbestos structure found. It is not the intention of this International Standard to make any interpretations based on health effects, and it is intended that a clear separation shall be made between recording of structure counting data, and later interpretation of those data. The system of coding specified in this International Standard permits a clear morphological description of the structures to be recorded in a concise manner suitable for later interpretation, if necessary, by a range of different criteria, without the necessity for re-examination of the specimens. In particular, the coding system is designed to permit the dimensions of each complex fibrous structure, and also whether these structures contain fibres longer than 5 µm, to be recorded. This approach permits later evaluations of the data to include considerations of particle respirability and comparisons with historical indices of asbestos exposure. Examples of the various types of morphological structure, and the manner in which these shall be recorded, are shown in figure C.1.

C.2 Structure definitions and treatment

Each fibrous structure that is a separate entity shall be designated as a primary structure. Each primary structure shall be designated as a fibre, bundle, cluster or matrix.

C.2.1 Fibre

Any particle with parallel or stepped sides, of minimum length 0,5 µm, and with an aspect ratio of 5/1 or greater, shall be defined as a fibre. For chrysotile asbestos, the single fibril shall be defined as a fibre. A fibre with stepped sides shall be assigned a width equal to the average of the minimum and maximum widths. This average shall be used as the width in determination of the aspect ratio.

C.2.2 Bundle

A grouping composed of apparently attached parallel fibres shall be defined as a bundle, with a width equal to an estimate of the mean bundle width, and a length equal to the maximum length of the structure. The overall aspect ratio of the bundle may have any value, provided that it contains individual constituent fibres with aspect ratios equal to or greater than 5/1. Bundles may exhibit diverging fibres at one or both ends.

C.2.3 Cluster

An aggregate of two or more randomly oriented fibres, with or without bundles, shall be defined as a cluster. Clusters occur as two varieties.

C.2.3.1 disperse cluster (type D): Disperse and open network, in which at least one of the individual fibres or bundles can be separately identified and its dimensions measured;

C.2.3.2 compact cluster (type C): Complex and tightly bound network, in which one or both ends of each individual fibre or bundle is (are) obscured, such that the dimensions of individual fibres and bundles cannot be unambiguously determined.

In practice, clusters can occur in which the characteristics of both types of cluster occur in the same structure. Where this occurs, the structure should be defined as a disperse cluster, and then a logical procedure should be followed by recording structure components according to the counting criteria. The procedure for treatment of clusters is illustrated by examples in figure C.2.

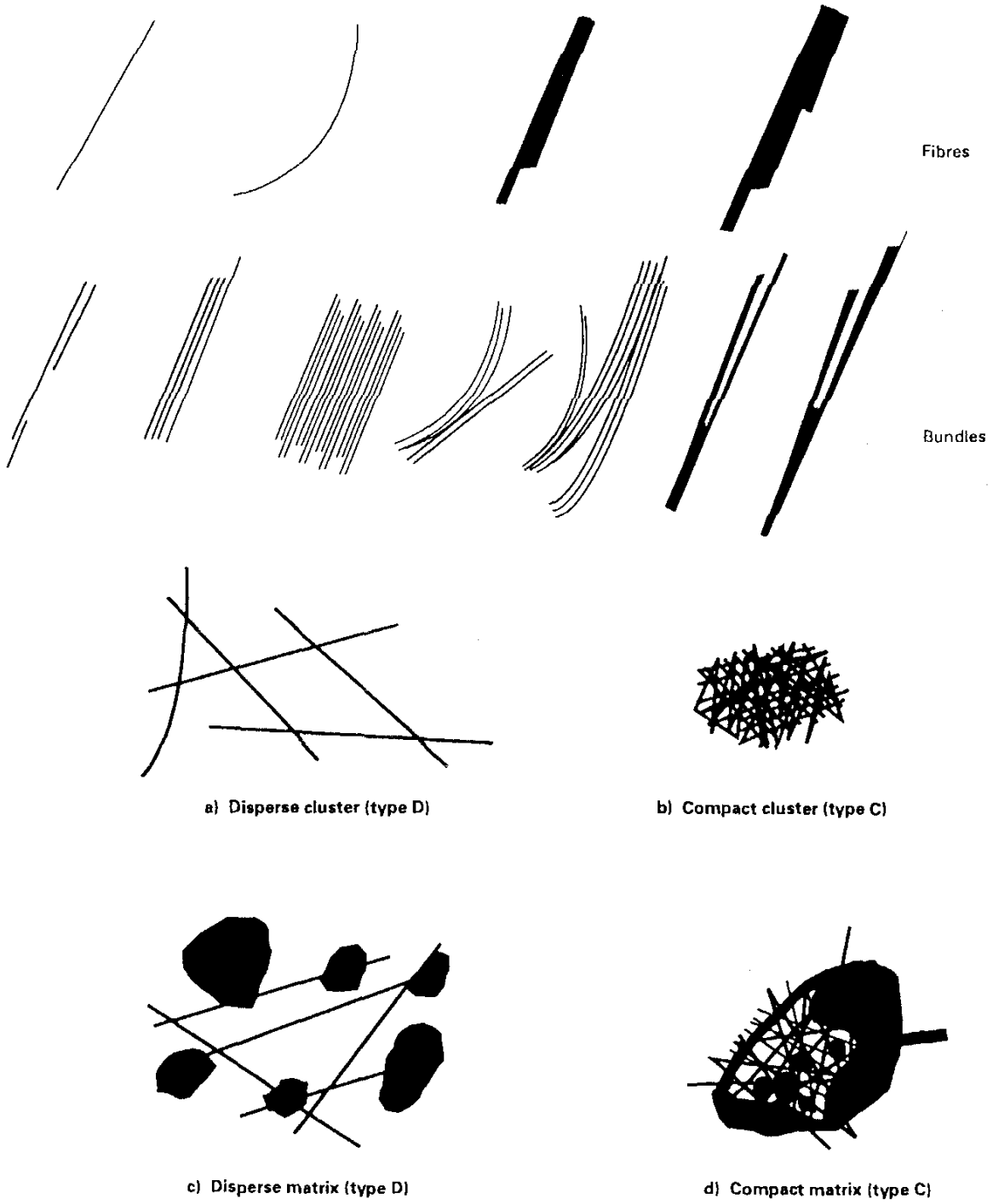
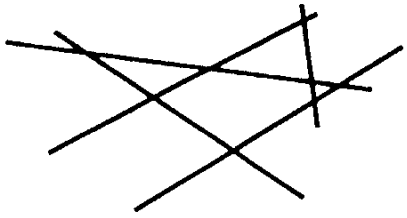


Figure C.1 — Fundamental morphological structure types

5 µm

Count as 1 compact cluster containing more than 9 fibres (all fibres shorter than 5 µm)

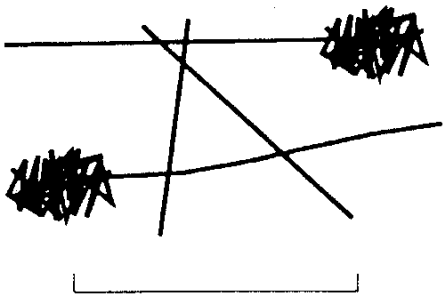
Record as CC+0



5 µm

Count as 1 disperse cluster consisting of 5 fibres, 4 of which are longer than 5 µm

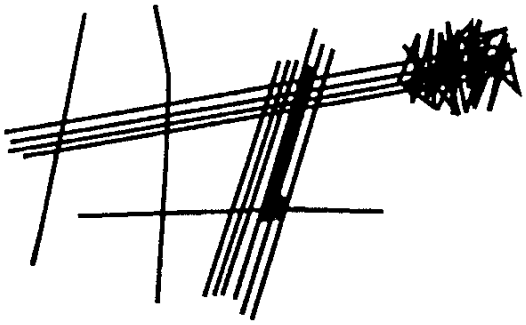
Record as CD54, followed by 5 fibres, each recorded as CF



5 µm

Count as 1 disperse cluster consisting of 4 fibres, 2 of which are longer than 5 µm, and 2 cluster residuals, each containing more than 9 fibres

Record as CD+2, followed by 4 fibres, each recorded as CF, and 2 cluster residual, each recorded as CR+0



5 µm

Count as 1 disperse cluster consisting of 3 fibres, 2 bundles, 1 of which is longer than 5 µm, and 1 cluster residual containing more than 9 fibres

Record as CD+1, followed by 3 fibres, each recorded as CF, 2 bundles, each recorded as CB, and 1 cluster residual recorded as CR+0

Figure C.2 — Examples of recording of complex asbestos clusters

C.2.4 Matrix

One or more fibres, or fibre bundles, may be attached to, or partially concealed by, a single particle or group of overlapping nonfibrous particles. This structure shall be defined as a matrix. The TEM image does not discriminate between particles which are attached to fibres, and those which have by chance overlapped in the TEM image. It is not known, therefore, whether such a structure is actually a complex particle, or whether it has arisen by a simple overlapping of particles and fibres on the filter.

Since a matrix structure may involve more than one fibre, it is important to define in detail how matrices shall be counted. Matrices exhibit different characteristics, and two types can be defined.

C.2.4.1 disperse matrix (type D): Structure consisting of a particle or linked group of particles, with overlapping or attached fibres or bundles in which at least one of the individual fibres or bundles can be separately identified and its dimensions measured.

C.2.4.2 compact matrix (type C): Structure consisting of a particle or linked group of particles, in which fibres or bundles can be seen either within the structure or projecting from it, such that the dimensions of individual fibres and bundles cannot be unambiguously determined.

In practice, matrices can occur in which the characteristics of both types of matrix occur in the same structure. Where this occurs, the structure should be assigned as a disperse matrix, and then a logical procedure should be followed by recording structure components according to the counting criteria. Examples of the procedure which shall be followed are shown in figure C.3.

C.2.5 Asbestos structure larger than 5 µm

Any fibre, bundle, cluster or matrix for which the largest dimension exceeds 5 µm. Asbestos structures larger than 5 µm do not necessarily contain asbestos fibres or bundles longer than 5 µm.

C.2.6 Asbestos fibre or bundle longer than 5 µm

An asbestos fibre of any width, or bundle of such fibres, which has a length exceeding 5 µm.

C.2.7 PCM equivalent structure

Any fibre, bundle, cluster or matrix with an aspect ratio of 3/1 or greater, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm. PCM equivalent structures do not necessarily contain fibres or bundles longer than 5 µm, or PCM equivalent fibres.

C.2.8 PCM equivalent fibre

Any particle with parallel or stepped sides, with an aspect ratio of 3/1 or greater, longer than 5 µm, and which has a diameter between 0,2 µm and 3,0 µm. For chrysotile, PCM equivalent fibres will always be bundles.

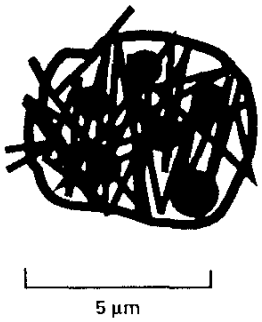
C.3 Other structure counting criteria

C.3.1 Structures which intersect grid bars

A structure which intersects a grid bar shall only be counted on two sides of the grid opening, as illustrated in figure C.4. Record the dimensions of the structure such that the obscured portions of components are taken to be equivalent to the unobscured portions, as shown by the broken lines in figure C.4. For example, the length of a fibre intersecting a grid bar is taken to be twice the unobscured length. Structures intersecting either of the other two sides shall not be included in the count.

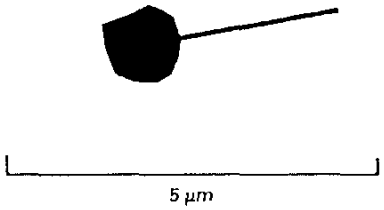
C.3.2 Fibres which extend outside the field of view

During scanning of a grid opening, count fibres which extend outside the field of view systematically, so as to avoid double-counting. In general, a rule should be established so that fibres extending outside the field of view in only two quadrants are counted. The procedure is illustrated by figure C.5. Measure the length of each of these fibre by moving the specimen to locate the other end of the fibre, and then return to the original field of view before continuing to scan the specimen. Fibres without terminations within the field of view shall not be counted.



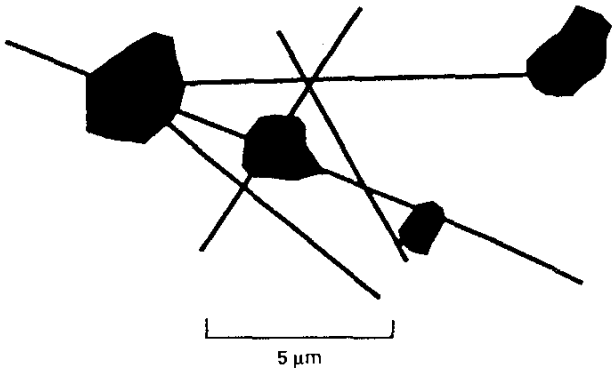
Count as 1 compact matrix, with all fibres shorter than 5 µm

Record as MC+0



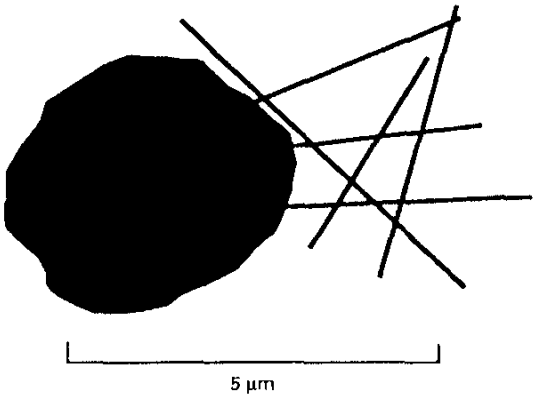
Count as 1 disperse matrix consisting of 1 fibre shorter than 5 µm

Record as MD10, followed by 1 fibre recorded as MF



Count as 1 disperse matrix consisting of 5 fibres, all longer than 5 µm

Record as MD55, followed by 5 fibres, each recorded as MF



Count as 1 disperse matrix, consisting if 3 fibres, 1 of which is longer than 5 µm, and 1 matrix residual containing 3 fibres

Record as MD61, followed by 3 fibres, each recorded as MF, and 1 matrix residual recorded as MR30

Figure C.3 — Examples of recording of complex asbestos matrices

© ISO

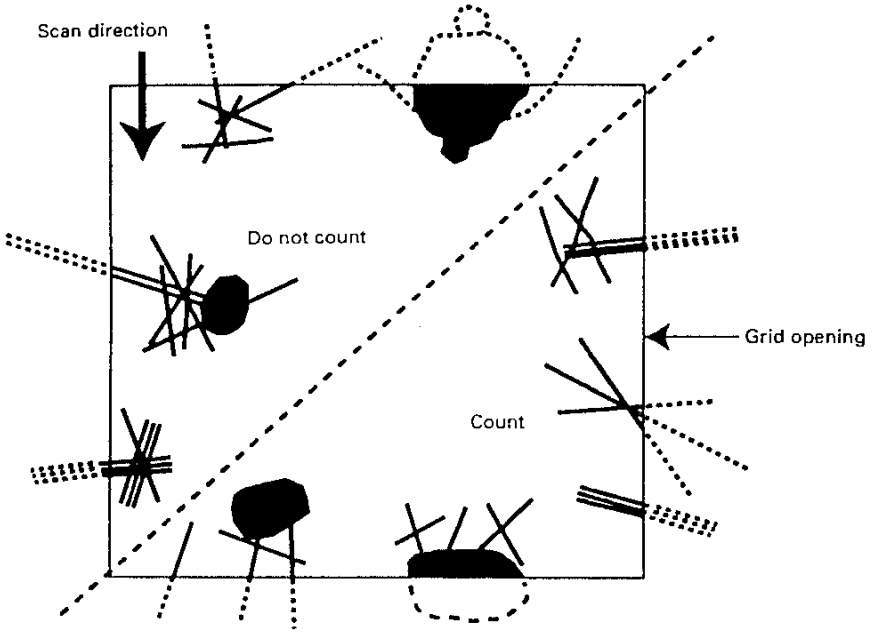


Figure C.4 — Example of counting of structures which intersect grid bars

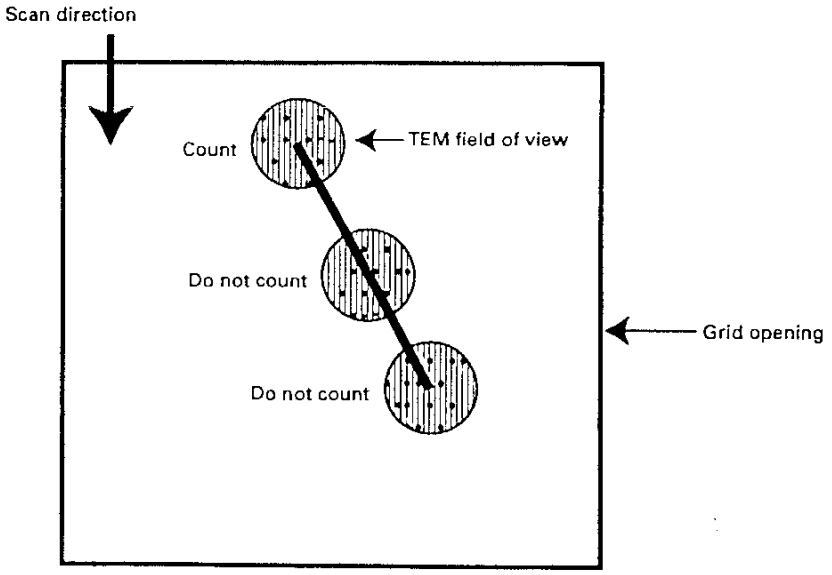


Figure C.5 — Example of counting of fibres which extend outside the field of view

C.4 Procedure for data recording

C.4.1 General

The morphological codes specified are designed to facilitate computer data processing, and to allow recording of a complete representation of the important features of each asbestos structure. The procedure requires that the microscopist classify each primary fibrous structure into one of the four fundamental categories: fibres, bundles, clusters and matrices.

C.4.2 Fibres

On the structure counting form, a fibre as defined in C.2.1 shall be recorded by the designation "F". If the fibre is a separately-counted part of a cluster or matrix, the fibre shall be recorded by the designation "CF", or "MF", depending on whether it is a component of a cluster or matrix.

C.4.3 Bundles

On the structure counting form, a bundle as defined in C.2.2 shall be recorded by the designation "B". If the bundle is a separately-counted part of a cluster or matrix, the bundle shall be recorded by the designation "CB", or "MB", depending on whether it is a component of a cluster or matrix.

C.4.4 Disperse clusters (type D)

On the structure counting form, an isolated cluster of type D as defined in C.2.3 shall be recorded by the designation "CD", followed by a two-digit number. The first digit represents the analyst's estimate of the total number of fibres and bundles comprising the structure. The digit shall be from 1 to 9, or designated as "+" if there are estimated to be more than 9 component fibres or bundles. The second digit shall represent, in the same manner, the total number of fibres and bundles longer than 5 µm contained in the structure. The overall dimensions of the cluster, in two perpendicular directions representing the maximum dimensions, shall be recorded. In order of decreasing length, up to 5 component fibres or bundles shall be separately recorded, using the codes "CF" (cluster fibre) and "CB" (cluster bundle). If, after accounting for prominent component fibres and bundles, a group of clustered fibres remains, this shall be recorded by the designation "CR" (cluster residual). If the remaining clustered fibres are present as more than one localized group, it may be necessary to record more than one cluster residual. Do not record more than 5 cluster residuals for any cluster. A cluster residual shall be measured and assigned a two-digit number, derived in the same manner as specified for the overall cluster. Optionally, if the number of component fibres and bundles in either the original cluster or the cluster residual is outside the range 1 – 9, additional information concerning the number of component fibres and bundles may be noted in the "comments" column.

C.4.5 Compact clusters (type C)

On the structure counting form, an isolated cluster of type C as defined in C.2.3 shall be recorded by the designation "CC", followed by a two-digit number. The two-digit number describing the numbers of component fibres and bundles shall be assigned in the same manner as for clusters of type D. The overall dimensions of the cluster in two perpendicular directions shall be recorded in the same manner as for clusters of type D. By definition, the constitutent fibres and bundles of compact clusters cannot be separately measured; therefore, no separate tabulation of component fibres or bundles can be made.

C.4.6 Disperse matrices (type D)

On the structure counting form, an isolated matrix of type D as defined in C.2.4 shall be recorded by the designation "MD", followed by a two-digit number. The two-digit number shall be assigned in the same manner as for clusters of type D. The overall dimensions of the matrix in two perpendicular directions shall be recorded in the same manner as for clusters of type D. In order of decreasing length, up to 5 component fibres or bundles shall be separately recorded, using the codes "MF" (matrix fibre) and "MB" (matrix bundle). If after accounting for prominent component fibres and bundles, matrix material containing asbestos fibres remains, this shall be recorded by the designation "MR" (matrix residual). If the remaining matrix fibres are present as more than one localized group, it may be necessary to record more than one matrix residual. Do not record more than 5 matrix residuals for any matrix. A matrix residual shall be measured and assigned a two-digit number, derived in the same manner as specified for the overall matrix. Optionally, if the number of component fibres or bundles in either the original matrix or the matrix residual is outside the range 1 – 9, additional information concerning the number of component fibres and bundles may be noted in the "comments" column.

C.4.7 Compact matrices (type C)

On the structure counting form, an isolated matrix of type C as defined in C.2.4 shall be recorded by the

designation "MC", followed by a two-digit number. The two-digit number shall be assigned in the same manner as for clusters of type D. The overall dimensions of the matrix in two perpendicular directions shall be recorded in the same manner as for clusters of type D. By definition, the constitutent fibres and bundles of compact matrices cannot be separately measured; therefore, no separate tabulation of component fibres or bundles can be made.

C.4.8 Procedure for recording of partially obscured fibres and bundles

The proportion of the length of a fibre or bundle that is obscured by other particulates shall be used as the basis for determining whether a fibre or bundle is to be recorded as a separate component or is to be considered as a part of a matrix of type C or part of a matrix residual. If the obscured length could not possibly be more than one-third of the total length, the fibre or bundle shall be considered a prominent feature to be separately recorded. The assigned length for each such partially obscured fibre or bundle shall be equal to the visible length plus the maximum possible contribution from the obscured portion. Fibres or bundles which appear to cross the matrix, and for which both ends can be located approximately, shall be included in the maximum of 5 and recorded according to the counting criteria as separate fibres or bundles. If the obscured length could be more than one third of the total length, the fibre or bundle shall be considered as a part of a compact matrix of type C or part of a matrix residual.

C.5 Special considerations for counting of PCM equivalent structures

Use 3/1 as the minimum aspect ratio for counting of PCM equivalent structures. This aspect ratio definition is required in order to achieve comparability of the results for this size range of structure with historical optical measurements, but use of this aspect ratio definition does not significantly affect the ability to interpret the whole fibre size distribution in terms of a minimum 5/1 aspect ratio. Some applications may require that a count be made of PCM equivalent structures only. The coding system permits discrimination between PCM equivalent structures that contain fibres and bundles longer than 5 µm and those that do not.

NOTE 16 In general, clusters and matrices will yield fewer components as the minimum dimensions specified for countable fibres are increased. Thus, it may be found that a particular structure yields a higher number of component fibres and bundles in a count for all fibre sizes than it does at a reduced magnification when only fibres and bundles longer than 5 µm are being counted. However, the requirement that component fibres and bundles be recorded in decreasing length order ensures that the data are consistent for a particular structure, regardless of the size category of fibres being counted and the magnification in use.

Annex D
(normative)

Fibre identification procedure

D.1 General

The criteria used for identification of asbestos fibres may be selected depending on the intended use of the measurements. In some circumstances, there can be a requirement that fibres shall be unequivocally identified as a specific mineral species. In other circumstances, there can be sufficient knowledge about the sample, so that rigorous identification of each fibre need not be carried out. The time required to perform the analysis, and therefore the cost of analysis, can vary widely depending on the identification criteria considered which are to be sufficiently definitive. The combination of criteria considered definitive for identification of fibres in a particular analysis shall be specified before the analysis is made, and this combination of criteria shall be referred to as the "level" of analysis. Various factors related to instrumental limitations and the character of the sample may prevent satisfaction of all of the specified fibre identification criteria for a particular fibre. Therefore, a record shall be made of the identification criteria which were satisfied for each suspected asbestos fibre included in the analysis. For example, if both ED and EDXA were specified to be attempted for definitive identification of each fibre, fibres with chrysotile morphology which, for some reason, do not give an ED pattern but which do yield an EDXA spectrum corresponding to chrysotile, are categorized in a way which conveys the level of confidence to be placed in the identification.

D.2 ED and EDXA techniques

D.2.1 General

Initially, fibres are classified into two categories on the basis of morphology: those fibres with tubular morphology, and those fibres without tubular morphology. Further analysis of each fibre is conducted using ED and EDXA methods. The following procedures should be used when fibres are examined by ED and EDXA.

The crystal structures of some mineral fibres, such as chrysotile, are easily damaged by the high current densities required for EDXA examination. Therefore, investigation of these sensitive fibres by ED should be completed before attempts are made to obtain EDXA spectra from the fibres. When more stable fibres, such as the amphiboles, are examined, EDXA and ED may be used in either order.

D.2.2 ED techniques

The ED technique can be either qualitative or quantitative. Qualitative ED consists of visual examination, without detailed measurement, of the general characteristics of the ED pattern obtained on the TEM viewing screen from a randomly oriented fibre. ED patterns obtained from fibres with cylindrical symmetry, such as chrysotile, do not change when the fibres are tilted about their axes, and patterns from randomly oriented fibres of these minerals can be interpreted quantitatively. For fibres which do not have cylindrical symmetry, only those ED patterns obtained when the fibre is oriented with a principal crystallographic axis closely parallel with the incident electron beam direction can be interpreted quantitatively. This type of ED pattern shall be referred to as a "zone-axis ED pattern". In order to interpret a zone-axis ED pattern quantitatively, it shall be recorded photographically and its consistency with known mineral structures shall be checked. A computer program may be used to compare measurements of the zone-axis ED pattern with corresponding data calculated from known mineral structures. The zone-axis ED pattern obtained by examination of a fibre in a particular orientation can be insufficiently specific to permit unequivocal identification of the mineral fibre, but is is often possible to tilt the fibre to another angle and to record a different ED pattern corresponding to another zone-axis. The angle between the two zone-axes can also be checked for consistency with the structure of a suspected mineral.

For visual examination of the ED pattern, the camera length of the TEM should be set to a low value of approximately 250 mm and the ED pattern should then be viewed through the binoculars. This procedure minimizes the possible degradation of the fibre by the electron irradiation. However, the pattern is distorted by the tilt angle of the viewing screen. A camera length of at least 2 m should be used when

the ED pattern is recorded, if accurate measurement of the pattern is to be possible. It is necessary that, when obtaining an ED pattern to be evaluated visually or to be recorded, the sample height shall be properly adjusted to the eucentric point and the image shall be focussed in the plane of the selected area aperture. If this is not done, there may be some components of the ED pattern which do not originate from the selected area. In general, it will be necessary to use the smallest available ED aperture.

For accurate measurements of the ED pattern, an internal calibration standard shall be used. A thin coating of gold, or another suitable calibration material, shall be applied to the underside of the TEM specimen. This coating may be applied either by vacuum evaporation or, more conveniently, by sputtering. The polycrystalline gold film yields diffraction rings on every ED pattern and these rings provide the required calibration information.

To form an ED pattern, move the image of the fibre to the centre of the viewing screen, adjust the height of the specimen to the eucentric position, and insert a suitable selected area aperture into the electron beam so that the fibre, or a portion of it, occupies a large proportion of the illuminated area. The size of the aperture and the portion of the fibre shall be such that particles other than the one to be examined are excluded from the selected area. Observe the ED pattern through the binoculars. During the observation, the objective lens current should be adjusted to the point where the most complete ED pattern is obtained. If an incomplete ED pattern is still obtained, move the particle around within the selected area to attempt to optimize the ED pattern, or to eliminate possible interferences from neighbouring particles.

If a zone-axis ED analysis is to be attempted on the fibre, the sample shall be mounted in the appropriate holder. The most convenient holder allows complete rotation of the specimen grid and tilting of the grid about a single axis. Rotate the sample until the fibre image indicates that the fibre is oriented with its length coincident with the tilt axis of the goniometer, and adjust the sample height until the fibre is at the eucentric position. Tilt the fibre until an ED appears which is a symmetrical, two dimensional array of spots. The recognition of zone-axis alignment conditions requires some experience on the part of the operator. During tilting of the fibre to obtain zone-axis conditions, the manner in which the intensities of the spots vary should be observed. If weak reflections occur at some points on a matrix of strong reflections, the possibility of twinning or multiple diffraction exists, and some caution should be exercised in the selection of diffraction spots for measurement and interpretation. A full discussion of electron diffraction and multiple diffraction can be found in the references by J.A. Gard [11] P.B. Hirsch *et al* [14] and H.R. Wenck [42] included in annex J. Not all zone-axis patterns which can be obtained are definitive. Only those which have closely spaced reflections corresponding to low indices in at least one direction should be recorded. Patterns in which all d-spacings are less than about 0,3 nm are not definitive. A useful guideline is that the lowest angle reflections should be within the radius of the first gold diffraction ring (111), and that patterns with smaller distances between reflections are usually the most definitive.

Five spots, closest to the centre spot, along two intersecting lines of the zone-axis pattern shall be selected for measurement, as shown in figure D.1. The distances of these spots from the centre spot and the four angles shown provide the required data for analysis. Since the centre spot is usually very overexposed, it does not provide a well-defined origin for these measurements. The required distances shall therefore be obtained by measuring between pairs of spots symmetrically disposed about the centre spot, preferably separated by several repeat distances. The distances shall be measured with a precision of better than 0,3 mm, and the angles to a precision of better than 2,5°. The diameter of the first or second ring of the calibration pattern (111 and 200) shall also be measured with a precision of better than 0,3 mm.

Using gold as the calibration material, the radius-based camera constant is given by

$$\lambda L = 0{,}117\ 74D \text{ mm·nm (first ring)}$$

$$\lambda L = 0{,}101\ 97D \text{ mm·nm (second ring)}$$

D.2.3 EDXA measurements

Interpretation of the EDXA spectrum may be either qualitative or quantitative. For qualitative interpretation of a spectrum, the X-ray peaks originating from the elements in the fibre are recorded. For quantitative interpretation, the net peak areas, after background subtraction, are obtained for the X-ray peaks originating from the elements in the fibre. This method provides quantitative interpretation for those minerals which contain silicon.

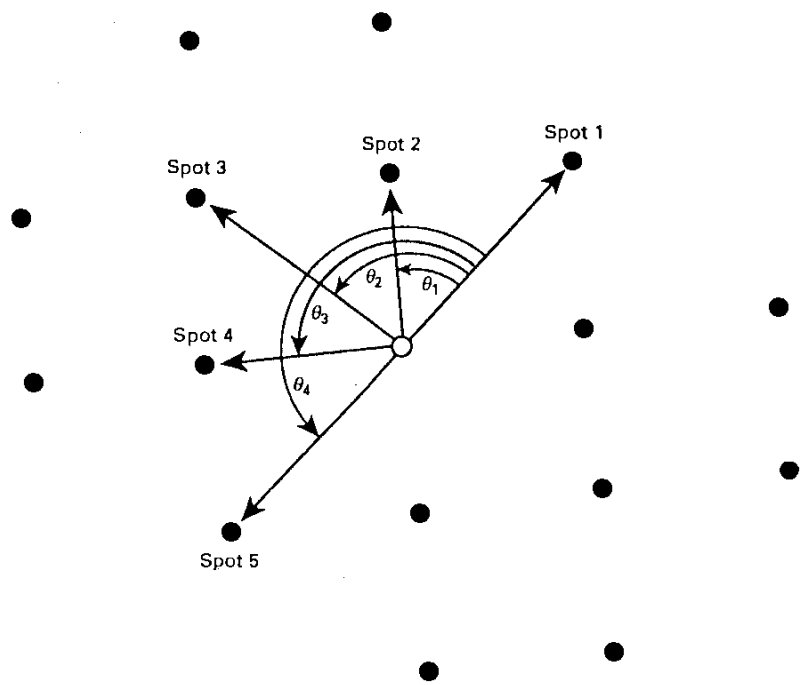


Figure D.1 — Example of measurement of zone-axis SAED patterns

To obtain an EDXA spectrum, move the image of the fibre to the centre of the screen and remove the objective aperture. Select an appropriate electron beam diameter and deflect the beam so that it impinges on the fibre. Depending on the instrumentation, it may be necessary to tilt the specimen towards the X-ray detector and, in some instruments, to use the Scanning Transmission Electron Microscopy (STEM) mode of operation.

The time for acquisition of a suitable spectrum varies with the fibre diameter, and also with instrumental factors. For quantitative interpretation, spectra should have a statistically valid number of counts in each peak. Analyses of small diameter fibres which contain sodium are the most critical, since it is in the low energy range that the X-ray detector is least sensitive. Consequently, it is necessary to acquire a spectrum for a period that is sufficiently long for the sodium to be detected in such fibres. It has been found that satisfactory quantitative an analyses can be obtained if acquisition is continued until the background subtracted silicon $K\alpha$ peak integral exceeds 10 000 counts. The spectrum should then be manipulated to subtract the background and to obtain the net areas of the elemental peaks.

After quantitative EDXA classification of some fibres by computer analysis of the net peak areas, it may be possible to classify further fibres in the same sample on the basis of comparison of spectra at the intrument. Frequently, visual comparisons can be made after somewhat shorter acquisition times.

D.3 Interpretation of fibre analysis data

D.3.1 Chrysotile

The morphological structure of chrysotile is characteristic, and with experience, can be recognized readily. However, a few holder minerals have a similar appearance, and morphological observation by itself is inadequate for most samples. The ED pattern obtained from chrysotile is quite specific for this mineral if the specified characteristics of the pattern correspond to those from reference chrysotile. However, depending on the past history of the fibre, and on a number of other factors, the crystal structure of a particular fibre may be damaged, and it may not yield and ED pattern. In this case, the EDXA spectrum may be the only data available to supplement the morphological observations.

D.3.2 Amphiboles

Since the fibre identification procedure for asbestos fibres other than chrysotile can be involved and time-consuming, computer programmes, such as that developed by B.L. Rhoades (see annex J, reference

[32]), are recommended for interpretation of zone-axis ED patterns. The published literature contains composition and crystallographic data for all of the fibrous minerals likely to be encountered in TEM analysis of air samples, and the compositional and structural data from the unknown fibre should be compared with the published data. Demonstration that the measurements are consistent with the data for a particular test mineral does not uniquely identify the unknown, since the possibility exists that data from other minerals may also be consistent. It is, however, unlikely that a mineral of another structural class could yield data consistent with that from an amphibole fibre identified by quantitative EDXA and two zone-axis ED patterns.

Suspected amphibole fibres should be classified initially on the basis of chemical composition. Either qualitative or quantitative EDXA information may be used as the basis for this classification. From the published data on mineral compositions, a list of minerals which are consistent in composition with that measured for the unknown fibre should be compiled. To proceed further, it is necessary to obtain the first zone-axis ED pattern, according to D.2.2.

It is possible to specify a particular zone-axis pattern for identification of amphibole, since a few patterns are often considered to be characteristic. Unfortunately, for a fibre with random orientation on a TEM grid, no specimen holder and goniometer currently available will permit convenient and rapid location of two preselected zone-axes. The most practical approach has been adopted, which is to accept those low index patterns which are easily obtained, and then to test their consistency with the structures of the minerals already preselected on the basis of the EDXA data. Even the structures of non-amphibole minerals in this preselected list shall be tested against the zone-axis data obtained for the unknown fibre, since non-amphibole minerals in some orientations may yield similar patterns consistent with amphibole structures.

The zone-axis ED interpretation shall include all minerals previously selected from the mineral data file as being chemically compatible with the EDXA data. This procedure will usually shorten the list of minerals for which solutions have been found. A second set of zone-axis data from another pattern obtained on the same fibre can then be processed, either as further confirmation of the identification, or to attempt elimination of an ambiguity. In addition, the angle measured between the orientations of the two zone-axes can be checked for consistency with the structures of the minerals. Caution should be exercised in rationalizing the inter-zone-axis angle, since if the fibre contains $\vec{c}$-axis twinning, the two zone-axis ED patterns may originate from the separate twin crystals. In practice, the full identification procedure will normally be applied to very few fibres, unless precise identification of all fibres is required for a particular reason.

D.4 Fibre classification categories

It is not always possible to proceed to a definitive identification of a fibre; this may be due to instrumental limitations or to the actual nature of the fibre. In many analyses, a definitive identification of each fibre may not actually be necessary if there is other knowledge available about the sample, or if the concentration is below a level of interest. The analytical procedure shall therefore take into account both instrumental limitations and varied analytical requirements. Accordingly, a system for fibre classification is used to permit accurate recording of data. The classifications are shown in tables D.1 and D.2, and are directed towards identification of chrysotile and amphibole respectively. Fibres shall be reported in these categories.

The general principle to be followed in this analytical procedure is first to define the most specific fibre classification which is to be attempted, or the "level" of analysis to be conducted. Then, for each fibre examined, record the classification which is actually achieved. Depending on the intended use of the results, criteria for acceptance of fibres as "identified" can then be established at any time after completion of the analysis.

In an unknown sample, chrysotile will be regarded as confirmed only if a recorded, calibrated ED pattern from one fibre in the CD categories is obtained, or if measurements of the ED pattern are recorded at the instrument. Amphibole will be regarded as confirmed only by obtaining recorded data which indicates exclusively the presence of amphiboles for fibres classified in the AZQ, AZZ or AZZQ categories.

Table D.1 — Classification of fibres with tubular morphology

Category	Description
TM	Tubular Morphology, not sufficiently characteristic for classification as chrysotile
CM	Characteristic Chrysotile Morphology
CD	Chrysotile SAED pattern
CQ	Chrysotile composition by Quantitative EDXA
CMQ	Chrysotile Morphology and composition by Quantitative EDXA
CDQ	Chrysotile SAED pattern and composition by Quantitative EDXA
NAM	Non-Asbestos Mineral

Table D.2 — Classification of fibres without tubular morphology

Category	Description
UF	Unidentified Fibre
AD	Amphibole by random orientation SAED (shows layer pattern of 0,53 nm spacing)
AX	Amphibole by qualitative EDXA. Spectrum has elemental components consistent with amphibole
ADX	Amphibole by random orientation SAED and qualitative EDXA
AQ	Amphibole by Quantitative EDXA
AZ	Amphibole by one Zone-axis SAED pattern
ADQ	Amphibole by random orientation SAED and Quantitative EDXA
AZQ	Amphibole by one Zone-axis SAED pattern and Quantitative EDXA
AZZ	Amphibole by two Zone-axis SAED patterns, with consistent interaxial angle
AZZQ	Amphibole by two Zone-axis SAED patterns, with consistent interaxial angle, and Quantitative EDXA
NAM	Non-Asbestos Mineral

D.4.1 Procedure for classification of fibres with tubular morphology suspected to be chrysotile

Occasionally, fibres are encountered which have tubular morphology similar to that of chrysotile, but which cannot be characterized further either by ED or EDXA. They may be non-crystalline, in which case ED techniques are not useful, or they may be in a position on the grid which does not permit an EDXA spectrum to be obtained. Alternatively, the fibre may be of organic origin, but the morphology and composition may not be sufficiently definitive enough to be disregarded. Accordingly, there is a requirement to record each fibre, and to specify how confidently each fibre can be identified. Classification of fibres will meet with various degrees of success. Figure D.2 shows the classification procedure to be used for fibres which display any tubular morphology. The chart is self explanatory, and every fibre is either rejected as a non-asbestos mineral (NAM), or classified in some way which by some later criterion could still contribute to the chrysotile fibre count.

Morphology is the first consideration, and if this is not similar to that usually seen in chrysotile standard samples, designate the initial classification as TM. Regardless of the doubtful morphology, examine the fibre by ED and EDXA methods according to figure D.2. Where the morphology is more definitive, it may be possible to classify the fibre as having chrysotile morphology (CM).

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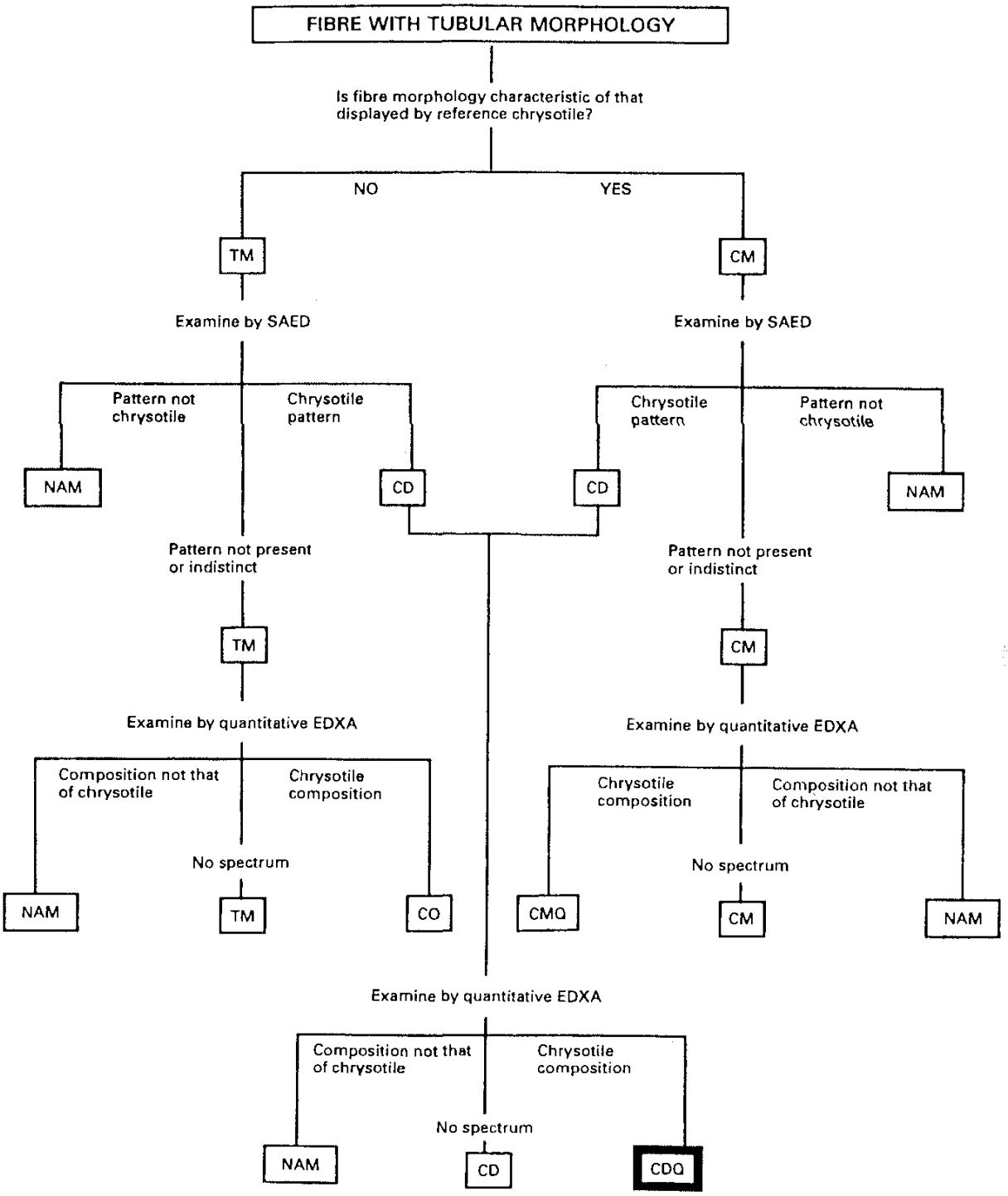

Figure D.2 — Classification chart for fibre with tubular morphology

For classification as CM, the morphological characteristics required are the following:

a) the individual fibrils should have high aspect ratios exceeding 5/1, and be about 30 nm to 40 nm in diameter;

b) the electron scattering power of the fibre at 60 kV to 100 kV accelerating potential should be sufficiently low for the internal structure to be visible;

c) there should be some evidence of an internal structure suggesting a tubular appearance similar to that shown by reference UICC chrysotile, which may degrade in the electron beam.

Examine every fibre having these morphological characteristics by the ED technique, and classify as chrysotile by ED (CD) only those which give diffraction patterns with the precise characteristics shown in figure D.3. The relevant features in this pattern for identification of chrysotile are as follows:

a) the (002) reflections should be examined to determine that they correspond closely to a spacing of 0,73 nm;

b) the layer line repeat distance should correspond to 0,53 nm;

c) there should be "streaking" of the (110) and (130) reflections.

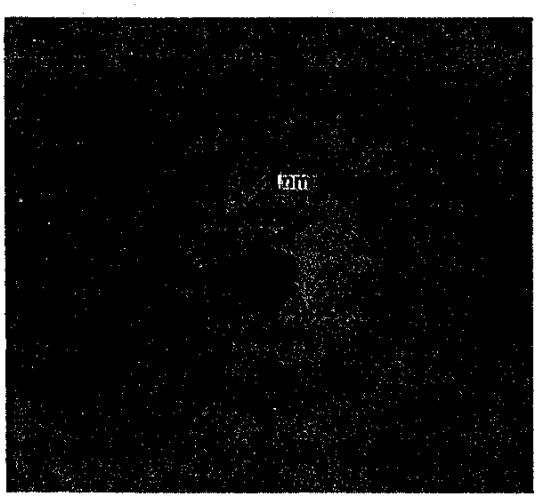

Figure D.3 — Chrysotile SAED pattern

Using the millimetre calibrations on the TEM viewing screen, these observations can readily be made at the instrument. If documentary proof of fibre identification is required, record a TEM micrograph of at least one representative fibre, and record its ED pattern on a separate film or plate. This film or plate shall also carry calibration rings from a known polycrystalline substance such as gold. This calibrated pattern is the only documentary proof that the particular fibre is chrysotile, and not some other tubular or scrolled species such as halloysite, palygorskite, talc or vermiculite. The proportion of fibres which can be successfully identified as chrysotile by ED is variable, and to some extent dependent on both the instrument and the procedures of the operator. The fibres that fail to yield an identifiable ED pattern will remain in the TM or CM categories unless they are examined by EDXA.

In the EDXA analysis of chrysotile there are only two elements which are relevant. For fibre classification, the EDXA analysis shall be quantitative. If the spectrum displays prominent peaks from magnesium and silicon, with their areas in the appropriate ratio, and with only minor peaks from other elements, classify the fibre as chrysotile by quantitative EDXA, in the categories CO, CMQ, or CDQ, as appropriate.

D.4.2 Procedure for classification of fibres without tubular morphology, suspected to be amphibole

Every particle without tubular morphology and which is not obviously of biological origin, with an aspect ratio of 5/1 or greater, and having parallel or stepped sides, shall be considered as a suspected amphibole fibre. Further examination of the fibre by ED and EDXA techniques will meet with a variable degree of success, depending on the nature of the fibre and on a number of instrumental limitations. It will not be possible to identify every fibre completely, even if time and cost are of no concern. Moreover, confirmation of the presence of amphibole can be achieved only by quantitative interpretation of zone-axis ED patterns, a very time-consuming procedure. Accordingly, for routine samples from unknown sources, this analytical procedure limits the requirement for zone-axis ED work to a minimum of one fibre representative of each compositional class reported. In some samples, it may be necessary to identify more fibres by the zone-axis technique. When analysing samples from well-characterized sources, the cost of identification by zone-axis methods may not be justified.

The 0,53 nm layer spacing of the random orientation ED pattern is not by itself diagnostic for amphibole, However, the presence of $\vec{c}$-axis twinning in many fi-

bres leads to contributions to the layers in the patterns by several individual parallel crystals of different axial orientations. This apparently random positioning of the spots along the layer lines, if also associated with a high fibre aspect ratio, is a characteristic of amphibole asbestos, and thus has some limited diagnostic value. If a pattern of this type is not obtained, the identity of the fibre is still ambiguous, since the absence of a recognizable pattern may be a consequence of an unsuitable orientation relative to the electron beam, or the fibre may be some other mineral species.

Figure D.4 shows the fibre classification chart to be used for suspected amphibole fibres. This chart shows all the classification paths possible in analysis of a suspected amphibole fibre, when examined systematically by ED and EDXA. Two routes are possible, depending on whether an attempt to obtain an EDXA spectrum or a random orientation ED pattern is made first. The normal procedure for analysis of a sample of unknown origin will be to examine the fibre by random orientation ED, qualitative EDXA, quantitative EDXA, and zone-axis ED, in this sequence. The final fibre classification assigned will be defined either by successful analysis at the maximum required level, or by the instrumental limitations. Any instrumental limitations which affect the quality of the results sh be noted. Record the maximum classification achieved for each fibre on the counting sheet in the appropriate column. The various classification categories can then be combined later in any desired way for calculation of the fibre concentration. The complete record of the results obtained when attempting to identify each fibre can also be used to re-assess the data if necessary.

In the unknown sample, zone-axis analysis will be required if the presence of amphibole is to be unequivocally confirmed. For this level of analysis, attempt to raise the classification of every suspected amphibole fibre to the ADQ category by inspection of the random orientation ED pattern and the EDXA spectrum. In addition, examine at least one fibre from each type of suspected amphibole found by zone-axis methods to confirm their identification. In most cases, because information exists about possible sources of asbestos in close proximity to the air sampling location, some degree of ambiguity of identification can be accepted. Lower levels of analysis can therefore be accepted for these situations.

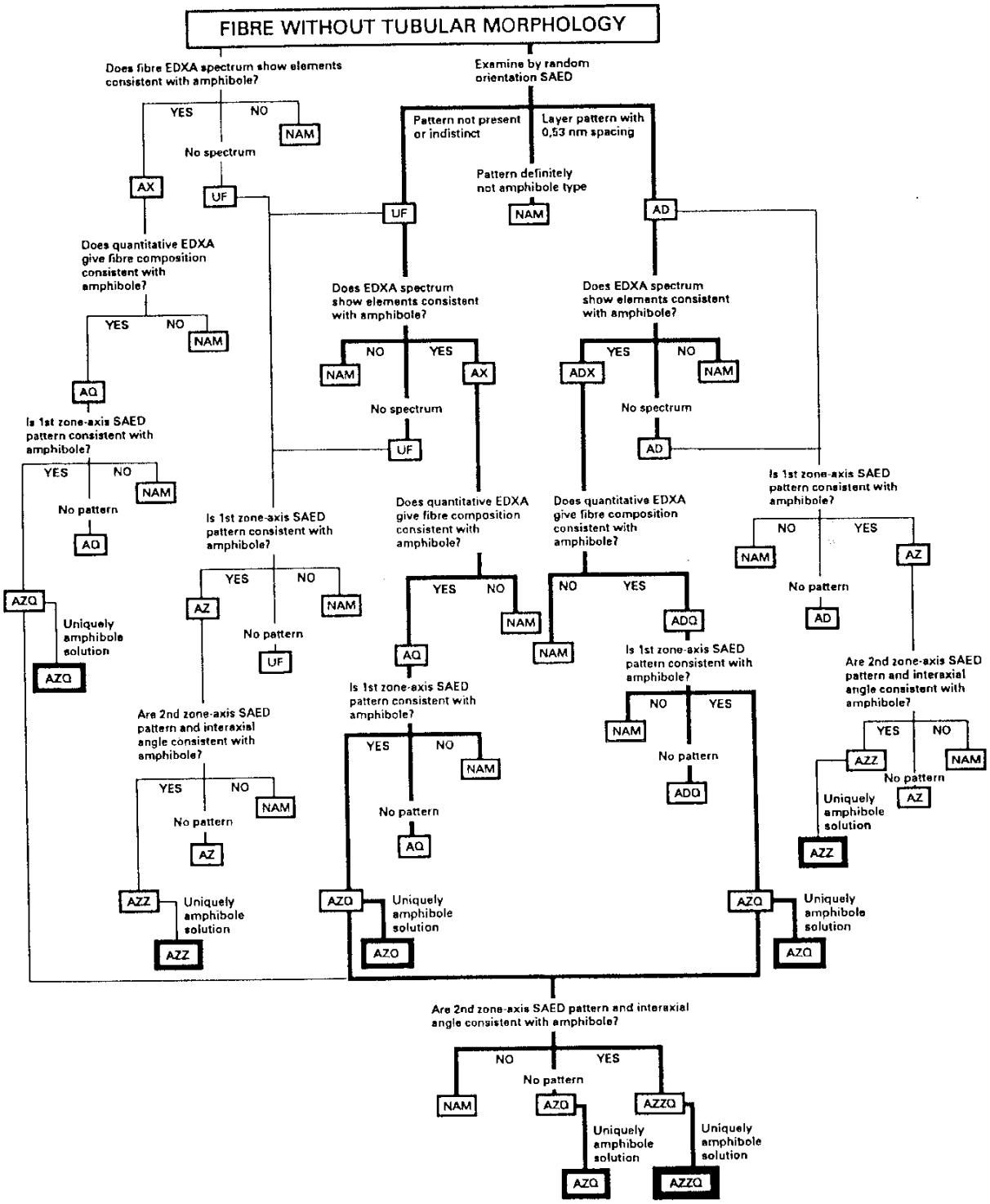

Figure D.4 — Classification chart for fibre without tubular morphology

Annex E
(normative)

Determination of the concentrations of asbestos fibres and bundles longer than 5 µm, and PCM equivalent asbestos fibres

In order to provide increased statistical precision and improved analytical sensitivity for those asbestos fibres and bundles longer than 5 µm, it may be decided to perform additional fibre counting at a lower magnification, taking account only into fibres and bundles within this dimensional range. The result shall be specified as "number of asbestos fibres and bundles longer than 5 µm". For this examination, use a magnification of approximately × 10 000, and continue to assign a morphological code to each structure according to the procedures specified in annex C. Record fibres and bundles only if their lengths exceed 5 µm. Record cluster and matrix components only if their lengths exceed 5 µm.

It may also be decided to provide increased statistical precision and improved analytical sensitivity for fibrous structures longer than 5 µm, with diameters between 0,2 µm and 3,0 µm, which have historically been the basis of risk estimation in the occupational environment (PCM equivalent asbestos fibres). Use a magnification of approximately × 5 000 for this extended fibre count. The result shall be specified as "number of PCM equivalent asbestos fibres". Asbestos structures within this dimensional range do not necessarily incorporate asbestos fibres or bundles longer than 5 µm.

Continue the extended sample examination until 100 asbestos structures have been counted, or until a sufficient area of the specimen has been examined to achieve the desired analytical sensitivity calculated according to table 1. The grid openings examined shall be divided approximately equally between a minimum of two specimen grids.

NOTES

17 The specimen area corresponding to the area of filter examined in the PCM fibre counting methods is 0,785 mm^2, and is equivalent to approximately 100 grid openings of a 200 mesh grid.

18 Some National Standards require that asbestos fibres longer than 2,5 µm, with diameters between 0,2 µm and 3,0 µm be counted. Use a magnification of × 5 000 for counting fibres within these dimensional ranges.

19 The minimum aspect ratio for definition of a fibre in PCM fibre counting methods and in some National Standards is 3/1. Use of a 3/1 aspect ratio is permitted in this International Standard, if this aspect ratio is mentioned in the test report.

The test reports shall include all of the items listed in clause 11.

Annex F
(normative)

Calculation of results

F.1 General

The results should be calculated using the procedures specified below. The results can be conveniently calculated using a computer programme.

F.2 Test for uniformity of distribution of fibrous structures on TEM grids

A chek shall be made using the chi-square test, to determine whether the asbestos structures found on individual grid openings are randomly and uniformly distributed among the grid openings. If the total number found in k grid openings is n, and the areas of the k individual frid openings are designated A_1 to A_k, then the total area of TEM specimen examined is

$$A = \sum_{i=1}^{i=k} A_i$$

The fraction of the total area examined which is represented by the individual grid opening area, p_i, is given by A_i/A. If the structures are randomly and uniformly dispersed over the k grid openings examined, the expected number of structures falling in one grid opening with area A_i is np_i. If the observed number of structures found on that grid opening is n_i, then

$$x^2 = \sum_{i=1}^{i=k} \frac{(n_i - np_i)^2}{np_i}$$

This value shall be compared with significance points of the chi-square distribution, having $(k - 1)$ degrees of freedom. Significance levels lower than 0,1 % may be cause for the sample analysis to be rejected, since this correspond to a very inhomogeneous deposit. If the structure count fails this test, the precision of the result will be uncertain, and if new air samples cannot be collected, additional grid openings may be examined or the sample may be prepared by an indirect method.

F.3 Calculation of the analytical sensitivity

Calculate the required analytical sensitivity S, expressed in number of structures per litre, using the following equation:

$$S = \frac{A_f}{kA_g V}$$

where

A_f is the area, in square millimetres, of sample collection filter;

A_g is the area, in square millimetres, of TEM specimen grid opening;

k is the number of grid openings examined;

V is the volume of air sampled, in litres.

F.4 Calculation of the mean and confidence interval of the structure concentration

In the structure count made according to this International Standard, a number of grid openings have been sampled from a population of grid openings, and it is required to determine the mean grid opening structure count for the population on the basis of this small sample. The interval about the sample mean which, with 95 % confidence, contains the population mean, is also required.

F.4.1 Calculation of the mean structure concentration

Calculate the mean structure concentration C, expressed in number of structures per litre, using the following equation:

$$C = Sn$$

where

S is the analytical sensitivity, expressed in number of structures per litre;

n is the total number of structures found on all grid openings examined.

F.4.2 Calculation of confidence intervals

The distribution of structures on the grid openings should theoretically approximate to a Poisson distribution. Because of fibre aggregation and size-dependent identification effects, the actual structure counting data often does not conform to the Poisson distribution, particularly at high structure counts. An assumption that the structure counting data are distributed according to the Poisson distribution can therefore lead to confidence intervals narrower than are justified by the data. Moreover, if the Poisson distribution is assumed, the variance is related only to the total number of structures counted. Thus, a particular structure count conducted on one grid opening is considered to have the same confidence interval as that for the same number of structures found on many grid openings. However, the area of sample actually counted is very small in relation to the total area of the filter, and for this reason, structures shall be counted on a minimum of four grid openings taken from different areas of the filter in order to ensure that a representative evaluation of the deposit is made.

At high structure counts, where there are adequate numbers of structures per grid opening to allow a sample estimate of the variance to be made, the distribution can be approximated to a Gaussian, with independent values for the mean and variance. Where the sample estimate of variance exceeds that implicit in the Poissonian assumption, use of Gaussian statistics with the variance defined by the actual data is the most conservative approach to calculation of confidence intervals.

At low structure counts, it is not possible to obtain a reliable sample estimate of the variance, and the distribution also becomes asymmetric but not necessarily Poissonian. For 30 structures and below, the distribution becomes asymmetric enough for the fit to a Gaussian to no longer be a reasonable one, and estimates of sample variance are unreliable. Accordingly, for counts below 31 structures, the assumption of a Poisson distribution shall be made for calculation of the confidence intervals.

F.4.3 Example of calculation of Poissonian 95 % confidence intervals

For total structure counts less than 4, the lower 95 % confidence limit corresponds to less than 1 structure. Therefore, it is not meaningful to quote lower confidence interval points for structure counts of less than 4, and the result shall be recorded as "less than" the corresponding one-sided upper 95 % confidence limit of the Poisson distribution, as follows:

0 structure ≙ 2,99 times the analytical sensitivity

1 structure ≙ 4,74 times the analytical sensitivity

2 structures ≙ 6,30 times the analytical sensitivity

3 structures ≙ 7,75 times the analytical sensitivity

For total counts exceeding 4, the 95 % confidence interval shall be calculated using the values shown in table F.1. Table F.1 gives the upper and lower limits of the two-sided Poissonian 95 % confidence interval for structure counts up to 470.

F.4.4 Example of calculation of Gaussian 95 % confidence intervals

Calculate the sample estimate of variance s^2 using the following equation:

$$s^2 = \frac{\sum_{i=1}^{i=k} (n_i - np_i)^2}{(k-1)}$$

where

n_i is the number of structures on the ith grid opening;

n is the total number of structures found in k grid openings;

p_i is the fraction of the total area examined represented by the ith grid opening;

k is the number of grid openings examined.

If the mean value of the structure count is calculated to be n, the upper and lower values of the Gaussian 95 % confidence interval are given respectively by

$$L_u = \frac{n}{k} + \frac{ts}{\sqrt{k}}$$

and

$$L_l = \frac{n}{k} - \frac{ts}{\sqrt{k}}$$

where

L_u is the upper 95 % confidence limit;

L_l is the lower 95 % confidence limit;

n is the total number of structures in all grid openings examined;

t is the value of Student's test (probability 0,975) for $(k - 1)$ degrees of freedom;

s is the standard deviation (square root of sample estimate of variance);

k is the number of grid openings examined.

F.4.5 Summary of procedure for calculation of results

In summary, structure counting data shall be calculated as follows:

No structures detected

The structure concentration shall be reported as less than the concentration equivalent ot the one-sided upper 95 % confidence limit of the Poisson distribution. This is equal to 2,99 times the analytical sensitivity.

From 1 to 3 structures

When 1 to 3 structures are counted, the result shall be reported as less than the corresponding one-sided upper 95 % confidence limit for the Poisson distribution. These are

1 structure ≙ 4,74 times the analytical sensitivity

2 structures ≙ 6,30 times the analytical sensitivity

3 structures ≙ 7,75 times the analytical sensitivity

From 4 to 30 structures

The mean structure concentration and the 95 % confidence intervals shall be reported on the basis of the Poissonian assumption, using the values shown in table F.1.

More than 30 structures

When more 30 structures are counted, both the Gaussian 95 % confidence interval and the Poissonian 95 % confidence interval shall be calculated. The larger of these two intervals shall be used to express the precision of the structure concentration. When the Gaussian 95 % confidence interval is selected for data reporting, the Poissonian 95 % confidence interval shall also be mentioned.

F.5 Calculation of structure length, width, and aspect ratio distributions

The distributions all approximate to logarithmic-normal, and therefore the size range intervals for calculation of the distribution shall be spaced logarithmically. The other characteristics required for the choice of size intervals are that they should allow for a sufficient number of size classes, while still retaining a statistically valid number of structures in each class. Interpretation is also facilitated if each size class repeats at 10 intervals, and if 5 µm is a size class boundary. A ratio from one class to the next of 1,468 satisfies all of these requirements and this value shall be used. The distributions, being approximately logarithmic-normal, when presented graphically, shall be plotted using a logarithmic ordinate scale and a Gaussian abscissa.

F.5.1 Calculation of structure length cumulative number distribution

This distribution allows the fraction of the total number of structures either shorter or longer than a given length to be determined. It is calculated using the following equation:

$$C(P)_k = \frac{\sum_{i=1}^{i=k} n_i}{\sum_{i=1}^{i=P} n_i} \times 100$$

where

$C(P)_k$ is the cumulative number percentage of structures which have lengths less than the upper bound of the kth class;

n_i is the number of structures in the ith length class;

P is the total number of length classes.

F.5.2 Calculation of structure width cumulative number distribution

This distribution allows the fraction of the total number of structures either narrower or wider than a given width to be determined. It is calculated in a similar way to that used in F.5.1, but using the structure widths.

F.5.3 Calculation of structure aspect ratio cumulative number distribution

This distribution allows the fraction of the total number of structures which have aspect ratios either smaller or larger than a given aspect ratio to be determined. It is calculated in a similar way to that used in F.5.1, but using the structure aspect ratios.

Table F.1 — Upper and lower limits of the Poissonian 95 % confidence interval of a count

Structure count	Lower limit	Upper limit	Structure count	Lower limit	Upper limit	Structure count	Lower limit	Upper limit
0	0	3,689[1]	46	33,678	61,358	92	74,164	112,83
1	0,025	5,572	47	34,534	62,501	93	75,061	113,94
2	0,242	7,225	48	35,392	63,642	94	75,959	115,04
3	0,619	8,767	49	36,251	64,781	95	76,858	116,14
4	1,090	10,242	50	37,112	65,919	96	77,757	117,24
5	1,624	11,669	51	37,973	67,056	97	78,657	118,34
6	2,202	13,060	52	38,837	68,192	98	79,557	119,44
7	2,814	14,423	53	39,701	69,326	99	80,458	120,53
8	3,454	15,764	54	40,567	70,459	100	81,360	121,66
9	4,115	17,085	55	41,433	71,591	110	90,400	132,61
10	4,795	18,391	56	42,301	72,721	120	99,490	143,52
11	5,491	19,683	57	43,171	73,851	130	108,61	154,39
12	6,201	20,962	58	44,041	74,979	140	117,77	165,23
13	6,922	22,231	59	44,912	76,106	150	126,96	176,04
14	7,654	23,490	60	45,785	77,232	160	136,17	186,83
15	8,396	24,741	61	46,658	78,357	170	145,41	197,59
16	9,146	25,983	62	47,533	79,482	180	154,66	208,33
17	9,904	27,219	63	48,409	80,605	190	163,94	219,05
18	10,668	28,448	64	49,286	81,727	200	173,24	229,75
19	11,440	29,671	65	50,164	82,848	210	182,56	240,43
20	12,217	30,889	66	51,042	83,969	220	191,89	251,10
21	13,00	32,101	67	51,922	85,088	230	201,24	261,75
22	13,788	33,309	68	52,803	86,207	240	210,60	272,39
23	14,581	34,512	69	53,685	87,324	250	219,97	283,01
24	15,378	35,711	70	54,567	88,441	260	229,36	293,62
25	16,178	36,905	71	55,451	89,557	270	238,75	304,23
26	16,983	38,097	72	56,335	90,673	280	248,16	314,82
27	17,793	39,284	73	57,220	91,787	290	257,58	325,39
28	18,606	40,468	74	58,106	92,901	300	267,01	335,96
29	19,422	41,649	75	58,993	94,014	310	276,45	346,52
30	20,241	42,827	76	59,880	95,126	320	285,90	357,08
31	21,063	44,002	77	60,768	96,237	330	295,36	367,62
32	21,888	45,175	78	61,657	97,348	340	304,82	378,15
33	22,715	46,345	79	62,547	98,458	350	314,29	388,68
34	23,545	47,512	80	63,437	99,567	360	323,77	399,20
35	24,378	48,677	81	64,328	100,68	370	333,26	409,71
36	25,213	49,840	82	65,219	101,79	380	342,75	420,22
37	26,050	51,000	83	66,111	102,90	390	352,25	430,72
38	26,890	52,158	84	67,003	104,00	400	361,76	441,21
39	27,732	53,315	85	67,897	105,11	410	371,27	451,69
40	28,575	54,469	86	68,790	106,21	420	380,79	462,18
41	29,421	55,622	87	69,684	107,32	430	390,32	472,65
42	30,269	56,772	88	70,579	108,42	440	399,85	483,12
43	31,119	57,921	89	71,474	109,53	450	409,38	493,58
44	31,970	59,068	90	72,370	110,63	460	418,92	504,04
45	32,823	60,214	91	73,267	111,73	470	428,47	514,50

1) The one-sided upper 95 % confidence limit for 0 structures is 2,99.

Annex G
(informative)

Strategies for collection of air samples

G.1 General

An important part of the sampling strategy is a statement of the purpose of the sampling programme. A sufficient number of samples should be collected so that the site is well characterized to the precision and accuracy desired, and also ensure that sample filters appropriately loaded for TEM analysis are obtained from all of the sampling locations.

G.2 Air sample collection in the outdoors environment

Weather conditions restrict the ability to collect satisfactory air samples in the outdoors environment, and whenever possible, sampling should be carried out in low-wind, low-humidity conditions. Detailed records of the weather conditions, windspeed and direction during the sampling period should be made. All available information concerning local topography, and the types and positions of sources should be recorded.

Sequential multipoint sampling is necessary to provide adequate characterization of complex sites and sources. It is recommended that multiple samples are taken upwind and downwind of the site, with a minimum of two samples in the downwind position expected to experience the maximum airborne concentration. The locations of the samplers should be carefully recorded.

G.3 Air sample collection inside buildings

Air samples are often collected inside buildings in which asbestos-containing construction materials are present, in order to determine whether these materials contribute to the asbestos fibre concentration in the building atmosphere. The optimum positions for collection of air samples can only be determined after a complete survey of the building to establish air movement patterns. Multiple samples should be collected in the area where asbestos building materials are present, and control samples should be collected in an adjacent area where no airborne asbestos fibres would be expected. The intakes for air conditioning systems are frequently used as the collection locations for control samples. Whenever possible, static samples should be taken over a period exceeding 4 h during normal activity in the building, at face velocities of between 4 cm/s and 25 cm/s.

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Annex H
(informative)

Methods for removal of gypsum fibres

It is common to find fibres of calcium sulfate (gypsum) in airborne particulates collected in buildings and urban environments, and particularly in samples collected where demolition or construction work is in progress. The fibres are readily released when plasters and cement products are disturbed. In some circumstances, particles of calcite or dolomite collected on an air filter can react with atmospheric sulfur dioxide, to form long fibres of gypsum. Gypsum fibres can give rise to high fibre counts by both optical and electron microscopy. The gypsum fibres are often 2 µm to 6 µm long, with aspect ratios greater than 10/1. Sometimes, these fibres appear similar to amphibole asbestos fibres, and in some samples they can be morphologically very similar to chrysotile. In the TEM, the larger fibres have high contrast and at high magnification often exhibit a characteristic mottled appearance which changes under electron beam irradiation. Some gypsum fibres, however, are not easily discriminated from asbestos without examination by EDXA. TEM specimens which contain many such gypsum fibres require an extended examination time in the TEM, because it is necessary to examine each of these fibres by EDXA before it can be rejected.

It is possible to remove gypsum fibres selectively by water extraction. A Jaffe washer (7.3.7), or a condensation washer (7.3.8), should be prepared, but using a water (6.1) as the solvent. The TEM specimens, which have been previously prepared and initially examined in the TEM, should be placed in the washer to allow dissolution of the fibres. If a Jaffe washer is used, the treatment time can be reduced by heating the washer to 90 °C to 100 °C for a few minutes. If a condensation washer is used, the gypsum fibres will be dissolved by treatment for approximately 10 min. The effect of this treatment is to remove the gypsum fibres, leaving carbon replicas (7.3.11) which are readily distinguished from asbestos fibres.

NOTE 20 This procedure should be used only when examination of the untreated TEM specimen grids shows the gypsum fibres to be isolated from any asbestos fibres present. Losses of asbestos fibres may occur if matrices of gypsum and asbestos are exposed to this procedure.

Annex J
(informative)

Bibliography

[1] Asbestos International Association (1979): *Reference method for the determination of asbestos fibre concentrations at workplaces by light microscopy (membrane filter method).* AIA health and safety publication, recommended technical method No. 1 (RTM1). Asbestos International Association, 68 Gloucester Place, London, W1H 3HL, England.

[2] BRADLEY, D.E. (1961): Replica and shadowing techniques. In: *Techniques for Electron Microscopy,* Blackwell Scientific Publications, Alden, Oxford, D.H. Kay (Ed.), pp. 96-152.

[3] BURDETT, G.J. and ROOD, A.P. (1982): Membrane-filter, direct transfer technique for the analysis of asbestos fibres or other inorganic particles by transmission electron microscopy. *Environmental Science and Technology,* **17**, pp. 643-648.

[4] CAMPBELL, W.J., BLAKE, R.L., BROWN, L.L., CATHER, E.E. and SJÖBERG, J.J. (1977): *Selected silicate minerals and their asbestiform varieties. Mineralogical definitions and identification-characterization.* Information circular 8751. United States Department of the Interior, Bureau of Mines, Washington, D.C.

[5] CHATFIELD, E.J. (1986): Asbestos measurements in workplaces and ambient atmospheres. In: *Electron microscopy in forensic, occupational, and environmental health sciences* (S. Basu and J.R. Millette, Eds.). Plenum Press, New York, pp. 149-186.

[6] CHATFIELD, E.J. (Editor) (1987): *Asbestos fibre measurements in building atmospheres.* Ontario Research Foundation, Sheridan Park Research Community, Mississauga, Ontario, Canada, L5K 1B3.

[7] CHATFIELD, E.J. and LEWIS, G.M. (1980): Development and application of an analytical technique for measurement of asbestos fibers in vermiculite. In: *Scanning Electron Microscopy*/1980/I, (O. Johari, Ed.), SEM Inc., AMF O'Hare, Chicago, Illinois 60666, USA.

[8] CLIFF, G. and LORIMER, G.W. (1975): The quantitative analysis of thin specimens. *Journal of Microscopy,* **103**, pp. 203-207.

[9] DEER, W.A., HOWIE, R.A. and ZUSSMAN, J. (1963): *Rock-forming minerals.* Longmans, London.

[10] Federal Register (1987): *Asbestos-containing materials in schools.* U.S. Environmental Protection Agency. Vol. 42, No. 210, October 30, 1987, pp. 41826-41905.

[11] GARD, J. A. (Editor) (1971): *The Electron Optical Investigation of Clays.* Mineralogical Society, 41 Queen's Gate, London S.W. 7.

[12] GAZE, R. (1965): The physical and molecular structure of asbestos. *Annals of the New York Academy of Science,* Vol. 132, pp. 23-30.

[13] HAWTHORNE, F.C. (1983): The crystal chemistry of the amphiboles. *Canadian Mineralogist,* Vol. 21, part 2, pp. 173-480.

[14] HIRSCH, P.B., HOWIE, A., NICHOLSON, R.B., PASHLEY, D.W. and WHELAN, M.J. (1965): *Electron microscopy of thin crystals.* Butterworths, London, pp. 18-23.

[15] HOLLAHAN, J.R. and BELL, A.T. (Editors) (1974): *Techniques and applications of plasma chemistry.* Wiley, New York.

[16] International Centre for Diffraction Data (1987): *Powder diffraction file.* International Centre for Diffraction Data, 1606 Park Lane, Swarthmore, Pennsylvania 19081, USA.

[17] International Mineralogical Association (1978): Nomenclature of amphiboles (compiled by B.E. Leake); *Canadian Mineralogist,* Vol. 16, p. 501.

[18] International Organization for Standardization (Organisation internationale de normalisation) (1993): ISO 8672:1993, *Air quality — Determination of the number concentration of airborne*

inorganic fibres by phase contrast optical microscopy — Membrane filter method.

[19] JAFFE, M.S. (1948): Handling and washing fragile replicas. *J. Applied Physics,* **19**, p. 1187.

[20] JOY, D.C., ROMIG, Jr. and GOLDSTEIN, J.I. (Editors) (1986): *Principles of analytical electron microscopy.* Plenum Press, New York and London.

[21] LEDOUX, R.L. (Editor) (1979): *Short course in mineralogical techniques of asbestos determination.* Mineralogical Association of Canada, Department of Mineralogy, Royal Ontario Museum, 100 Queen's Park, Toronto, Ontario Canada M5S 2C6.

[22] LEVADIE, B. (Editor) (1984): *Definitions for asbestos and other health-related silicates.* ASTM Special Technical Publication 834. American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103, USA.

[23] MICHAEL, J.R. and WILLIAMS, D.B. (1987): A consistent definition of probe size and spatial resolution in the analytical electron microscope. *J. Mic.,* **147**, pp. 289-303.

[24] MICHAELS, L. and CHISSICK, S.S. (Editors) (1979): *Asbestos: Properties, Applications and Hazards,* Vol, 1, Wiley, New York.

[25] National Bureau of Standards Special Publication 506 (1978): *Workshop on asbestos: definitions and measurement methods.* U.S. Government Printing Office, Washington, D.C. 20402.

[26] National Bureau of Standards Special Publication 619 (1982): *Asbestos standards: material and analytical methods.* U.S. Government Printing Office, Washington, D.C. 20402.

[27] National Institute for Occupational Safety and Health (1989): *NIOSH Method 7400,* Revision #3, 5/15/89. U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, 4676 Columbia Parkway, Cincinnati, Ohio 45226, USA.

[28] National Institute for Occupational Safety and Health (1989): *NIOSH Method 7402,* Revision #1, 5/15/89. U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control, National Institute for Occupational Safety and Health, 4676 Columbia Parkway, Cincinnati, Ohio 45226, USA.

[29] NATRELLA, M.G. (1966): *Experimental statistics*: National Bureau of Standards Handbook 91. U.S. Government Printing Office, Washington, D.C. 20402.

[30] ORTIZ, L.W. and ISOM, B.L: (1974): Transfer technique for electron microscopy of membrane filter samples. *American Industrial Hygiene Association Journal,* **35**, 7, pp. 423-425.

[31] PEARSON, E.S. and HARTLEY, H.O. (1958): *Biometrica tables for statisticians,* Vol. 1, Cambridge University Press, 32 East 57th Street, New York, N.Y. 22, USA.

[32] RHOADES, B.L. (1976): *XIDENT-A computer technique for the direct indexing of electron diffraction spot patterns.* Research Report 70/76. Dept. of Mechanical Engineering, Univ. of Canterbury, Christchurch, New Zealand.

[33] RING, S.J. (1980): Identification of amphibole fibers, including asbestos, using common electron diffraction patterns. In: *Electron Microscopy and X-ray Applications to Environmental and Occupational Health Analysis,* (Ed. P.A. Russe Vol. II, Ann Arbor Press, Ann Arbor, Michigan 48106, USA.

[34] RUSSELL, P.A. and HUTCHINGS, A.E. (1978): *Electron Microscopy and X-ray Applications to Environmental and Occupational Health Analysis.* Ann Arbor Science Publishers Inc., P.O. Box 1425, Ann Arbor, Michigan 48106, USA.

[35] SMALL, J.A., HEINRICH, K.F.J., NEWBURY, D.E. and MYKLEBUST, R.L. (1979): Progress in the development of the peak-to-background method for the quantitative analysis of single particles with the electron probe. *Scanning Electron Microscopy*/1979/II, (O. Johari, Ed.). SEM Inc., AMF O'Hare, Chicago, Illinois 60666, USA.

[36] SMALL, J.A., STEEL, E.B. and SHERIDAN, P.J. (1985): Analytical standards for the analysis of chrysotile asbestos in ambient environments. *Analytical Chemistry,* **57**, pp. 204-208.

[37] SMITH, J.E. and JORDAN, M.L. (1964): Mathematical and graphical interpretation of the log-normal law for particle size distribution analysis. *J. Colloid Science,* **19**, pp. 549-559.

[38] SPURNY, K.R., STOBER, H., OPELIA, H. and WEISS, G. (1979): On the evaluation of fibrous

particles in remote ambient air. *Science of the Total Environment*/1979/II, pp. 1-40.

[39] SPURNY, K.R. (Editor) (1986): *Physical and chemical characterization of individual airborne particles*. Wiley, New York.

[40] STEEL, E.B. and SMALL, J.A. (1985): Accuracy of transmission electron microscopy for the analysis of asbestos in ambient environments. *Analytical Chemistry*, **57**, pp. 209-213.

[41] STEEL, E.B. and WYLIE, A. (1981): Mineralogical characteristics of asbestos. In: *Geology of Asbestos Deposits*, (P.H. Riorden, Ed.), SME-AIME, pp. 93-101.

[42] WENK, H.R. (Editor) (1976): *Electron microscopy in mineralogy*. Springer-Verlag, New York.

[43] YADA, K. (1967): Study of chrysotile asbestos by a high resolution electron microscope. *Acta Crystallographica*, **23**, pp. 704-707.

[44] ZUSSMAN, J. (1979): The mineralogy of asbestos. In: *Asbestos: Properties, Applications and Hazards*, John Wiley and Sons, pp. 45-67.

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Price based on 51 pages

ASBESTOS SAMPLING

SOP#: 2015
DATE: 11/17/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

Asbestos has been used in many commercial products including building materials such as flooring tiles and sheet goods, paints and coatings, insulation, and roofing asphalts. These products and others may be found at hazardous waste sites hanging on overhead pipes, contained in drums, abandoned in piles, or as part of a structure. Asbestos tailing piles from mining operations can also be a source of ambient asbestos fibers. Asbestos is a known carcinogen and requires air sampling to assess airborne exposure to human health. This Standard Operating Procedure (SOP) provides procedures for asbestos air sampling by drawing a known volume of air through a mixed cellulose ester (MCE) filter. The filter is then sent to a laboratory for analysis. The U.S. Environmental Protection Agency/Environmental Response Team (U.S. EPA/ERT) uses one of four analytical methods for determining asbestos in air. These include: U.S. EPA's Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air for Transmission Electron Microscopy (TEM)[1]; U.S. EPA's Modified Yamate Method for TEM[2]; National Institute for Occupational Safety and Health (NIOSH) Method 7402 (direct method only) for TEM; and NIOSH Method 7400 for Phase Contrast Microscopy (PCM)[3]. Each method has specific sampling and analytical requirements (i.e., sample volume and flow rate) for determining asbestos in air.

The U.S. EPA/ERT typically follows procedures outlined in the TEM methods for determining mineralogical types of asbestos in air and for distinguishing asbestos from non-asbestos minerals. The Phase Contrast Microscopy (PCM) method is used by U.S. EPA/ERT as a screening tool since it is less costly than TEM. PCM cannot distinguish asbestos from non-asbestos fibers, therefore the TEM method may be necessary to confirm analytical results. For example, if an action level for the presence of fibers has been set and PCM analysis indicates that the action level has been exceeded, then TEM analysis can be used to quantify and identify asbestos structures through examination of their morphology crystal structures (through electron diffraction), and elemental composition (through energy dispersive X-ray analysis). In this instance samples should be collected for both analyses in side by side sampling trains (some laboratories are able to perform PCM and TEM analysis from the same filter). The Superfund method is designed specifically to provide results suitable for supporting risk assessments at Superfund sites, it is applicable to a wide range of ambient air situations at hazardous waste sites. U.S. EPA's Modified Yamate Method for TEM is also used for ambient air sampling due to high volume requirements. The PCM and TEM NIOSH analytical methods require lower sample volumes and are typically used indoors; however, ERT will increase the volume requirement for outdoor application.

Other Regulations pertaining to asbestos have been promulgated by U.S. EPA and OSHA. U.S. EPA's National Emission Standards for Hazardous Air Pollutants (NESHAP) regulates asbestos-containing waste materials. NESHAP establishes management practices and standards for the handling of asbestos and emissions from waste disposal operations (40 CFR Part 61, Subparts A and M). U.S. EPA's 40 CFR 763 (July 1, 1987)[4] and its addendum 40 CFR 763 (October 30, 1987)[4] provide comprehensive rules for the asbestos abatement industry. State and local regulations on these issues vary and may be more stringent than federal requirements. The OSHA regulations in 29 CFR 1910.1001 and 29 CFR 1926.58 specify work practices and safety equipment such as respiratory protection and protective clothing when handling asbestos. The OSHA standard for an 8-hour, time-weighted average (TWA) is 0.2 fibers/cubic centimeters of air. This standard pertains to fibers with a length-to-width ratio of 3 to 1 with a fiber length >5 µm[5,6]. An action level of 0.1 fiber/cc (one-half the OSHA standard) is the level U.S. EPA has established in which employers must initiate such activities as air monitoring, employee training, and

medical surveillance[5,6].

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Prior to sampling, the site should be characterized by identifying on-site as well as off-site sources of airborne asbestos. The array of sampling locations and the schedule for sample collection, is critical to the success of an investigation. Generally, sampling strategies to characterize a single point source are fairly straightforward, while multiple point sources and area sources increase the complexity of the sampling strategy. It is not within the scope of this SOP to provide a generic asbestos air sampling plan. Experience, objectives, and site characteristics will dictate the sampling strategy.

During a site investigation, sampling stations should be arranged to distinguish spatial trends in airborne asbestos concentrations. Sampling schedules should be fashioned to establish temporal trends. The sampling strategy typically requires that the concentration of asbestos at the source (worst case) or area of concern (downwind), crosswind, as well as background (upwind) contributions be quantified. See Table 1 (Appendix A) for U.S. EPA/ERT recommended sampling set up for ambient air. Indoor asbestos sampling requires a different type of strategy which is identified in Table 2 (Appendix A). It is important to establish background levels of contaminants in order to develop a reference point from which to evaluate the source data. Field blanks and lot blanks can be utilized to determine other sources.

Much information can be derived from each analytical method previously mentioned. Each analytical method has specific sampling requirements and produce results which may or may not be applicable to a specific sampling effort. The site sampling objectives should be carefully identified so as to select the most appropriate analytical method. Additionally, some preparation (i.e., lot blanks results) prior to site sampling may be required, these requirements are specified in the analytical methods.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Sample Preservation

No preservation is required for asbestos samples.

3.2 Sample Handling, Container and Storage Procedures

1. Place a sample label on the cassette indicating a unique sampling number. Do not put sampling cassettes in shirt or coat pockets as the filter can pick up fibers. The original cassette box is used to hold the samples.

2. Wrap the cassette individually in a plastic sample bag. Each bag should be marked indicating sample identification number, total volume, and date.

3. The wrapped sampling cassettes should be placed upright in a rigid container so that the cassette cap is on top and cassette base is on bottom. Use enough packing material to prevent jostling or damage. Do not use vermiculite as packing material for samples. If possible, hand carry to lab.

4. Provide appropriate documentation with samples (i.e., chain of custody and requested analytical methodology).

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Flow rates exceeding 16 liters/minute (L/min) which could result in filter destruction due to (a) failure of its physical support under force from the increased pressure drop; (b) leakage of air around the filter mount so that the filter is bypassed, or (c) damage to the asbestos structures due to increased impact velocities.

4.1 U.S. EPA's Superfund Method

4.1.1 Direct-transfer TEM Specimen Preparation Methods

Direct-Transfer TEM specimen preparation methods have the following significant interferences:

- The achievable detection limit is restricted by the particulate density on the filter, which in turn is controlled by the sampled air volume and the total suspended particulate concentration in the atmosphere being sampled.

- The precision of the result is dependent on the uniformity of the deposit of asbestos structures on the sample collection filter.

- Air samples must be collected so that they have particulate and fiber loadings within narrow ranges. If too high a particulate loading occurs on the filter, it is not possible to prepare satisfactory TEM specimens by a direct-transfer method. If too high a fiber loading occurs on the filter, even if satisfactory TEM specimens can be prepared, accurate fiber counting will not be possible.

4.1.2 Indirect TEM Specimen Preparation Methods

Indirect TEM specimen preparation methods have the following interferences:

- The size distribution of asbestos structures is modified.

- There is increased opportunity for fiber loss or introduction of extraneous contamination.

- When sample collection filters are ashed, any fiber contamination in the filter medium is concentrated on the TEM specimen grid.

It can be argued that direct methods yield an under-estimate of the asbestos structure concentration because many of the asbestos fibers present are concealed by other particulate material with which they are associated. Conversely, indirect methods can be considered to yield an over-estimate because some types of complex asbestos structures disintegrate during the preparation, resulting in an increase in the numbers of structures counted.

4.2 U.S. EPA's Modified Yamate Method for TEM

High concentrations of background dust interfere with fiber identification.

4.3 NIOSH Method for TEM

Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification.

4.4 NIOSH Method for PCM

PCM cannot distinguish asbestos from non-asbestos fibers; therefore, all particles meeting the counting criteria are counted as total asbestos fibers. Fiber less than 0.25 um in length will not be detected by this method. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

5.0 EQUIPMENT/MATERIALS

5.1 Sampling Pump

The constant flow or critical orifice controlled sampling pump should be capable of a flow-rate and pumping time sufficient to achieve the desired volume of air sampled.

The lower flow personal sampling pumps generally provide a flow rate of 20 cubic centimeters/minute (cc/min) to 4 L/min. These pumps are usually battery powered. High flow pumps are utilized when flow rates between 2 L/min to 20 L/min are required. High flow pumps are used for short sampling periods so as to obtain the desired sample volume. High flow pumps usually run on AC power and can be plugged into a nearby outlet. If an outlet is not available then a generator should be obtained. The generator should be positioned downwind from the sampling pump. Additional voltage may be required if more than one pump is plugged into the same generator. Several

electrical extension cords may be required if sampling locations are remote.

The recommended volume for the Superfund method (Phase I) requires approximately 20 hours to collect. Such pumps typically draw 6 amps at full power so that 2 lead/acid batteries should provide sufficient power to collect a full sample. The use of line voltage, where available, eliminates the difficulties associated with transporting stored electrical energy.

A stand should be used to hold the filter cassette at the desired height for sampling and the filter cassette shall be isolated from the vibrations of the pump.

5.2 Filter Cassette

The cassettes are purchased with the required filters in position, or can be assembled in a laminar flow hood or clean area. When the filters are in position, a shrink cellulose band or adhesive tape should be applied to cassette joints to prevent air leakage.

5.2.1 TEM Cassette Requirements

Commercially available field monitors, comprising 25 mm diameter three-piece cassettes, with conductive extension cowls shall be used for sample collection. The cassette must be new and not previously used. 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. The cowls should be constructed of electrically conducting material to minimize electrostatic effects. The filter shall be backed by a 5 µm pore size MCE filter (Figure 1, Appendix B).

5.2.2 PCM Cassette Requirements

NIOSH Method 7400, PCM involves using a 0.8 to 1.2 µm mixed cellulose ester membrane, 25 mm diameter, 50 mm conductive cowl on cassette (Figure 2, Appendix B). Some labs are able to perform PCM and TEM analysis on the same filter; however, this should be discussed with the laboratory prior to sampling.

5.3 Other Equipment

- Inert tubing with glass cyclone and hose barb
- Whirlbags (plastic bags) for cassettes
- Tools - small screw drivers
- Container - to keep samples upright
- Generator or electrical outlet (may not be required)
- Extension cords (may not be required)
- Multiple plug outlet
- Sample labels
- Air data sheets
- Chain of Custody records

6.0 REAGENTS

Reagents are not required for the preservation of asbestos samples.

7.0 PROCEDURES

7.1 Air Volumes and Flow Rates

Sampling volumes are determined on the basis of how many fibers need to be collected for reliable measurements. Therefore, one must estimate how many airborne fibers may be in the sampling location.

Since the concentration of airborne aerosol contaminants will have some effect on the sample, the following is a suggested criteria to assist in selecting a flow rate based on real-time aerosol monitor (RAM) readings in milligrams/cubic meter (mg/m^3).

	Concentration	Flow Rate
Low RAM readings:	<6.0 mg/m^3	11-15. L/min
Medium RAM readings:	>6.0 mg/m_3	7.5 L/min
High RAM readings:	>10. mg/m^3	2.5 L/min

In practice, pumps that are available for environmental sampling at remote locations operate under a maximum load of approximately 12 L/min.

7.1.1 U.S. EPA's Superfund Method

The Superfund Method incorporates an indirect preparation procedure to provide flexibility in the amount of deposit that be can be tolerated on the sample filter and to allow for the selective concentration of asbestos prior to analysis. To minimize contributions to background contamination from asbestos present in the plastic matrices of membrane filters while allowing for sufficient quantities of asbestos to be collected, this method also requires the collection of a larger volume of air per unit area of filter than has traditionally been collected

for asbestos analysis. Due to the need to collect large volumes of air, higher sampling flow rates are recommended in this method than have generally been employed for asbestos sampling in the past. As an alternative, samples may be collected over longer time intervals. However, this restricts the flexibility required to allow samples to be collected while uniform meteorological conditions prevail.

The sampling rate and the period of sampling should be selected to yield as high a sampled volume as possible, which will minimize the influence of filter contamination. Wherever possible, a volume of 15 cubic meters (15,000 L) shall be sampled for those samples intended for analysis only by the indirect TEM preparation method (Phase 1 samples). For those samples to be prepared by both the indirect and the direct specimen preparation methods (Phase 2 samples), the volumes must be adjusted so as to provide a suitably-loaded filter for the direct TEM preparation method. One option is to collect filters at several loadings to bracket the estimated optimum loading for a particular site. Such filters can be screened in the laboratory so that only those filters closest to optimal loading are analyzed. It has been found that the volume cannot normally exceed 5 cubic meters (5000 L) in an urban or agricultural area, and 10 cubic meters (10,000 L) in a rural area for samples collected on a 25 mm filter and prepared by a direct-transfer technique.

An upper limit to the range of acceptable flow rates for this method is 15 L/min. At many locations, wind patterns exhibit strong diurnal variations. Therefore, intermittent sampling (sampling over a fixed time interval repeated over several days) may be necessary to accumulate 20 hours of sampling time over constant wind conditions. Other sampling objectives also may necessitate intermittent sampling. The objective is to design a sampling schedule so that samples are collected under uniform conditions throughout the sampling interval. This method provides for such options. Air volumes collected on Phase I samples are maximized (<16 L/min). Air volumes collected on Phase 2 samples are limited to provide optimum loading for filters to be prepared by a direct-transfer procedure.

7.1.2 U.S. EPA's Modified Yamate Method for TEM

U.S. EPA's TEM method requires a minimum volume of 560 L and a maximum volume of 3,800 L in order to obtain an analytical sensitivity of 0.005 structures/cc. The optimal volume for TEM is 1200 L to 1800 L. These volumes are determined using a 200 mesh EM grid opening with a 25-mm filter cassette. Changes in volume would be necessary if a 37-mm filter cassette is used since the effective area of a 25 mm (385 sq mm) and 37 mm (855 sq m) differ.

7.1.3 NIOSH Method for TEM and PCM

The minimum recommended volume for TEM and PCM is 400 L at 0.1 fiber/cc. Sampling time is adjusted to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for eight hours (700 to 2800 L) is appropriate in non-dusty atmospheres containing 0.1 fiber/cc. Dusty atmospheres i.e., areas with high levels of asbestos, require smaller sample volumes (<400 L) to obtain countable samples.

In such cases, take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3,000 to 10,000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If > 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration. Do not exceed 0.5 mg total dust loading on the filter.

7.2 Calibration Procedures

In order to determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the instrument. Sampling pumps should be calibrated immediately before and after each use. Preliminary calibration should be conducted using a primary calibrator such as a soap bubble type calibrator, (e.g., a Buck Calibrator, Gilibrator, or equivalent primary calibrator) with a representative filter cassette installed between the pump and the calibrator. The representative sampling cassette can be reused for calibrating other pumps that will be used for asbestos sampling. The same cassette lot used for sampling should also be used for the calibration. A sticker should be affixed to the outside of the extension cowl marked "Calibration Cassette."

A rotameter can be used provided it has been recently precalibrated with a primary calibrator. Three separate constant flow calibration readings should be obtained both before sampling and after sampling. Should the flow rate change by more than 5% during the sampling period, the average of the pre- and post-calibration rates will be used to calculate the total sample volume. The sampling pump used shall provide a non-fluctuating air-flow through the filter, and shall maintain the initial volume flow-rate to within ± 10% throughout the sampling period. The mean value of these flow-rate measurements shall be used to calculate the total air volume sampled. A constant flow or critical orifice controlled pump meets these requirements. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, the sampling shall be terminated. Flexible tubing is used to connect the filter cassette to the sampling pump.Sampling pumps can be calibrated prior to coming on-site so that time is saved when performing on-site calibration.

7.2.1 Calibrating a Personal Sampling Pump with an Electronic Calibrator

1. See Manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 3, Appendix B) using a sampling pump, electronic calibrator, and a representative filter cassette. The same lot sampling cassette used for sampling should also be used for calibrating.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 foot) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the electronic calibrator.

4. Turn the electronic calibrator and sampling pump on. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

5. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

6. Perform the calibration three times until the desired flow rate of ± 5% is attained.

7.2.2 Calibrating a Rotameter with an Electronic Calibrator

1. See manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 4, Appendix B) using a sampling pump, rotameter, and electronic calibrator.

3. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6º vertical.

4. Turn the electronic calibrator and sampling pump on.

5. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

6. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

7. Record the electronic calibrator flow rate reading and the corresponding rotameter reading. Indicate these values on the rotameter (sticker). The rotameter should be able to work within the desired flow range. Readings can also be calibrated for 10 cm^3 increments for Low Flow rotameters, 500 cm^3 increments for medium flow rotameters and 1 liter increments for high flow rotameters.

8. Perform the calibration three times until the desired flow rate of ± 5% is attained. Once on site, a secondary calibrator, i.e., rotameter may be used to calibrate sampling pumps.

7.2.3 Calibrating a Personal Sampling Pump with a Rotameter

1. See manufacturer's manual for Rotameter's Operational Instructions.

2. Set up the calibration train as shown in (Figure 5, Appendix B) using a rotameter, sampling pump, and a representative sampling cassette.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 ft) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the rotameter.

4. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6º vertical.

5. Turn the sampling pump on.

6. Turn the flow adjust screw (or knob) on the personal sampling pump until the float ball on the rotameter is lined up with the precalibrated flow rate value. A sticker on the rotameter should indicate this value.

7. A verification of calibration is generally performed on-site in the clean zone immediately prior to the sampling.

7.3. Meteorology

It is recommended that a meteorological station be established. If possible, sample after two to three days of dry weather and when the wind conditions are at 10 mph or greater. Record wind speed, wind direction, temperature, and pressure in a field logbook. Wind direction is particularly important when monitoring for asbestos downwind from a fixed source.

7.4 Ambient Sampling Procedures

7.4.1 Pre-site Sampling Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain necessary sampling equipment and ensure it is in working order and fully charged (if necessary).

3. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety plan.

4. Once on-site the calibration is performed in the clean zone. The calibration procedures are listed in Section 7.2.

5. After calibrating the sampling pump, mobilize to the sampling location.

7.4.2 Site Sampling

1. To set up the sampling train, attach the air intake hose to the cassette base. Remove the cassette cap (Figure 6 and 7, Appendix B). The cassette should be positioned downward, perpendicular to the wind

2. If AC or DC electricity is required then turn it on. If used, the generator should be placed 10 ft. downwind from the sampling pump.

3. Record the following in a field logbook: date, time, location, sample identification number, pump number, flow rate, and cumulative time.

4. Turn the pump on. Should intermittent sampling be required, sampling filters must be covered between active periods of sampling. To cover the sample filter: turn the cassette to face upward, place the cassette cap on the cassette, remove the inlet plug from the cassette cap, attach a rotameter to the inlet opening of the cassette cap to measure the flow rate, turn off the sampling pump, place the inlet plug into the inlet opening on the cassette cap. To resume sampling: remove the inlet plug, turn on the sampling pump, attach a rotameter to measure the flow rate, remove the cassette cap, replace the inlet plug in the cassette cap and invert the cassette, face downward and perpendicular to the wind.

5. Check the pump at sampling midpoint if sampling is longer than 4 hours. The generators may need to be regased depending on tank size. If a filter darkens in appearance or if loose dust is seen in the filter, a second sample should be started.

6. At the end of the sampling period, orient the cassette up, turn the pump off.

7. Check the flow rate as shown in Section 7.2.3. When sampling open-faced, the sampling cap should be replaced before post calibrating. Use the same cassette used for sampling for post calibration (increase dust/fiber loading may have altered the flow rate.

8. Record the post flow rate.

9. Record the cumulative time or run.

10. Remove the tubing from the sampling cassette. Still holding the cassette upright, replace the inlet plug on the cassette cap and the outlet plug on the cassette base.

7.4.3. Post Site Sampling

1. Follow handling procedures in Section 3.2, steps 1-4.

2. Obtain an electronic or hard copy of meteorological data which occurred during the sampling event. Record weather: wind speed, ambient temperature, wind direction, and precipitation. Obtaining weather data several days prior to the sampling event can also be useful.

7.5 Indoor Sampling Procedures

PCM analysis is used for indoor air samples. When analysis shows total fiber count above the OSHA action level 0.1 f/cc then TEM (U.S. EPA's Modified Yamate Method) is used to identify asbestos from non-asbestos fibers.

Sampling pumps should be placed four to five feet above ground level away from obstructions that may influence air flow. The pump can be placed on a table or counter. Refer to Table 2 (Appendix A) for a summary of indoor sampling locations and rationale for selection.

Indoor sampling utilizes high flow rates to increased sample volumes (2000 L for PCM and 2800 to 4200 L for TEM) in order to obtain lower detection limits below the standard, (i.e., 0.01 f/cc or lower [PCM] and 0.005 structures/cc or lower [TEM]).

7.5.1 Aggressive Sampling Procedures

Sampling equipment at fixed locations may fail to detect the presence of asbestos fibers. Due to limited air movement, many fibers may settle out of the air onto the floor and other surfaces and may not be captured on the filter. In the past, an 8-hour sampling period was recommended to cover various air circulation conditions. A quicker and more effective way to capture asbestos fibers is to circulate the air artificially so that the fibers remain airborne during sampling. The results from this sampling option typifies worst case condition. This is referred to as aggressive air sampling for asbestos. Refer to Table 2 for sample station locations.

1. Before starting the sampling pumps, direct forced air (such as a 1-horsepower leaf blower or large fan) against walls, ceilings, floors, ledges, and other surfaces in the room to initially dislodge fibers from surfaces. This should take at least 5 minutes per 1000 sq. ft. of floor.

2. Place a 20-inch fan in the center of the room. (Use one fan per 10,000 cubic feet of room space.) Place the fan on slow speed and point it toward the ceiling.

3. Follow procedures in Section 7.4.1 and 7.4.2 (Turn off the pump and then the fan(s) when sampling is complete.).

4. Follow handling procedures in Section 3.2, steps 1-4.

8.0 CALCULATIONS

The sample volume is calculated from the average flow rate of the pump multiplied by the number of minutes the pump was running (volume = flow rate X time in minutes). The sample volume should be submitted to the laboratory and identified on the chain of custody for each sample (zero for lot, field and trip blanks).

The concentration result is calculated using the sample volume and the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

Follow all QA/QC requirements from the laboratories as well as the analytical methods.

9.1 TEM Requirements

1. Examine lot blanks to determine the background asbestos structure concentration.

2. Examine field blanks to determine whether there is contamination by extraneous asbestos structures during specimen preparation.

3. Examine of laboratory blanks to determine if contamination is being introduced during critical phases of the laboratory program.

4. To determine if the laboratory can satisfactorily analyze samples of known asbestos structure concentrations, reference filters shall be examined. Reference filters should be maintained as part of the laboratory's Quality Assurance program.

5. To minimize subjective effects, some specimens should be recounted by a different microscopist.

6. Asbestos laboratories shall be accredited by the National Voluntary Laboratory Accreditation Program.

7. At this time, performance evaluation samples for asbestos in air are not available for Removal Program Activities.

9.2 PCM Requirements

1. Examine reference slides of known concentration to determine the analyst's ability to satisfactorily count fibers. Reference slides should be maintained as part of the laboratory's quality assurance program.

2. Examine field blanks to determine if there is contamination by extraneous structures during sample handling.

3. Some samples should be relabeled then submitted for counting by the same analyst to determine possible bias by the analyst.

4. Participation in a proficiency testing program such as the AIHA-NIOSH proficiency analytical testing (PAT) program.

10.0 DATA VALIDATION

Results of quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures. More specifically, when entering an unknown situation involving asbestos, a powered air purifying respirator (PAPR) (full face-piece) is necessary in conjunction with HEPA filter cartridges. See applicable regulations for action level, PEL, TLV, etc. If previous sampling indicates asbestos concentrations are below personal health and safety levels, then Level D personal protection is adequate.

12.0 REFERENCES

(1) Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air, Part 1: Method, EPA/540/2-90/005a, May 1990, and Part 2: Technical Background Document, EPA/540/2-90/005b, May 1990.

(2) Methodology for the Measurement of Airborne Asbestos by Electron Microscopy, EPA's Report No. 68-02-3266, 1984, G. Yamate, S.C. Agarwal, and R. D. Gibbons.

(3) National Institute for Occupational Safety and Health. NIOSH Manual of Analytical Method. Third Edition. 1987.

(4) U.S. Environmental Protection Agency. Code of Federal Regulations 40 CFR 763. July 1, 1987. Code of Federal Regulations 40 CFR 763 Addendum. October 30, 1987.

(5) U.S. Environmental Protection Agency. Asbestos-Containing Materials in Schools; Final Rule and Notice. 52 FR 41826.

(6) Occupational Safety and Health Administration. Code of Federal Regulations 29 CFR 1910.1001. Washington, D.C. 1987.

APPENDIX A

Tables

TABLE 1. SAMPLE STATIONS FOR OUTDOOR SAMPLING		
Sample Station Location	Sample Numbers	Rationale
Upwind/Background[1]	Collect a minimum of two simultaneous upwind/background samples 30º apart from the prevailing windlines.	Establishes background fiber levels.
Downwind	Deploy a minimum of 3 sampling stations in a 180 degree arc downwind from the source.	Indicates if asbestos is leaving the site.
Site Representative and/or Worst Case	Obtain one site representative sample which shows average condition on-site or obtain worst case sample (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

[1] More than one background station may be required if the asbestos originates from different sources.

APPENDIX A (Cont'd)

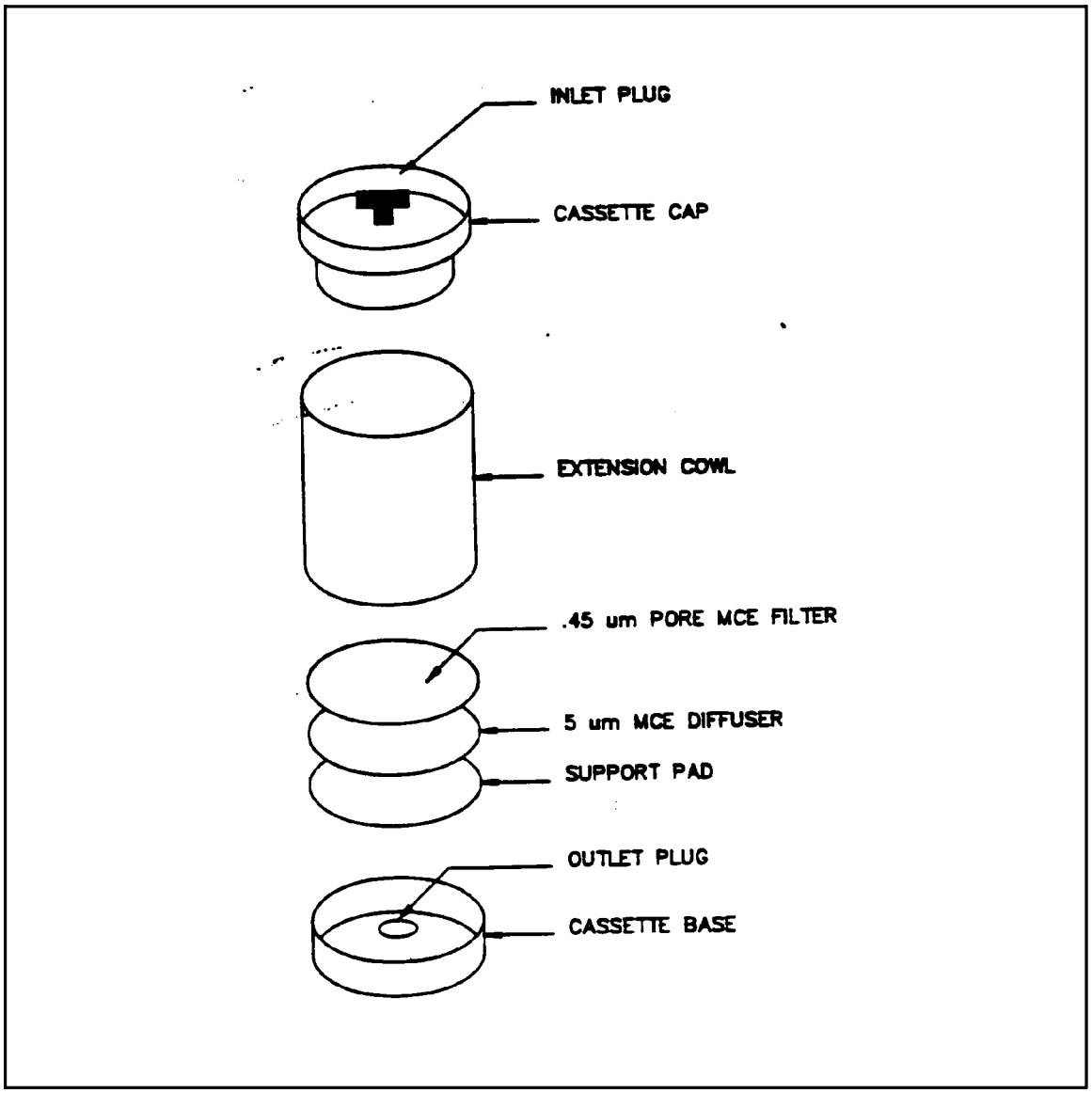
Tables

TABLE 2 SAMPLE STATIONS FOR INDOOR SAMPLING		
Sample Station Location	Sample Numbers	Rationale
Indoor Sampling	If a work site is a single room, disperse 5 samplers throughout the room. If the work site contains up to 5 rooms, place at least one sampler in each room. If the work site contains more than 5 rooms, select a representative sample of the rooms.	Establishes representative samples from a homogeneous area.
Upwind/Background	If outside sources are suspected, deploy a minimum of two simultaneous upwind/background samples 30º apart from the prevailing windlines.	Establish whether indoor asbestos concentrations are coming from an outside source.
Worst Case	Obtain one worst case sample, i.e., aggressive sampling (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

APPENDIX B

Figures

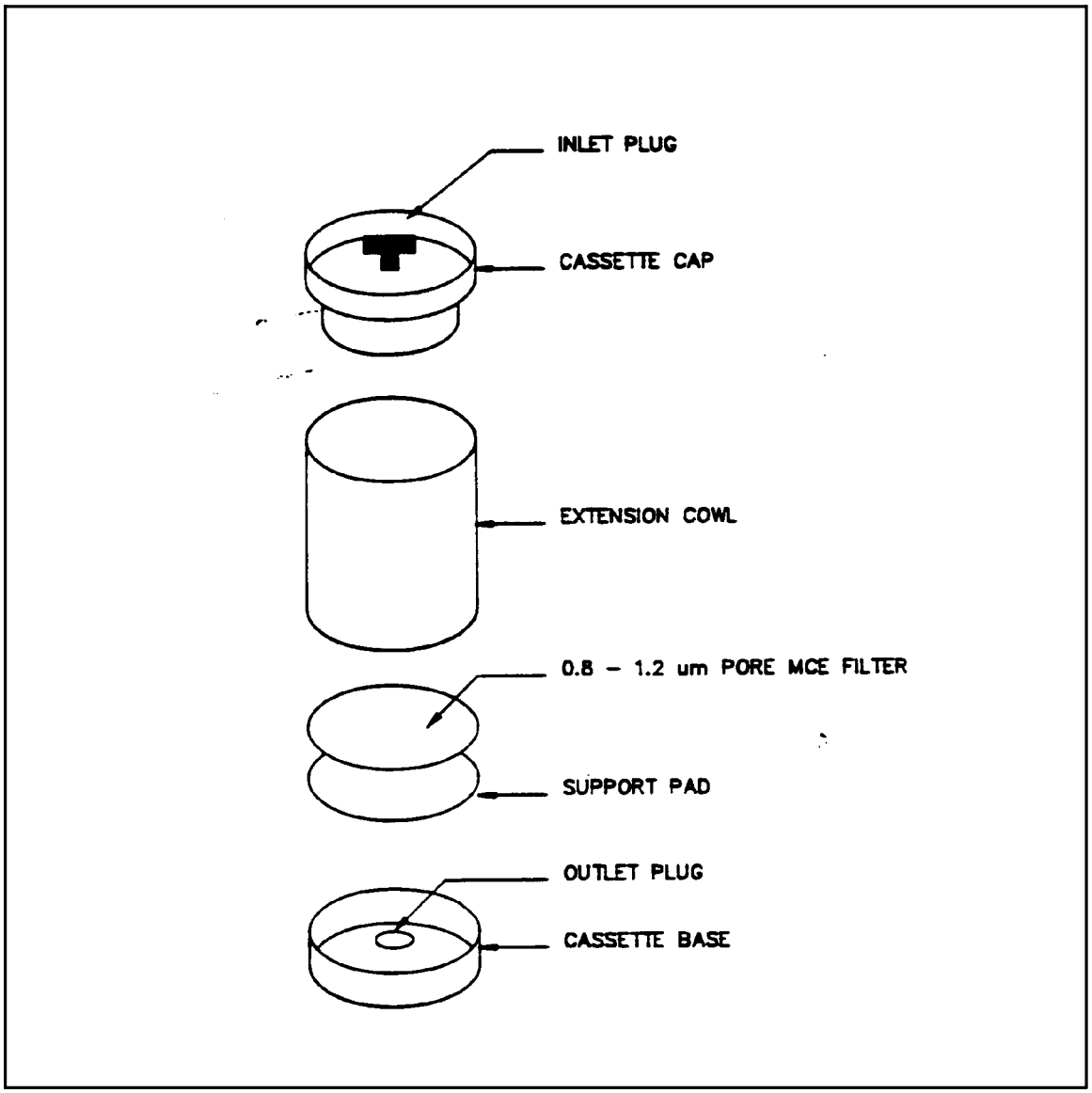
FIGURE 1. Transmission Electron Microscopy Filter Cassette



APPENDIX B (Cont'd)

Figures

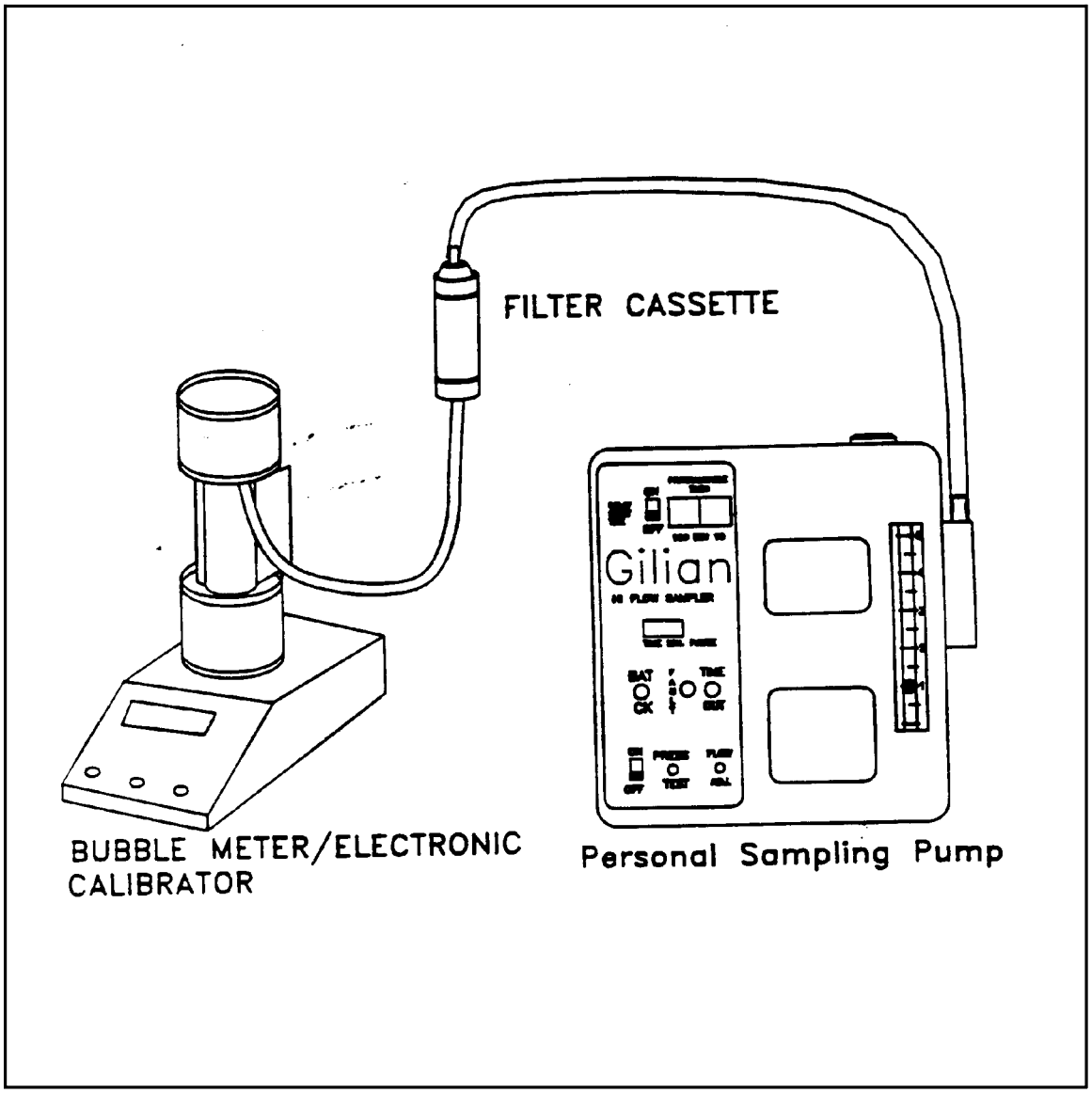
FIGURE 2. Phase Contrast Microscopy Filter Cassette



APPENDIX B (Cont'd)

Figures

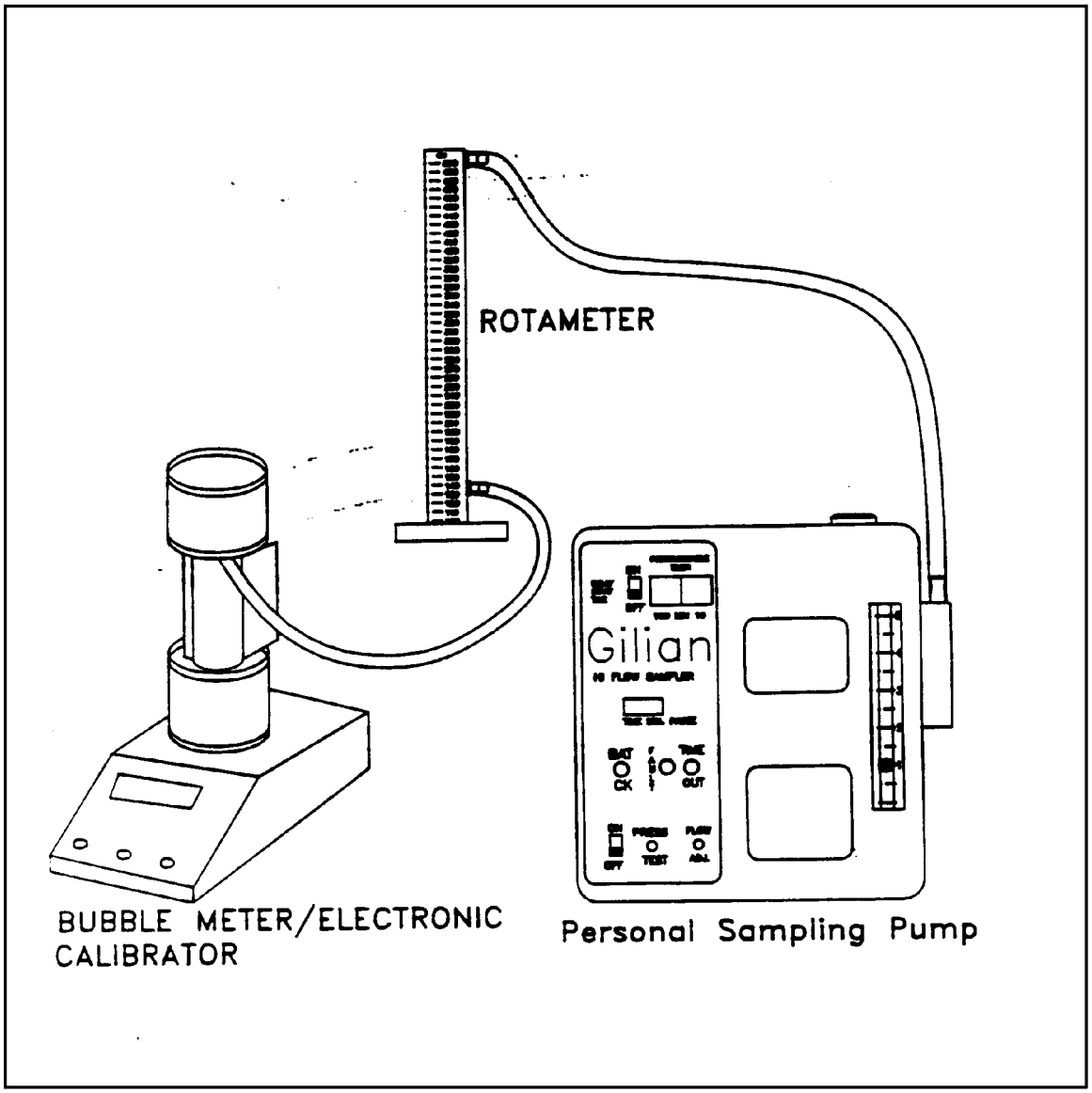
FIGURE 3. Calibrating a Personal Sampling Pump with a Bubble Meter



APPENDIX B (Cont'd)

Figures

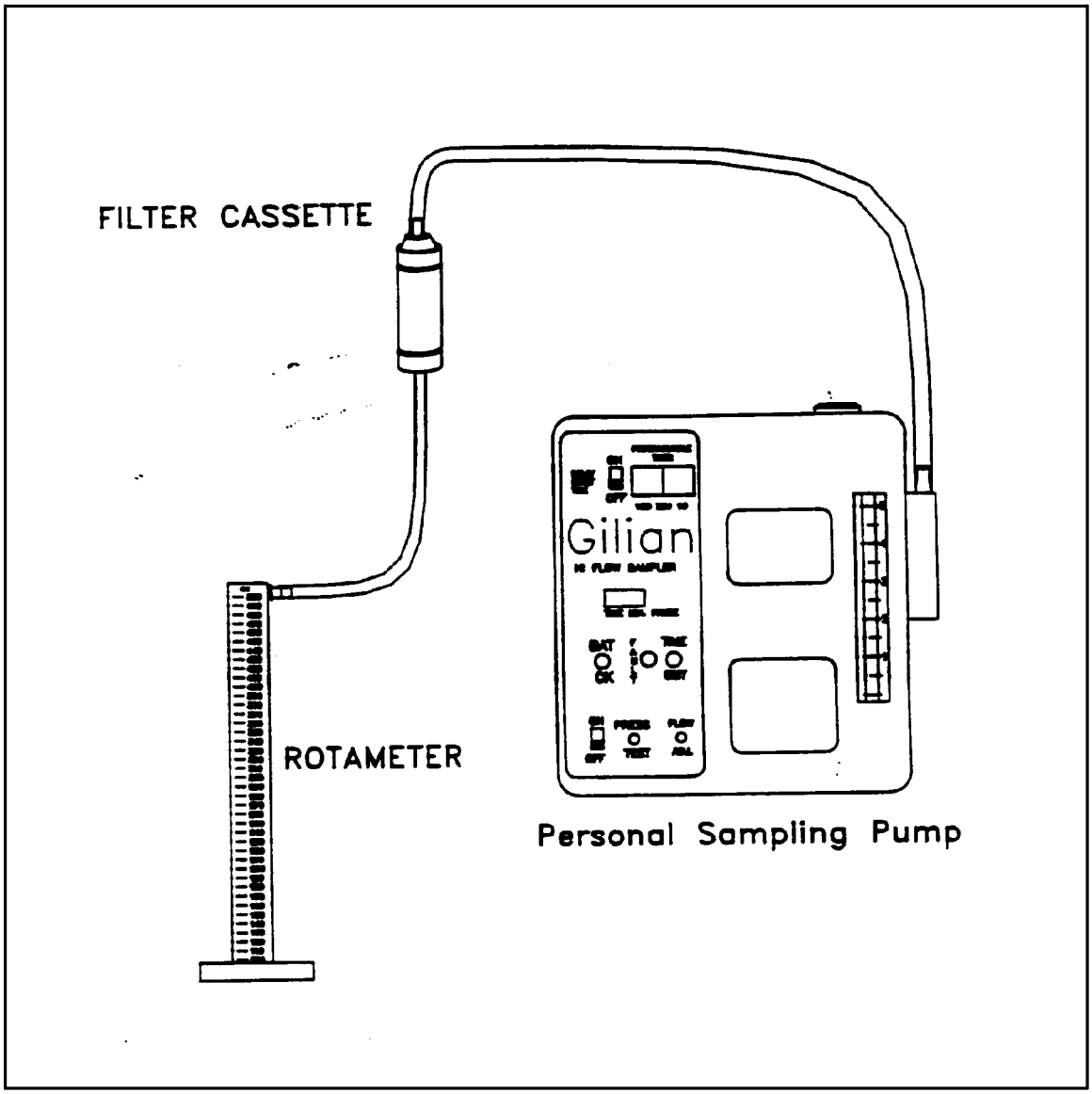
FIGURE 4. Calibrating a Rotameter with a Bubble Meter



APPENDIX B (Cont'd)

Figures

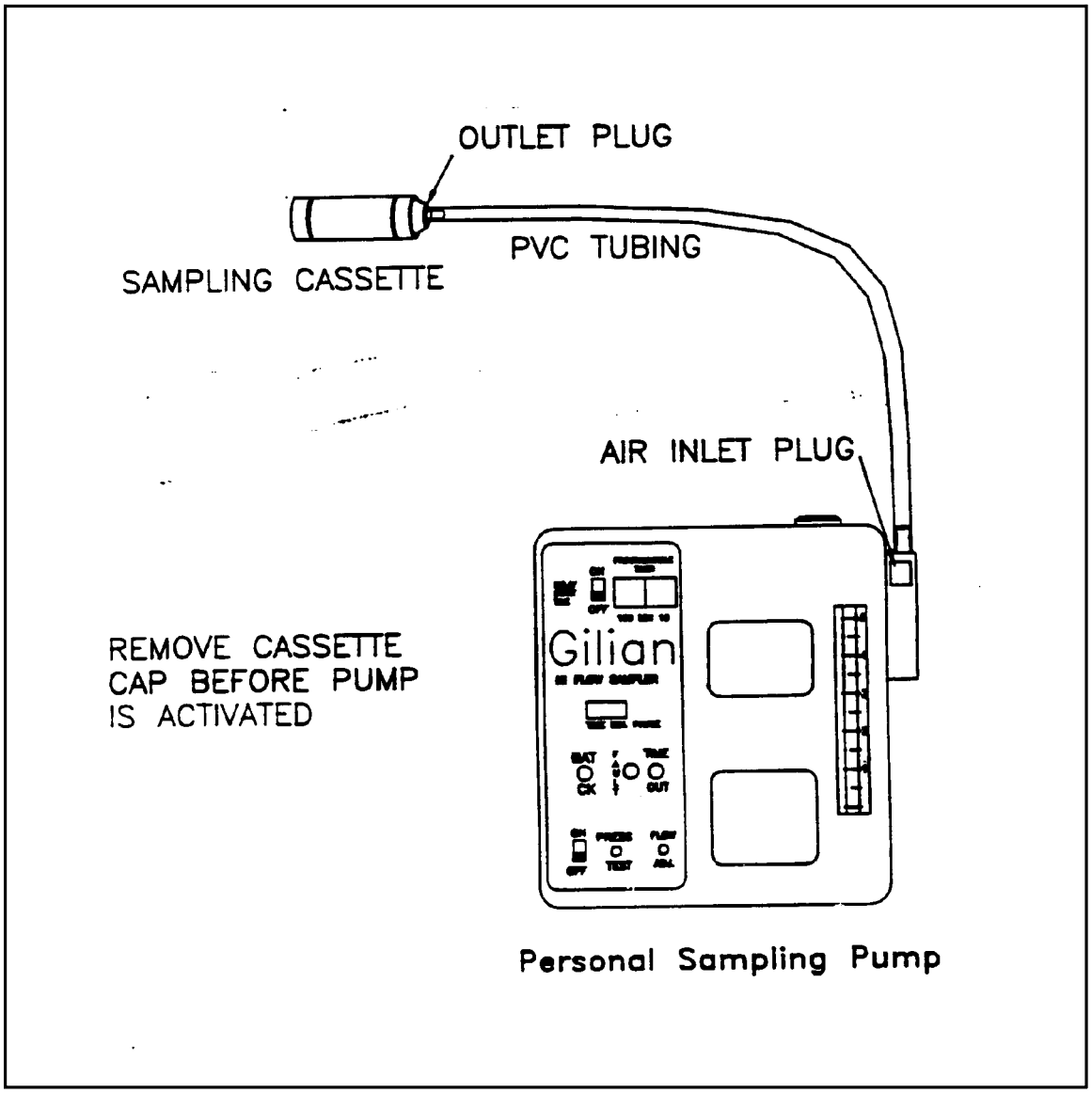
FIGURE 5. Calibrating a Sampling Pump with a Rotameter



APPENDIX B (Cont'd)

Figures

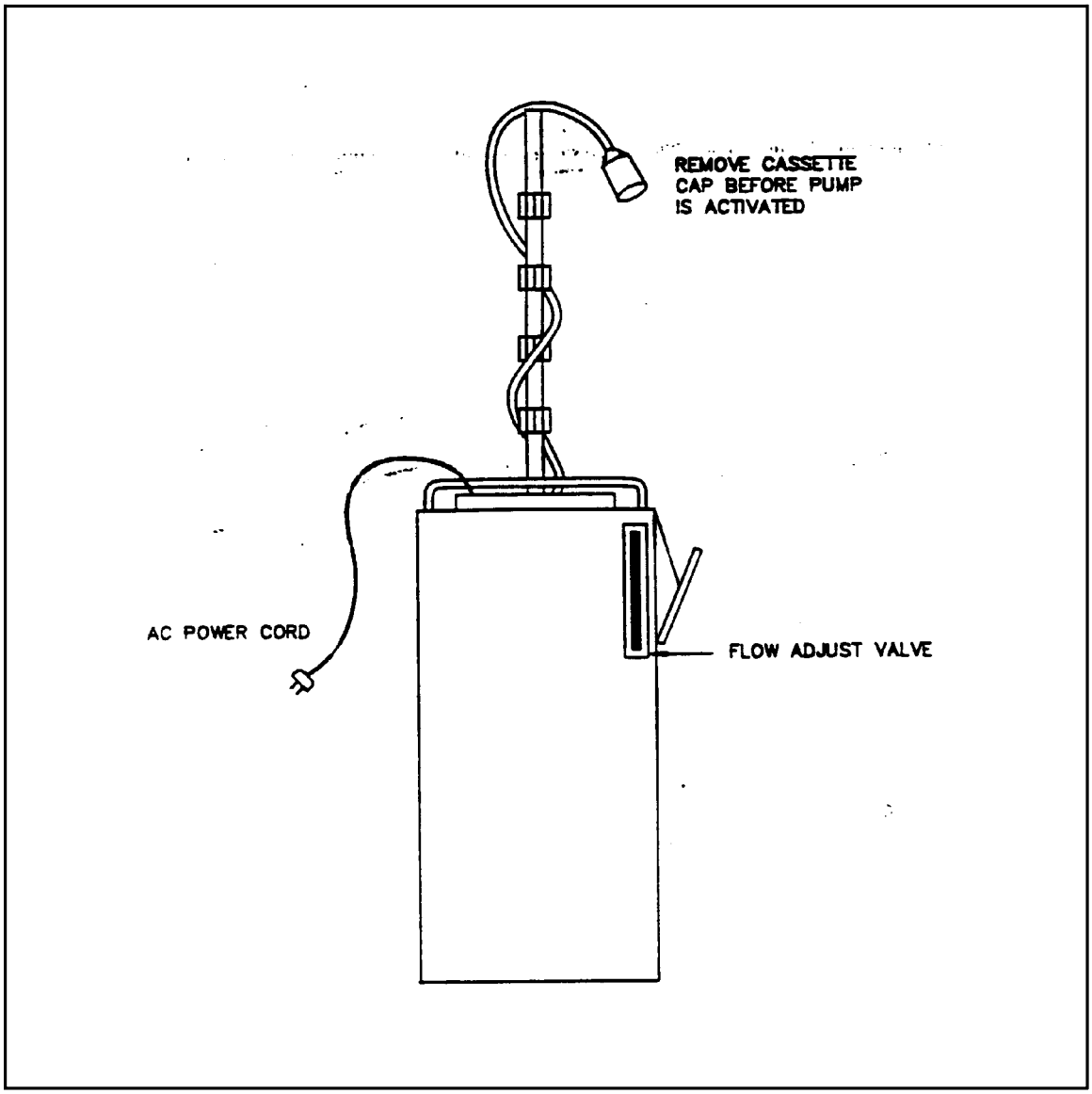
FIGURE 6. Personal Sampling Train for Asbestos



APPENDIX B (Cont'd)

Figures

FIGURE 7. High Flow Sampling Train for Asbestos



Libby Asbestos Project
Draft Pilot Study
30-point Dust Composite Sampling

Sample Locations

- Sample points will be collected from areas classified on a scale of accessibility. This scale was designed by EPA and used during assessments of residential properties after the World Trade Center (WTC) attacks:
    1. **Accessible** areas refer to locations where exposures are most likely to occur – places where dust accumulates and is encountered daily. This includes soft surfaces such as carpet (not including movable floor mats), upholstered furniture, floors and waist-high hard surfaces such as counter tops and non-carpeted floors.
    2. **Infrequently accessed** areas refer to locations where dust may accumulate, but exposures are likely to occur infrequently. This includes areas on tops of shelves, entertainment centers, and refrigerators, etc.
    3. **Inaccessible** areas refer to locations where dust may accumulate but exposures occur only rarely, such as behind refrigerators or other large infrequently moved objects.

- To the extent possible, the sub-sample locations will be collected from each type of accessibility area as indicated below.
    1. **Accessible** target areas, if present, and in order of priority:
        a. Flooring (soft or hard surface) at the main entrance used by occupants
        b. Flooring at the secondary or less heavily used entrance to the home
        c. Flooring in the center of the most frequently used living area of the home
        d. Flooring in an acknowledged or evident route of high traffic (i.e., hallway or other thoroughfare)
        e. Flooring in the kitchen
        f. Kitchen counter tops
        g. Table tops in the following rooms: dining room, living room, or family room
        h. Table tops (e.g., night stands, bureaus) in bedrooms
        i. Window sills in the dining room, living room, or family room
        j. Window sills in the bedrooms
        k. Upholstered furniture in the living room

    2. **Infrequent** target areas, if present, and in order of priority:
        a. Inside forced air floor or ceiling vents in the living room
        b. Inside forced air floor or ceiling vents in the bedrooms
        c. Top of the refrigerator
        d. Top of bookshelves
        e. Shelves of bookshelves

f. Top of the hot water heater
g. Beneath the sofa or other large pieces of furniture in the living room
h. Beneath the bed or other large pieces of furniture in bedrooms
i. Inside kitchen cabinets most frequently accessed

3. **Inaccessible** target areas, if present, and in order of priority:
   a. Beneath infrequently moved heavy appliances (e.g., refrigerator, washing machine, etc.)
   b. Corners of closets or other similar small areas not frequently accessed or cleaned

Sample Collection

A total of 4 samples will be collected as follows from the main living floor at each property:

- One 30-point composite sample from the main living floor
- One 15-point composite sample from the main living floor
- Two 3-point composite samples per the current site protocol (EPA 2003) on the main living floor

Each sub-sample point will cover 100 square centimeters (cm^2) using disposable paper templates for measurement. The pilot study will be completed using sampling procedures described in ASTM 5755-03 (unless noted).

- Point distribution - The 30- and 15-point composite samples should be collected from approximately the same number of sub-sample locations from **Accessible** and **Infrequent** target areas at each property. These types of areas should account for 80% of the locations sampled. That is, for a 30-pt sample, 24 points should be collected from **Accessible** and **Infrequent** target areas, and 6 from **Inaccessible** target areas. For a 15-pt sample, 12 points should be collected from **Accessible** and **Infrequent** target areas and 3 from **Inaccessible** target areas.

- Flow rates – A flow rate of 2 liters/minute should be used for collection of all samples. Flow rates should be verified before sample collection begins and after sample collection is completed for all sample types (30-point, 15-point, and 3-point composites). An additional calibration check should be performed for the 30-point and 15-point samples halfway through the collection period.

- Duration of sample collection – The 30-point composite samples should be collected over 15 minutes, with each template collected in 30 seconds. The 15-point composite samples should be collected over 7.5 minutes, with each template collected in 30 seconds. The two 3-point composite samples should each be collected over 6 minutes, with each template collected in 2 minutes.

Documentation

For the purposes of the pilot study, a field sample data sheet and logbook entry will be completed for each sample. For logbook entries, in addition to information required by CDM SOP 4-1 (Field Logbook Content and Control), the following will be recorded: Index (i.e., sample) ID, specific location and area (in cm^2) of each sub-sample, total flow rate, sample time, and times/flow rates of all calibration checks.

Sample Custody

All pilot study dust samples will be handled in accordance with current project sample custody procedures.

Long term questions

- Is it necessary to document the location of all 30 or 15 points if this becomes our long term procedure? – If so how will FSDSs and Libby2 need to be modified? (See mockup of FSDSs)
- Is it also necessary to document all start and stop times – the times will occur within the same minute for back to back sub-locations? Or can just the total flow rate and sample time be given?

APPENDIX B

TAPE TROY FIELD OFFICE (TFO) MODIFICATION RECORDS

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 Asbestos Property Evaluation Work Plan
- ○ Other (Title and approval date): _____

Site-Specific Guidance/SOP (Number and Revision No.) (check one):

- ◉ CDM-LIBBY-10, Revision 0 (30-point dust sample collection)
- ○ CDM-LIBBY-05, Revision 2 (30-point soil sample collection)
- ○ CDM-LIBBY-06, Revision 1 (Visible Vermiculite Estimation)

Other (Title, Number/Revision): _____

Requester: CATHERINE LECOURS Title: Project Mgr
Company: Tetra Tech EM Inc / DEQ Date: 5-8-07

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): p4 and p5 of SOP p.6 30 aliquot is now 10 aliquot and distribution is 4 accessible, 4 infrequent and 2 inaccessible

Field logbook and page number where Modification is documented (or attach associated correspondence): N/A

Potential Implications of Modification: less aliquot of dust sample

Duration of Modification (check one):

- ○ Temporary
  - Date(s): _____ AD- _____
  - BD(s)- _____ TT(s)- _____
- ◉ Permanent (Proposed Text Modification Section) Effective Date: 5-8-07

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): will indicate modification for TAPE in TAPE-Specific work plan

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: 5-8-07
(DEQ Project Manager or designate)

EPA Review and Approval: _____ Date: 5/8/07
(USEPA RPM or designate)

Revised April 29, 2007

Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO-00002 (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 Asbestos Property Evaluation Work Plan
- ○ Other (Title and approval date): _____

Site-Specific Guidance/SOP (Number and Revision No.) (check one):

- ○ CDM-LIBBY-10, Revision 0 (30-point dust sample collection)
- ○ CDM-LIBBY-05, Revision 2 (30-point soil sample collection)
- ◉ CDM-LIBBY-06, Revision 1 (Visible Vermiculite Estimation)

Other (Title, Number/Revision): _____

Requester: CATHERINE LeCOURS Title: Project Manager
Company: Tetra Tech EM Inc / DEQ Date: 5·8·07

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): maximum # of P.I. is 30 for a single use area, evenly distributed throughout area and corresponds with sample aliquot points

Field logbook and page number where Modification is documented (or attach associated correspondence): N/A

Potential Implications of Modification: Less visible inspection points for TAPE inspections

Duration of Modification (check one):

- ○ Temporary
  - Date(s): _____ AD- _____
  - BD(s)- _____ TT(s)- _____
- ◉ Permanent (Proposed Text Modification Section) Effective Date: 5·8·07

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): will indicate modification for TAPE in TAPE · specific work plan

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: 5·8·07
(DEQ Project Manager or designate)

EPA Review and Approval: _____ Date: 5/8/07
(USEPA RPM or designate)

Revised April 29, 2007

Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO-0 0 0 0 3 (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 Asbestos Property Evaluation Work Plan
- ○ Other (Title and approval date): _____

Site-Specific Guidance/SOP (Number and Revision No.) (check one):

- ● CDM-LIBBY-10, Revision 0 (30-point dust sample collection)
- ○ CDM-LIBBY-05, Revision 2 (30-point soil sample collection)
- ○ CDM-LIBBY-06, Revision 1 (Visible Vermiculite Estimation)

Other (Title, Number/Revision): _____

Requester: CATHERINE LECOURS Title: Project Manager
Company: Tetra Tech EM Inc / DEQ Date: 7-12-07

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): to utilize the prioritization scheme from the Dust Composite Sampling Pilot Study, Rev 0 dated May 16, 2007 for the TAPE aliquot dust collection

Field logbook and page number where Modification is documented (or attach associated correspondence): N/A

Potential Implications of Modification: increase probability of well represented dust sample

Duration of Modification (check one):

- ○ Temporary
  - Date(s): _____ AD- _____
  - BD(s)- _____ TT(s)- _____
- ● Permanent (Proposed Text Modification Section) Effective Date: 7-12-07

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): addition of heirarchy to work plan Section 4.4.2.1

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: [signature] Date: 7-12-07
(DEQ Project Manager or designate)

EPA Review and Approval: [signature] Date: 7/12/07
(USEPA RPM or designate)

Revised April 29, 2007

Record of Modification

to the

Troy Sampling and Quality Assurance Project Plan

Field Activities

TFO- 0 0 0 0 4 (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.

TFO–4 was prepared in response to a specific modification on a single parcel (AD-200917).

TFO-4 has been scanned to the parcel's electronic file and does not affect the overall project.

Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities

TFO-0 0 0 0 5 (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 Asbestos Property Evaluation Work Plan
- ○ Other (Title and approval date): _____

Site-Specific Guidance/SOP (Number and Revision No.) (check one):

- ○ CDM-LIBBY-10, Revision 0 (30-point dust sample collection)
- ○ CDM-LIBBY-05, Revision 2 (30-point soil sample collection)
- ○ CDM-LIBBY-06, Revision 1 (Visible Vermiculite Estimation)

Other (Title, Number/Revision): _____

Requester: CATHERINE LeCOURS Title: PROJECT MANAGER
Company: Tetra Tech EM Inc / DEQ Date: 8-1-07

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 of TPE, only collect dust samples if visible vermic in exterior or interior, former mine or ARD are affirmative.

Field logbook and page number where Modification is documented (or attach associated correspondence): N/A

Potential Implications of Modification: not collect dust samples at all properties

Duration of Modification (check one):

- ○ Temporary
  - Date(s): _____ AD- _____
  - BD(s)- _____ TT(s)- _____
- ◉ Permanent (Proposed Text Modification Section) Effective Date: 8-1-07

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): N/A

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: [signature] Date: 8-1-07
(DEQ Project Manager or designate)

EPA Review and Approval: [signature] Date: 8-1-07
(USEPA RPM or designate)

Revised April 29, 2007

2007-Sep-21 11:59 AM U.S. EPA (422lmr) 3033127203 2/3

Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities

TFO-0 0 0 0 6 (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):

- (X) 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, Revision 0 (30-point dust sample collection)
- O CDM-LIBBY-05, Revision 2 (30-point soil sample collection)
- O CDM-LIBBY-06, Revision 1 (Visible Vermiculite Estimation)

Other (Title, Number/Revision): _____

Requester: CATHERINE LeCOURS Title: PROJECT MANAGER
Company: Tetra Tech EM Inc / DEQ Date: 8-23-07

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 of TAPE, supercedes TFO-00005 and reverts dust sampling to all buildings

Field logbook and page number where Modification is documented (or attach associated correspondence): N/A

Potential Implications of Modification: collect dust samples at all buildings

Duration of Modification (check one):

- O Temporary
  - Date(s): _____ AD- _____
  - BD(s)- _____ TT(s)- _____
- (X) Permanent (Proposed Text Modification Section) Effective Date: 8-23-07

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): N/A

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: [signature] Date: 8-23-07
(DEQ Project Manager or designate)

EPA Review and Approval: [signature] Date: 8-23-07
(USEPA RPM or designate)

Revised April 29, 2007

2007-Sep-21 11:59 AM U.S. EPA (422lmr) 3033127203 3/3

Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO-00007 (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 Asbestos Property Evaluation Work Plan through SOP change
- ○ Other (Title and approval date): _____

Site-Specific Guidance/SOP (Number and Revision No.) (check one):

- ○ CDM-LIBBY-10, Current Revision (30-point dust sample collection)
- (●) 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 MGR
Company: Tetra Tech EM Inc DEQ Date: 11·15·07

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 of SOP c
Collect soil samples in SLA's with visible vermic based on higher frequency of homeowner purchase / potting soil

Field logbook and page number where Modification is documented (or attach associated correspondence):
N/A

Potential Implications of Modification: ~~not clear~~ further characterization of SLA's

Duration of Modification (check one):

- ○ Temporary
  - Date(s): _____ AD- _____
  - BD(s)- _____ TT(s)- _____
- (●) Permanent (Proposed Text Modification Section) Effective Date: 11·16·07

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): _____

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: [signature] Date: 11·16·07
(DEQ Project Manager or designate)

EPA Review and Approval: [signature] Date: 11/16/07
(USEPA RPM or designate)

Revised April 29, 2007

Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO – 0 0 0 0 8 (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 Asbestos Property Evaluation Work Plan
- ○ Other (Title and approval date):_____

Site-Specific Guidance/SOP (Number and Revision No.) (check one):

- ○ CDM-LIBBY-10, Current Revision (30-point dust sample collection)
- ○ 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: Montana DEQ Date: 04/29/2008

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): See following pages – revisit of select parcels from the 2007 inspections for confirmation and description of visible vermiculite in use areas and sampling of use areas

Field logbook and page number where Modification is documented (or attach associated correspondence):

Potential Implications of Modification: _____

Duration of Modification (check one):

- ○ Temporary
  - Date(s):_____ AD-_____
  - BD(s)-_____ TT(s)-_____
- ● Permanent (Proposed Text Modification Section) Effective Date: May 8, 2008

Proposed Text Modifications in Associated Document (attach additional sheets if necessary):_____

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 8, 2008
(DEQ Project Manager or designate)

EPA Review and Approval: Kathryn S. Hernandez Date: May 13, 2008
(USEPA RPM or designate)

Revised April 29, 2007

Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO - 0 0 0 0 9 (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 Asbestos Property Evaluation Work Plan
- ○ Other (Title and approval date): _____

Site-Specific Guidance/SOP (Number and Revision No.) (check one):

- ○ CDM-LIBBY-10, Current Revision (30-point dust sample collection)
- ○ 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 Date: June 3, 2008

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): Collect "field split" samples only when Use Area contains sufficient volume. Split will involve collecting double the volume of soil, homogenizing in single bowl, filling two separate baggies and identifying with two separate sample identification numbers, referenced to each other in Scribe.

Field logbook and page number where Modification is documented (or attach associated correspondence):
_____

Potential Implications of Modification: None
_____

Duration of Modification (check one):

- ○ Temporary
  - Date(s): _____ AD- _____
  - BD(s)- _____ TT(s)- _____
- ● Permanent (Proposed Text Modification Section) Effective Date: June 4, 2008

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): Split samples will only be collected for a limited time, therefore no text changes are necessary.

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: 6-05-08
(DEQ Project Manager or designate)

EPA Review and Approval: Jocelyn S. Hernandez Date: 6/05/08
(USEPA RPM or designate)

Revised April 29, 2007

RECORD OF MODIFICATION
TFO-00008

TO THE

TROY ASBESTOS PROPERTY EVALUATION WORK PLAN

CONFIRMATION AND DESCRIPTION OF VISIBLE VERMICULITE IN USE AREAS
AND
SAMPLING OF SPECIFIC USE AREAS
Troy Operable Unit Number 7
of the Libby Asbestos Superfund Site

Prepared for:

MONTANA DEPARTMENT OF ENVIRONMENTAL QUALITY
Remediation Division
P.O. Box 200901
Helena, Montana 59620

Contract Number 402026
Task Order Number 3 & 20

Prepared by:

TETRA TECH EM INC.
Power Block Building, Suite 612
7 West 6th Avenue
Helena, Montana 59601
(406) 442-5588

May 13, 2008

CONFIRMATION AND DESCRIPTION OF VISIBLE VERMICULITE IN USE AREAS AND SAMPLING OF SPECIFIC USE AREAS

As part of the Troy Asbestos Property Evaluation (TAPE) project 2008 field season, Tetra Tech will complete an inspection and/or collect a soil sample for approximately 462 Troy Operable Unit 7 (OU7) parcels that contain approximately 1,743 Use Areas for confirmation, semi-quantification, and description of visible vermiculite in the exterior soils and collection of soil samples from Specific Use Areas (SUA). **Table 1** provides the number of OU7 parcels and Use Areas to be inspected and sampled. The primary reasons for these inspections and additional sampling will be to address:

(a) the poor correlation between the PLM-VE analytical results and the visible vermiculite observations recorded at Use Areas in OU7 through the confirmation and semi-quantification of the presence or absence of visible vermiculite in Use Areas inspected in 2007,

(b) the need for soil samples from all SUAs as documented in Record of Modification TFO-00007 through the collection of samples from SUAs not previously sampled, and

(c) the accurate description of visible vermiculite observed and reported as "store purchased potting soil" in order to confirm the relationship between vermiculite observed in "potting soil" and analytical results in OU7 that may differ from that relationship as observed in OU4 through documentation of such descriptions during the confirmation and sampling identified above in items (a) and (b).

TABLE 1: Number of Troy Parcels and Use Areas to be Inspected and Sampled

Objectives	Number of Parcels	Number of Use Areas (excluding Non Use Areas)
One: Confirm and semi-quantify the presence or absence of visible vermiculite in Use Areas [item (a) above] and accurately describe the visible vermiculite if present [item (c) above]	462	1,743
Two: Collect soil samples from SUAs not sampled in 2007 [item (b) above]	115	144
Total Unique Numbers (not additive; some parcels/Use Areas under both objectives)	**462**	**1,743**

Two objectives were developed to determine the types and numbers of parcels and Use Areas to inspect and sample.

Objective One: **Confirm and semi-quantify the presence or absence of visible vermiculite in Use Areas and accurately describe the visible vermiculite if present.** The number of parcels to inspect was derived by subtracting the sum of the parcels that would be revisited because they meet one of the emergency removal criteria[1] requiring a pre-design inspection (PDI) plus the parcels with only Non Use Areas (essentially undeveloped properties) from 535, the total number of parcels inspected during 2007. The equation below provides a mathematical expression for this determination:

Parcels for Inspection = 535 – (Parcels meeting removal criteria[1] + Parcels with only Non Use Areas)

Objective Two: **Collect soil samples from SUAs sampled in 2007.** Based on preliminary data from 2007, DEQ, in consultation with EPA, modified the sampling protocol to include a soil sample from all SUAs, regardless of presence or absence of visible vermiculite. (Please see Record of Modification TFO-00007.) Therefore, DEQ has directed Tetra Tech to collect soil samples from SUAs not sampled in 2007.

Tetra Tech's approach for completing these objectives includes training six Tetra Tech TAPE field staff plus three TAPE managers (the Field Team Leader and two quality assurance [QA] managers) to accurately recognize, identify and semi-quantify visible vermiculite in exterior soils, if present. These nine Tetra Tech individuals will be trained by two experienced CDM field persons for approximately four days and will be known as Tetra Tech's Visible Vermiculite (VV) Core team members. The six TAPE field staff will be in the field more than 50 percent of the field season and four of the six will be working on TAPE inspections 100 percent of the time. The four days of training inspections will provide the opportunity for each Tetra Tech VV Core team member to work side by side with CDM, asking questions and learning the skills necessary to identify and semi-quantitatively assess the amount visible vermiculite present. On Day 1, the nine Tetra Tech VV Core team members will meet with CDM staff for up to two hours to go over the basics and get ready for first site visit. The group will be divided into two teams with one CDM person for each team. Each team will visit two to three Troy parcels on Day 1. Days 2, 3, and 4 will have similar schedules with each of the two teams visiting two to three Troy parcels per day depending on the size of parcels and the number of Use Areas. Tetra Tech will identify the training parcels, in consultation with CDM, and schedule the inspections with the property owners. The Troy parcels visited during the four training days will include a majority of parcels where visible vermiculite was recorded during the 2007 TAPE inspections. There will be a mixture of parcels with visible vermiculite at multiple locations and some with visible vermiculite only in distinct, small areas. At least

[1] Concentration of Libby asbestos in soil greater than or equal to 1 percent of sample volume.

two Troy parcels (one for each team) will have no exterior triggers for cleanup. The 2007 inspection and analytical results for these Troy parcels will not be provided to the Tetra Tech VV Core team members prior to the training inspections so that there will be no preconceived ideas or biases. Later during the 2008 field season, qualified CDM staff may periodically spend one or two days with the TAPE inspection teams to ensure the TAPE teams are identifying and documenting visible vermiculite consistent with CDM-LIBBY-06 (Appendix B to the TAPE Work Plan). A TAPE field team schedule will be provided to Volpe and CDM so audits may be planned accordingly.

After Tetra Tech completes approximately 100 parcel inspections for Objective One, the visible vermiculite results and descriptions from 2008 will be summarized and compared to the results of the visible vermiculite inspections and descriptions of 2007 from those same Use Areas. The results' comparisons will be reported to the DEQ and EPA and a meeting will be scheduled with all parties to determine the necessity of continuing with the remaining Objective One inspections. Tetra Tech will inspect all parcels identified for Objective Two, and complete all activities in Objective One for those parcels, regardless of the results of the comparison described above. Tetra Tech will continue to conduct the 2008 TAPE inspections for Troy parcels, including inspections of Use Areas for visible vermiculite and the description of such vermiculite, as described in the TAPE Work Plan (Tetra Tech, July 2007) as modified. Tetra Tech will bring on additional staff, if needed, to complete the necessary number of scheduled 2008 TAPE inspections, plus the visible vermiculite inspections and sampling of Use Areas. Tetra Tech will ensure that all new 2008 TAPE inspection teams have at least one Tetra Tech VV Core team member to verify the presence or absence of visual vermiculite and describe the vermiculite in the Use Area soils. Tetra Tech believes that all scheduled TAPE 2008 inspections and these additional inspections and sampling will be completed on time and within the estimated 2008 budget because of the following reasons and protocol modifications to Tetra Tech's 2008 field efforts:

1. Tetra Tech will staff the 2008 field team with approximately 60 percent (6 out of 10) dedicated and season-long field team members who will reside in Troy for the entire field season. Dedicated field staff will provide more experience and consistency for making visual field determinations, such as the identification of visible vermiculite in Use Area soils.
2. TAPE inspections during 2007 typically averaged 2 to 4 hours to complete (depending on size of parcel, number of inspectors [2 or 3], and number of buildings). Throughout the 2007 summer field season there were many shorter periods of time (30 to 90 minutes) when field crews did not have a scheduled TAPE inspection, but were able to "fit in" a less labor-intensive exterior only TAPE inspection (for example, open space, undeveloped properties) or re-visit parcels due to protocol and data collection modifications. The majority of these smaller, easily scheduled, short, fill-in efforts were completed during 2007. Completing the activities for Objectives One and Two may take from approximately 20 minutes up to a few hours (depending on size of parcel, and travel time to and from the site) and will be easy to fit in and complete on a regular fill-in basis.

Use Areas, except Non Use Areas, will be inspected for visible vermiculite in the soil and surface materials, semi-quantitatively assessed, and sampled (if necessary). For each inspection, the field team will bring along: (1) copies of the field sketches from 2007; (2) field forms for recording inspection data; and (3) field equipment to conduct the visible vermiculite inspection and to collect the soil samples.

Use Areas will be inspected and sampled using the same overriding procedures as the 2007 TAPE inspections, as modified, except a hand lens or magnifying glass will be used to better observe and identify small pieces of vermiculite in the Use Area soils or surface materials Each soil aliquot will be examined for the presence of visible vermiculite and the amount of vermiculite will be semi-quantified as none, low, intermediate, or high using the procedures defined in CDM-LIBBY-06 (Appendix B to the TAPE Work Plan) and accurately described. The Use Area will be closely inspected for the presence of visible vermiculite, but the soil aliquot will not be placed in a plastic bag or bowl for compositing or sampling, unless necessary to meet Objective Two. After careful inspection for visible vermiculite, each soil aliquot will be returned to its approximate original location.

To minimize field recording time and file transfer time, the inspection results (none, low, intermediate, high, description, and sample identification number) will be recorded in a tabular format on field forms created specifically for these activities. The PDAs will not be used to enter data and GPS locations will not be collected for these inspections. At the end of each day that an inspection is performed, the tabulated results will be entered into Scribe by the sample database coordinator. A second inspection property sketch will be prepared for TAPE parcels that have Use Area changes (including Use Areas that no longer exist), different Use Area boundaries, or if visible vermiculite was observed from new or different locations during the inspection. If the parcel Use Areas are identical to those shown on the original 2007 TAPE inspection sketch and no new visible vermiculite is observed, only the notes and details of the inspection will be recorded. The 2008 inspection field team may elect to photo-document specific conditions or changes to the parcel, especially if visible vermiculite is found during the inspection. All new photographs will be recorded on the field forms, downloaded and saved into the Troy parcel electronic file. All inspection field forms will also be scanned and saved into the Troy parcel electronic file. Tetra Tech will manage the 2008 inspection data and any changes to the 2007 inspection data for visible vermiculite and sampling according to the approved TAPE Data Management Plan.

Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO – 0 0 0 1 0 (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 Asbestos Property Evaluation Work Plan
- ○ Other (Title and approval date):_____

Site-Specific Guidance/SOP (Number and Revision No.) (check one):

- ○ CDM-LIBBY-10, Current Revision (30-point dust sample collection)
- ○ 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: Montana DEQ Date: June 13, 2008

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): Void TFO-00009 (no longer collect "field split" soil samples) and increase soil sample volume by 50%.

Field logbook and page number where Modification is documented (or attach associated correspondence):
_____

Potential Implications of Modification: None.

Duration of Modification (check one):

- ○ Temporary
  - Date(s):_____ AD-_____
  - BD(s)-_____ TT(s)-_____
- ● Permanent (Proposed Text Modification Section) Effective Date: June 13, 2008

Proposed Text Modifications in Associated Document (attach additional sheets if necessary):_____
_____
_____

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: 6-13-08
(DEQ Project Manager or designate)

EPA Review and Approval: Kathryn S. Hernandez Date: 6-14-08
(USEPA RPM or designate)

Revised April 29, 2007

Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO - 0 0 0 1 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 Asbestos Property Evaluation Work Plan
- ○ Other (Title and approval date): _____

Site-Specific Guidance/SOP (Number and Revision No.) (check one):

- ○ CDM-LIBBY-10, Current Revision (30-point dust sample collection)
- ○ 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 Date: December 22, 2008

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): Cease the re-inspection of use areas pursuant to TFO-00008. Please see following memorandum for justification.

Field logbook and page number where Modification is documented (or attach associated correspondence): _____

Potential Implications of Modification: None.

Duration of Modification (check one):

- ○ Temporary
  - Date(s): _____ AD- _____
  - BD(s)- _____ TT(s)- _____
- ● Permanent (Proposed Text Modification Section) Effective Date: December 22, 2008

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): None for the TAPE document, only the record of modification.

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: 12.22.08
(DEQ Project Manager or designate)

EPA Review and Approval: _____ Date: 12-22-08
(USEPA RPM or designate)

Revised April 29, 2007

August 6, 2008

To: Kathryn Hernandez, Liaison RPM
Troy Operable Unit, Libby Asbestos Site

From: Catherine LeCours, State Project Manager
Troy Operable Unit, Libby Asbestos Site

Subject: Re-Inspection for Visible Vermiculite at Troy properties

Attached, please find a spreadsheet containing the results of 100 parcels/522 Use Area re-inspections performed by Tetra Tech. The re-inspections were performed pursuant to TFO-08 of the TAPE. You are already in receipt of the CDM Memorandum dated July 31, 2008 entitled "Troy Visible Vermiculite Inspection, First Event."

The data is summarized as follows:

Number of Use Areas where:	Tetra Tech TFO-08	CDM First Event
Greater than 50% of the inspection points reported as Intermediate or High were re-evaluated to None or Low	17	1
Greater than 50% of the inspection points reported as None or Low were re-evaluated to Intermediate or High	4	1
All inspection points were reported as None and re-evaluated to at least one point in Low [1]	96	6
At least one inspection point was reported as either Low, Intermediate or High and re-evaluated to all points in None [2]	32	13

[1] Note: Based upon casual observation of the results, the vast majority of these were re-evaluated to less than 10 inspection points identified (primarily one or two only) in the Low category in 2008. The majority of these had analytical results in the A or B1 bins.

[2] Note: Based upon casual observation of the results, the vast majority of these originally had less than 10 inspection points identified (primarily one or two only) in the Low category. Analytical results are not commonly available as most of these were SUA's with visible in 2007 and thus not sampled. However, samples were collected this year as part of the re-inspection activities.

All accessible Specific Use Areas requiring a soil sample pursuant to Objective 2 of TFO-08 have been sampled.

Based on the summary of these two activities and the questions surrounding the actual reproducibility of the visual point inspections, I would like to propose that the activities pursuant to TFO-08 cease at this time.

Please don't hesitate to contact me with any questions you may have regarding this data.

I look forward to your affirmative response and will prepare another TFO accordingly.

Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO - 0 0 0 1 2 (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 Asbestos Property Evaluation Work Plan
- ○ Other (Title and approval date): _____

Site-Specific Guidance/SOP (Number and Revision No.) (check one):

- ● CDM-LIBBY-10, Current Revision (30-point dust sample collection)
- ○ 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 Date: December 22, 2008

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): Eliminate the collection of dust samples pursuant to the TAPE Work Plan. Please see attached Memorandum for justification.

Field logbook and page number where Modification is documented (or attach associated correspondence): _____

Potential Implications of Modification: No dust samples will be collected in residences inspected in the future.

Duration of Modification (check one):

- ○ Temporary
  - Date(s): _____ AD- _____
  - BD(s)- _____ TT(s)- _____
- ● Permanent (Proposed Text Modification Section) Effective Date: December 22, 2008

Proposed Text Modifications in Associated Document (attach additional sheets if necessary): None for the TAPE document, only the record of modification.

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: 12·22·08
(DEQ Project Manager or designate)

EPA Review and Approval: _____ Date: 12-22-08
(USEPA RPM or designate)

Revised April 29, 2007

December 22, 2008

To: Victor Ketellapper, Project Team Lead
Libby Asbestos Site

From: Catherine LeCours, State Project Manager
Troy Operable Unit, Libby Asbestos Site

Subject: Collection of Dust Samples for the Troy Asbestos Property Inspections pursuant to the TAPE Work Plan

Currently, dust samples are being collected at each commercial and residential parcel in the Troy Operable Unit pursuant to the TAPE Work Plan (Tetra Tech, July 2007). Upon review of the available dust analytical results in comparison to the other removal "triggers" identified for the site, we have concluded the dust analytical results do not contribute meaningful information from which we make clean up decisions.

Specifically, in Troy, we have identified 78 buildings (or parts of buildings) in which the field inspection teams noted visible vermiculite in interior living spaces (excluding attics). Not one of the associated dust samples were above our current trigger level of 5,000 s/cc. Conversely, we have 6 dust samples above the current trigger level with no interior visible vermiculite noted. Only two of those parcels have no other explanation to explain the presence of the vermiculite. Therefore, the lack of information provided by a dust analytical result does not justify the expense of the collection.

Please note that dust samples are no longer collected in OU 4. From the historical samples, less than three of those have been used independently for any decision making.

I am requesting that you approve TFO-00012 to discontinue the collection of dust samples effective immediately.

Thank you for your consideration and please do not hesitate to contact me if you have further questions or concerns.

Record of Modification

to the

Troy Sampling and Quality Assurance Project Plan

Field Activities

TFO – 0 0 0 1 3 (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 Asbestos Property Evaluation Work Plan
- ○ Other (Title and approval date):_____

Site-Specific Guidance/SOP (Number and Revision No.) (check one):

- ○ CDM-LIBBY-10, Current Revision (30-point dust sample collection)
- ○ 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: Montana DEQ Date: May 27, 2009

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): Perform aggressive attic inspections to thoroughly identify the presence of vermiculite attic insulation. These procedures will be implemented in future inspections as well as re-visits when possible. See attached Standard Operating Procedure for TAPE Aggressive Attic Inspection Activites.

Field logbook and page number where Modification is documented (or attach associated correspondence):

_____

Potential Implications of Modification: Additional time and effort to complete attic inspections with a more thorough and quantitative result.

Duration of Modification (check one):

- ○ Temporary

  Date(s):_____ AD-_____

  BD(s)-_____ TT(s)-_____

- ● Permanent (Proposed Text Modification Section) Effective Date: May 27, 2009

Proposed Text Modifications in Associated Document (attach additional sheets if necessary):_____

_____

_____

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: 5-27-09
(DEQ Project Manager or designate)

EPA Review and Approval: _____ Date: 6-8-09
(USEPA RPM or designate)

Revised April 29, 2007

STANDARD OPERATING PROCEDURES
FOR
TROY ASBESTOS PROPERTY EVALUATION
AGGRESSIVE ATTIC INSPECTION ACTIVITIES
TO VERIFY PRESENCE OR ABSENCE OF
VERMICULITE-CONTAINING INSULATION

Prepared for:

MONTANA DEPARTMENT OF ENVIRONMENTAL QUALITY
Remediation Division
P.O. Box 200901
Helena, Montana 59620

Contract Number 407026
Contract Task Order Number 41

Prepared by:

TETRA TECH EM INC.
Power Block Building, Suite 612
7 West 6th Avenue
Helena, Montana 59601
(406) 442-5588

May 2009

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1.0 SCOPE OF WORK

The work specified in this Standard Operating Procedure (SOP) includes all procedures necessary to complete aggressive attic inspection activities in areas above ceilings of primary and secondary buildings during the Troy Asbestos Property Evaluation (TAPE) inspections. These procedures supplement existing TAPE attic inspection procedures outlined in Section 4.4.1 of the TAPE Work Plan and Section 9.1 of the Health and Safety Plan in which attic access was limited to field technicians' torso levels. Libby amphibole (LA) can not be identified by visual inspection; therefore, field technicians will look specifically for vermiculite-containing insulation (VCI) during the attic inspections. All forms of vermiculite identified in attics, including expanded or unexpanded material will be deemed "VCI" by the field inspectors. The existing TAPE attic inspection procedures were sufficient to determine the presence or absence of VCI in most attics, but were not aggressive enough to determine the presence of VCI in all attics. The more aggressive procedures described in this SOP will be needed when there are limited attic access locations and the entire attic space can not be adequately inspected from the available access hatch locations. This SOP addresses attic inspection activities in partially finished or unfinished attics when a field technician is required to enter the attic with his/her full body. Such work includes, but is not limited to: installation of work area isolation and engineering controls; inspection activities as specified in this document; and equipment and personal decontamination procedures. All potential asbestos-related VCI inspection activities must be performed in compliance with applicable federal, state, and local regulations, and this document.

2.0 GENERAL TECHNICAL REQUIREMENTS

This section describes the general technical requirements for attic inspection procedures and provides guidelines for performing the work. All sections of this SOP shall be used when Tetra Tech EMI, Inc. (Tetra Tech) field personnel are performing aggressive attic inspections.

2.1 PERSONAL DOCUMENTATION REQUIREMENTS

- AHERA Contractor/Supervisor or Worker training certificates for all field technicians.
- Personnel respirator fit test records.
- Personnel medical examination records.

2.2 ISOLATION AND ENGINEERING CONTROLS

- Deactivate or isolate the HVAC system servicing the staging room.
- Seal passive air return grilles with one layer of polyethylene sheeting.
- Use fire resistant polyethylene sheeting materials.
- Install and activate a high efficiency particulate air (HEPA) filtered 600 cfm air filtration unit at an attic vent furthest from the access hatch point of entry, The unit will be activated once the access hatch is opened and remain running until decontamination procedures are completed.

2.3 POLYETHYLENE SHEETING

- Use fire resistant polyethylene sheeting materials.
- Use one layer of 6 mil on the floor and walls of the staging chamber.
- Use one layer of 6 mil over the passive HVAC return duct grills in the staging room.
- Use one layer of 6 mil on the floor outside the staging chamber.

2.4 EQUIPMENT

- Personal Protective Equipment (PPE)
- HEPA vacuum
- Polyethylene sheeting
- Asbestos disposal bags
- Ladder
- 600 cfm air filtration unit
- Water misting sprayer
- Duct tape and painter’s tape
- Disposable towels
- 5-gallon wash basin
- Flashlight and electric lights
- Support planks
- Camera
- Staging chamber

2.5 STAGING CHAMBER CONSTRUCTION

- A staging chamber shall be used for interior accessed aggressive attic inspections only. Staging chambers will not be required when accessing attics from the exterior of the buildings.
- The staging chamber will consist of a single stage “pop up” design mini-containment and will serve as the clean room, wash area and equipment room. The staging chamber shall be large enough to house a ladder and all necessary equipment. Modified staging chamber designs may be installed on site in areas where a pop up units can not be used; for example at stairways or within closets.
- The staging chamber will be installed at the base of the interior attic access hatch. A staging chamber will not be required when an exterior access hatch is used.
- A wash basin decontamination system shall be used inside the staging chamber.
- Equipment and waste decontamination will be completed inside the staging chamber.

2.6 PERSONAL PROTECTIVE EQUIPMENT

- The minimum respiratory protection required shall be half-face air purifying respirators upon successful completion of a negative exposure assessment (NEA), or upon providing of documentation meeting the requirements of 29 CFR 1926.1101 showing personnel air sampling data which justifies this level of respiratory protection.
- Prior to a successful NEA, all initial aggressive attic inspections shall be performed using powered air purifying respirators or full-face negative pressure respirators.
- Disposable outer garments such as Tyvek or polypropylene coveralls including head/foot coverings shall be worn. Inspectors shall use a double suit method.
- Protective eyewear should be worn if powered air purifying respirators or full face respirators are not used.
- Bump caps or hardhats and knee pads should be worn.

2.7 TRAINING

- A certified contractor/supervisor shall be on-site at all times during aggressive attic inspections. Training for the contractor/supervisor must meet the requirements of 29 CFR 1926.32 (f) and the EPA's Model Accreditation Plan (40 CFR 763).
- Field technicians who conduct aggressive attic inspections must have completed either AHERA worker training or contractor/supervisor training. Worker training must also meet the requirements of 29 CFR 1926.32. The minimum worker training must be a 32-hour OSHA-approved course.

2.8 AIR MONITORING BY TETRA TECH

- Tetra Tech will conduct personnel exposure air monitoring assessments using the OSHA compliance method (National Institute of Safety and Health [NIOSH] 7400 phase contrast microscopy [PCM]) during the first attic inspections of the field season and periodically throughout the remainder of the field season. Tetra Tech will discontinue or modify the procedures if fiber levels exceed limits of 0.1 fibers per cubic centimeter of air (f/cc) inside the attic or 0.01 f/cc in the staging chamber outside the attic. Work stoppage may also be required if other health and safety related issues are identified that could lead to injury of workers.
- At the onset of the field season, Tetra Tech will collect background air samples to establish baseline airborne fiber levels in the staging rooms prior to beginning the work. Samples will be collected for transmission electron microscopy (TEM) analysis and will be archived during the project. Background sample analysis by TEM will only be required if perimeter stationary air sample results exceed the specified requirements of 0.01 f/cc by the PCM analytical method.
- Tetra Tech will collect perimeter stationary air samples using a PCM method during the initial aggressive attic inspection procedures to document airborne fiber levels outside the staging chambers and in the staging rooms. The results will be provided the same day, or at a minimum of 24-hour turn-around time. The sensitivity of the PCM method is limited, making asbestos fibers difficult to distinguish from non-asbestos fibers. NIOSH Method 7400 will be used to analyze air samples by PCM. This method does not accurately distinguish between asbestos and non-asbestos fibers and simply considers any fiber with a length-to-width ratio of 3:1 to be an asbestos fiber. Therefore, the PCM method will be used to document the general cleanliness of aggressive attic access procedures.
- Air samples will be collected to monitor airborne asbestos fibers levels in a work area. Samples will be collected using personal monitoring pumps or larger-volume floor pumps. The samples will be used to establish the respiratory protection requirements for workers. Air samples will also be collected after a response action or abatement project to evaluate whether the work area has been adequately cleaned.

2.9 GENERAL SAFETY HAZARDS

- Assess each building for general safety hazards before entering attics and avoid attic entry if safety is in jeopardy.
- Insects and nests: Have the property owners use wasp/hornet spray to kill insects and nests prior to accessing attic areas. A waiting period may be required after application to ensure that nests have been abandoned. Cover all skin surfaces with PPE. Avoid access if safety is in jeopardy.
- Photo document the existing condition of the attic access hatch and ceilings prior to the attic entry. Note any existing cracks or other damage to the ceilings and discuss the presence with the property owner prior to the attic entry.
- Rodents/animals: Cover all skin surfaces with PPE. Avoid disturbing droppings and wear respirators at all times. Avoid access if safety is in jeopardy.

- Broken joist or unstable ceilings: Assess ceilings prior to and during attic access and avoid access if safety is in jeopardy.
- Electrical wiring: Avoid all electrical wiring and assume that all wiring is potentially hazardous. Avoid access if safety is in jeopardy.
- Exposed nails: Beware of nails at all times. Wear head, eye, hand and knee protection at all times.
- Unstable objects or debris: Avoid disturbing objects or debris stored in the attic. Do not relocate items stored inside the attics when moving about.
- High temperature hazards: High temperatures will present safety hazards during the summer season. Aggressive attic access procedures should be scheduled during morning hours whenever possible. Attic inspections should be limited to no more than 10 minutes. Operate an air filtration unit for at least 10 minutes prior to accessing excessively hot attics to provide cooling and fresh air intake. Safe Work Practices 6-15 and 6-16 discuss heat and cold stress and include monitoring methods appropriate for the season and location of work (see Appendix B of the TAPE HASP).

2.10 PROPERTY DAMAGE AND OTHER EMERGENCIES

- Ceiling Damage: Assess the condition of all ceilings before beginning the inspection procedures and avoid access if safety is in jeopardy. The second technician remaining below the ceiling should inspect the ceilings throughout the inspection process to ensure that damage does not occur. Notify the owner in all cases if ceiling damage occurs during the inspection procedure. If a ceiling breach occurs, discontinue the inspection procedure immediately and get off of the ceiling. If non-VCI debris falls into the living spaces, seal the ceiling breach and clean up the debris immediately. The owners will be informed that the TAPE DEQ representative will be contacting them and full damage repairs will be made. If VCI debris falls into the living space, notify the TAPE field manager immediately. Request that the property owner vacate the affected room(s) during the cleanup procedure. Isolate the effected/contaminated room with polyethylene critical barriers on doors and HVAC ducts. Seal the ceiling breach, wet wipe, and HEPA vacuum all contaminated debris. The TAPE field manager will supervise cleaning of the effected room(s) and will collect air clearance samples prior to recommending that the property owner re-occupy the area. A detailed incident report, including photographic documentation, should be compiled throughout the process. The owner will be informed that the TAPE DEQ representative will be contacting them to discuss the extent of repairs to be made.
- Health & Safety Emergency: If an injury occurs to an inspection team member during a procedure, discontinue the inspection immediately and leave the attic. If the injury is serious and emergency medical assistance is required, call 911 immediately and then notify the TAPE field office and TAPE field manager.

2.11 FACILITIES

- Electrical power will be supplied by the property owner.

- Water will be brought on-site by Tetra Tech. Water for decontamination procedures will be containerized in Hudson sprayers and/or sealable 5-gallon buckets.

2.12 TRANSPORTATION AND DISPOSAL

- Tetra Tech will transport and dispose of all contaminated materials in accordance with all applicable federal, state, and local regulations.

2.13 PREPARATION

- Two inspection team members are required for this procedure.
- Post warning signs at entrances to the staging chamber.
- Install staging chamber and ladder at base of the interior attic access hatch.
- Install a high efficiency particulate air (HEPA) filtered 600 cfm air filtration unit at an attic vent furthest from the access hatch point of entry.
- Cover as large an area as possible (up to 6 feet by 6 feet floor area) under the work area with 6-mil polyethylene sheeting.
- Prepare an asbestos disposal bag and bag any waste that is generated.
- Don 2 layers of Tyvek or polypropylene overalls, gloves, respirator, and other PPE.
- Use high-powered flashlights during all attic inspection procedures.

2.14 EXECUTION

- Set up ladder inside the staging chamber. Seal a polyethylene sheeting flap from the top of the staging chamber to outside of the access hatch using painter's tape or duct tape. Care should be taken to apply tape to surfaces without damaging paint or ceiling texture.
- Remove access hatch carefully by placing it atop an adjacent ceiling area. HEPA vacuum any gross debris on top of the access hatch.
- The 600 cfm air filtration unit will be activated once the access hatch is opened and remain running until decontamination procedures are completed.
- If VCI is observed at any point during the attic inspection, the inspection team member will promptly complete the visual inspection of the attic from that point and leave the attic.
- Wet any big pieces of VCI debris that falls from the attic with a Hudson sprayer and place it in the asbestos disposal bag.
- Install adequate lighting to ensure safe access in the attic.
- Perform the inspection work in the attic being careful to remain on top of the ceiling joists at all times. The inspectors should rotate 2 or 3 (10-inch by 3-foot) planks on top of the ceiling joists to support their weight when moving within the attic. While kneeling on the planks wearing knee pads, the inspectors should support their weight evenly centered between the ceiling joists at all times.

2.15 CLEAN-UP

- The following clean-up practices will be employed for all attic inspections regardless of presence of VCI.
- Wet wipe the ladder and tools that were used to perform the inspection work.
- While inside the staging chamber, HEPA vacuum coveralls after descending the ladder. Remove coveralls and place in an asbestos disposal bag.
- Wet wipe the interior of the staging chamber before disassembly or removal from the site.
- Wet wipe hands, face and exposed PPE with wet towels prior to removing respirator.
- Remove respirator and wipe the respirator with a wet rag. Place the respirator into a bag. Later, clean the respirator according to the procedure outlined in the respiratory protection section of the TAPE HASP.
- Mist, roll, and place the polyethylene sheeting in the asbestos disposal bag.
- Detach and de-activate the 600 cfm air filtration unit from the attic vent. Seal the contaminated inlet side of the unit with polyethylene sheeting and duct tape before transporting it.
- Double bag all waste.
- Dispose of all LA asbestos contaminated waste in accordance with all applicable Federal, State, and Local regulations.

3.0 PROCEDURAL SEQUENCING

The following provides a recommended progression of the work at the site:

1) Use exterior access hatches first when they are available.

2) Photo document the existing condition of access hatches and ceilings throughout the inspection area. Note any existing cracks or other damage to the ceilings and discuss their presence with the property owner prior to the attic entry.

3) Shut down all heating and air conditioning units and keep them "off" throughout inspection activities. Seal the air supply and return ductwork serving the staging room with airtight and watertight critical barriers.

4) Install the staging chamber below the interior access hatch. If the access hatch is located inside a closet or another location where personal items must be moved prior to installing the chamber, request permission from the owner to move and/or cover items with polyethylene sheeting before starting set up. Take photos of existing conditions prior to disturbing any personal items and be sure to replace all items appropriately upon completion of the inspection procedures.

5) Conduct personnel exposure assessments air sampling to document a negative exposure assessment at the beginning of the field season attic inspections and periodically during the season. Personnel air monitoring will be collected during multiple inspections the first day to determine an 8-hour time weighted average (TWA) as well as a 30-minute short term exposure limit (STEL). Personnel air monitoring samples will be analyzed on a 24-hour turnaround basis. Initial negative exposure assessments will be conducted to document at least one attic with VCI.

6) Conduct stationary air sampling in areas adjacent to the staging chamber during the initial inspections of the field season and periodically during the season. Stationary air monitoring samples will be analyzed on a 24-hour turnaround basis. Initial stationary air monitoring will be conducted to document at least one attic inspection with VCI.

7) Install and activate a HEPA filtered 600 cfm air filtration unit at an attic vent furthest from the access hatch point of entry. The unit will be activated once the access hatch is opened and remain running until decontamination procedures are completed. Operate the air filtration unit for at least 10 minutes prior to accessing excessively hot attics to provide cooling and fresh air intake.

8) Conduct a preparation inspection of the work area to ensure containment integrity prior to starting the aggressive attic inspection.

9) Containerize debris routinely during aggressive attic inspection activities. Exercise caution to avoid tracking contamination from the attic to the "clean" staging chamber or staging room.

10) Once the inspection is finished, complete decontamination as outlined in Section 2.15.

Record of Modification
to the
Troy Sampling and Quality Assurance Project Plan
Field Activities
TFO – 0 0 0 1 4 (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 Asbestos Property Evaluation Work Plan
- ○ Other (Title and approval date):_____

Site-Specific Guidance/SOP (Number and Revision No.) (check one):

- ○ CDM-LIBBY-10, Current Revision (30-point dust sample collection)
- ○ 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: Montana DEQ Date: June 17, 2009

Description of Modification (attach additional sheets if necessary; state section and page numbers of each document that are affected by the proposed modification): Include public roads and alley inspections to the standard TAPE protocol. See attached Protocol Modification for TAPE Inspection and Sampling of Roads and Alleys for Visible Vermiculite.

Field logbook and page number where Modification is documented (or attach associated correspondence):
_____

Potential Implications of Modification: Additional data for the Remedial Investigation to further define the nature and extent of contamination.

Duration of Modification (check one):

○ Temporary
Date(s):_____ AD-_____
BD(s)-_____ TT(s)-_____

● Permanent (Proposed Text Modification Section) Effective Date: July 3, 2009

Proposed Text Modifications in Associated Document (attach additional sheets if necessary):_____
_____
_____

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: 6-30-09
(DEQ Project Manager or designate)

EPA Review and Approval: [signature] Date: 6-30-09
(*USEPA RPM or designate*)
Victor Ketellapper

Revised April 29, 2007

PROTOCOL MODIFICATION

TO THE

TROY ASBESTOS PROPERTY EVALUATION (TAPE) PROJECT WORK PLAN

INSPECTION AND SAMPLING OF ROADS AND ALLEYS FOR VISIBLE VERMICULITE

Troy Operable Unit Number 7
of the Libby Asbestos Superfund Site

Prepared for:

MONTANA DEPARTMENT OF ENVIRONMENTAL QUALITY
Remediation Division
P.O. Box 200901
Helena, Montana 59620

Contract Number 402026
Task Order Number 46

Prepared by:

TETRA TECH EM INC.
Power Block Building, Suite 612
7 West 6th Avenue
Helena, Montana 59601
(406) 442-5588

July 7, 2009

1.0 SCOPE OF WORK

This modification to the Troy Asbestos Property Evaluation (TAPE) work plan (WP) includes all procedures necessary to complete inspection and sampling of roads and alleys in the Troy operable unit (OU7) of the Libby Asbestos Superfund site. The methods and procedures described in this modification supplement the existing TAPE inspection procedures outlined in the TAPE WP (Tetra Tech EM Inc. 2007). This modification does not require any changes to field quality control (QC) procedures, data management or project quality assurance and QC procedures identified in the TAPE WP.

The vermiculite ore body at the Libby mine (Libby vermiculite) contains naturally occurring forms of asbestos. The predominant types of asbestos minerals found at the Libby Asbestos Superfund site are known as amphibole asbestos. Asbestos is a recognized human carcinogen and is classified as a hazardous substance as defined by 40 Code of Federal Regulations (CFR) Section 302.4 of the National Contingency Plan. TAPE field inspections are conducted using unaided human visual observations to identify visible vermiculite in soils. Human visual observations cannot identify asbestos minerals, such as Libby Amphibole (LA). Therefore in addition to the visual inspection, soil samples will be collected and analyzed for LA.

The extent of LA contamination within OU7 is unknown. Based on historical information and information received for Libby and Troy, DEQ is not aware that road and alley surfaces were covered with vermiculite from the Libby mine, or that this Libby vermiculite product was mixed with other sand or gravel materials and placed on road and alley surfaces within OU7. The main source areas, the mine and processing facilities, are located in Libby. Libby vermiculite was used for home insulation and as a soil amendment in yards, gardens, and other soil areas. Libby vermiculite from historical use as home insulation, or from erosion and transport of vermiculite in yard and garden soils may have migrated to roads and alleys that are in close proximity. In addition, the transporting of Libby vermiculite to Troy for yards, gardens, and other soil areas, and for home insulation purposes may have accidentally contaminated road and alley surfaces. Property inspections performed to date in OU7 included most driveways and some road edges. This modification provides the scope of work to systematically inspect and sample the roads and alleys during the 2009 field season.

This TAPE WP modification addresses the data quality objectives (DQOs), a classification of roads and alleys in OU7 for inspection and sampling, and the methods and procedures for collecting soil samples.

2.0 TROY DATA QUALITY OBJECTIVES

This section presents the DQOs for the TAPE inspection and sampling of roads and alleys. The DQOs are qualitative and quantitative statements developed through the seven-step DQO process (EPA 2000a; 2000b). The DQOs help to clarify the study objectives, define the most appropriate data to collect, and describe the conditions under which to collect the data. They specify tolerable limits on decision errors that will be used as the basis for establishing the quantity and quality of data needed to support decision-making. The DQOs are used to develop a scientific and resource-effective design for data collection.

Step 1- State the Problem

Previous investigations have shown that vermiculite ore from the Libby mine was used as a soil amendment in yards, gardens, flower beds, and other soil areas in Libby and Troy. Libby vermiculite was also used for home insulation. The vermiculite ore at the Libby mine contains naturally occurring forms of asbestos, including LA, which have been recognized as human carcinogens. As a result, residents of Libby and Troy may be exposed to LA from contact with Libby vermiculite.

Libby vermiculite from historical use as home insulation, or from erosion and transport of vermiculite in yard and garden soils may have migrated to roads and alleys. In addition, the transporting of Libby vermiculite to Troy may have resulted in accidental spills that may have contaminated road and alley surfaces. Because most roads and alleys are on public land with few, if any, access restrictions, there may be exposure to LA from contact with road and alley soils. These exposures may present an unacceptable risk of adverse health effects under certain exposure conditions.

The magnitude of the potential exposure is unknown. Additional data are needed to support decisions about short term and long term response actions for roads and alleys in OU7.

Step 2 - Identify the Decision

The decisions EPA and DEQ seek to make are:

1) Does visible vermiculite found on road and alley surfaces in OU7 need to be removed?

2) Do specific roads and alleys in OU7 contain levels of LA in soil that need to be addressed by short term response actions?

Step 3 – Identify Inputs to the Decision

Each decision identified in Step 2 requires specific inputs:

Decision: Does visible vermiculite found on road and alley surfaces in OU7 need to be removed?

Input: Reliable visual observations for visible vermiculite on road and alley surfaces obtained through systematic inspections of all roads and alleys across OU7.

Decision: Do soils in specific roads and alleys in OU7 contain levels of LA that need to be addressed by short term response actions?

Inputs: Measurements of the average concentration of LA in soil in specified road and alley classes in OU7:

1. Paved roads with no curbs and gutters,
2. Gravel roads,
3. Alleys,
4. Dirt roads, and
5. Other roads (driveways and undeveloped right-of-ways).

Step 4 - Define the Study Boundaries

Vermiculite and other LA-contaminated wastes were historically transported from the Libby mine and randomly used for amending soils in yards, gardens, and other soil areas in OU7. The use of Libby vermiculite for insulating homes and other buildings in OU7 is also expected to be random. The potential locations where Libby vermiculite amended soils have eroded and migrated onto Troy roads and alleys are unknown. Therefore, DEQ has determined that all roads and alleys in OU7 will be inspected and sampled under this TAPE WP modification. U.S. Highway 2 and State Highway 56 are not part of OU7 and will be evaluated under different Libby Asbestos Superfund site operable units.

Operable Unit 7 covers approximately 3,560 acres. The Troy city limits lie entirely within OU7 and encompasses approximately 540 acres. The road and alley distances, by the six classifications, are in Table 2-1. The percent for each road class in and out of the Troy city limits is also in Table 2-1. Soil sampling densities will vary in order to characterize the different human risk exposure scenarios for in-town and out-of-town roads and alleys. One 30-aliquot composite soil sample will be collected from approximately every 500 feet of road or alley (one city block) within the Troy city limits. One 30-aliquot composite soil sample will be collected from every 1,320 feet (1/4 mile) length of road outside the Troy

city limits. All 30-aliquot composite surface soil samples will be collected from the zero- to six-inch depth at regular spacing intervals along and across the roads and alleys.

TABLE 2-1
TROY OU7 ROAD AND ALLEY DISTANCES AND PERCENTAGES

Class No.	Description	Total Distance (linear ft)	In Troy City Limits		Out of Troy City Limits	
			Percent	Distance (linear ft)	Percent	Distance (linear ft)
1	Paved Roads (with curbs & gutters)	0 [a]	0	0	0	0
2	Paved Roads (no curbs or gutters)	85,572	36	30,708	64	54,864
3	Gravel Roads	65,450	21	13,604	79	51,846
4	Alleys	11,876	100	11,876	0	0
5	Dirt Roads	2,525	0	0	100	2,525
6	Driveways/undeveloped rights-of-way	5,215	0	0	100	5,215
Totals		**170,638**		**56,188**		**114,450**

Notes:
[a] Only U.S. Hwy 2 qualifies as Class No. 1 and it is excluded from this sampling effort.

All data required to support project objectives will be collected at each road or alley parcel. Completion of the required inspection and surface soil sampling for all OU7 roads and alleys is expected to be completed during the 2009 TAPE field season.

Step 5 - Develop Decision Rules

Decision: Does visible vermiculite need to be removed from specific road and alley parcels in OU7?

Decision Rule: If uncontained, migrating, visible vermiculite is observed in roads or alleys, the parcel may require a response action that may include removing the visible vermiculite.

Decision: Do specific road and alley parcels in OU7 contain levels of LA in soil that need to be addressed by short term response actions?

Decision Rule: If visible vermiculite is observed within a road and alley parcel and the LA concentration in the composite soil sample exceeds the action level for cleanup, the road or alley will be cleaned up in a short term response action.

Step 6 - Specify Tolerable Limits on Decision Errors

Two types of decision errors are possible. A Type I (false negative) decision error would occur if a decision is made that a response action is not necessary when in fact, a response action is necessary. A Type II (false positive) decision error would occur if a decision is made to undertake a response action when in fact, it is not necessary.

For the TAPE project, EPA and DEQ are most concerned about establishing limits on the occurrence of Type I errors. EPA recommends the following limit on Type I decision errors:

- No more than a 10% chance of not taking a response action when one is required

For soil, if the measured average concentration of LA in the composited soil sample from a given road or alley parcel is less than the action level for soil, there should be no more than a 10% chance that the true average exceeds the action level.

Because a composite sampling approach has been chosen for soil, information on individual soil sample variability within each road or alley parcel will not be available. This is not thought to be a serious concern since the analytical method for detecting LA in soil is semi-quantitative. Analytical results for LA in soil are assigned to one of four concentration bins ranging from non-detect (Bin A), trace – less than 0.2%, but LA detected (Bin B1), between 0.2% and 1% (Bin B2), and greater than 1% (Bin C). As part of its investigation of the Libby Asbestos Superfund site, EPA performed a study on the accuracy of the analytical method for LA in soil; polarized light microscopy (PLM) - visual estimation (VE). Results indicated that 77% of reference soil samples were accurately assigned to the appropriate bin, and, of the reference samples not accurately reported, 22 out of 23 (96%) were reported in a concentration bin higher than the reference concentration. These results indicate that the analytical method is more likely to overestimate than underestimate the true concentration of LA in soil.

Step 7 – Optimize the Design for Obtaining Data

For soil, a composite sampling design has been selected to measure the average concentration within road and alley parcels. Composite sampling involves physically combining and homogenizing the 30 soil aliquots to form a single sample. If the concentrations of a contaminant could be measured accurately in the individual aliquots as well as in their composite, and if the compositing process is carried out properly, then the concentration measured in the composite sample is expected to be equal to the average of the concentrations measured in the individual aliquots (assuming no measurement errors).

Estimating the Number of Samples Required

For soil, one composite sample consisting of 30 aliquots will be collected from each road and alley parcel. This sampling protocol equates to an estimate of approximately 200 samples collected across all road and alley parcels within OU7. The sampling depth will be 0- to-6 inches since this is the depth most likely to be routinely disturbed by activities within this parcel (vehicle traffic, road grading).

The sampling design of composite samples for soil was selected because this design results in approximately the same precision of an estimated average concentration over the sampling area as that provided by the same number of individual samples.

Estimate the Required Analytical Sensitivity

Although EPA has made no final decisions about the level of LA in soil that requires a short term response action specifically in OU7, current EPA decisions at the Libby Asbestos Superfund site are based on LA action levels of 1% or greater by weight for soil.

In the absence of an EPA decision about the specific action levels for LA in soil in OU7, for planning purposes, the analytical sensitivities must be low enough to detect LA at levels below the action levels for other areas of the site. The required analytical sensitivities and analytical methods for the TAPE program are:

Soil: Practical Quantitation Limit = 0.2 weight percent LA asbestos
Sampling Method: Guidance CDM-Libby-05
Preparation Method: SOP EPA SRC-Libby-01
Analytical Method: SOP SRC-Libby-03 (Revision 2)

Equipment Blanks:

Analytical Sensitivity: Read 10 grid openings

Analytical Method: (AHERA) modified counting rules (L≥0.5 µm, AR≥3:1) and preparation techniques per Libby laboratory modification forms for investigative samples (Do not use water testing protocols).

3.0 ROAD AND ALLEY INSPECTIONS AND SOIL SAMPLING

The following text provides a summary of the methods employed for the identification and classification of road and alley parcels, collection of data through interviews, soil sampling methods and procedures, and data collection and documentation requirements.

3.1 PARCEL IDENTIFICATION AND CLASSIFICATION

Prior to initiation of inspection and soil sampling, the road and alley parcels will be identified and assigned unique AD numbers. Each parcel will be 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 the city limits). A summary of road and alley classifications is in Table 3-1. Soil sampling strategies for each parcel are based on the classification. Table 3-1 also has a summary of sampling methods and densities for each road or alley classification. Road and alley parcels (with AD numbers) will be presented on a large-scale drawing (separate from this work plan) available at the Troy Information Center. Additionally, individual parcel maps will be prepared for each road and alley and included in the AD file for use by field staff during the inspections and sampling.

3.2 VERBAL INTERVIEWS

The Troy and County road department personnel, City clerk, and Mayor will be interviewed to discuss concerns and obtain historical information about the alleys. Additional knowledgeable persons may also be identified and interviewed as necessary. The interviews will be used to gather any information regarding the known use of vermiculite in road and alley parcels. This information will be recorded in the field logbook. All data collected through the verbal interviews will be included in the AD file for use by field staff during the parcel inspections and sampling.

3.3 VISUAL INSPECTION

Tetra Tech field personnel will inspect for visible vermiculite in all road and alley parcels identified within OU7. Field notes will be recorded for the presence or absence of visible vermiculite either on the surface or in the soil aliquots, regardless of the road or alley surface material (asphalt, concrete, gravel, dirt). All notes regarding specific road and alley parcels will reference the parcel AD number.

3.4 SOIL SAMPLING

After completing the surface visual inspection of each road or alley, the TAPE field team will collect soil samples following the procedures described the TAPE WP. Soil sampling for each road and alley parcel will include the following steps:

- Identify sample aliquot locations
- Collect sample aliquots and assess the sample aliquots for visible vermiculite
- Record locations on field sketch
- Record parcel location using a global positioning system (GPS) receiver

One soil sample will be collected from each road and alley parcel not fully covered by asphalt or concrete to determine potential LA concentrations. Samples will consist of 30-point composites, with the subsample aliquots collected at evenly spaced intervals that will alternate from left, to center, to right on dirt/gravel surfaced roads or alleys (Figure 1). Where road surfaces are paved but road edges are not, a 30-point composite sample will be collected from evenly spaced locations along the adjacent road edge/borrow ditch. If road edges/borrow ditches are not of equal proportion on both sides of the parcel, composite sample aliquots should be collected at a rate consistent with the percentage of non-paved surfaces on each side of the road or alley parcel (Figure 2). The TAPE field team will collect all soil sample aliquots with the minimum amount of disturbance to the surface. Sampling locations in roads and alleys will be carefully restored by filling all sample locations with surrounding soil/gravel. To ensure consistency, all TAPE field teams will have the same training and guidelines. Training will include “brainstorming” potential parcel scenarios and discussing proposed sampling approaches.

Composite sampling will be implemented to provide representative sampling results for the entire road or alley parcel. Table 3-1 has a summary of sampling methods and densities for the six classifications of roads and alleys. Road and alley classifications are assigned by the Database Manager prior to the field team inspecting and sampling.

TABLE 3-1

TROY OU 7 ROAD AND ALLEY CLASSIFICATION

Class #	Class Name	Brief Description	Sampling Method	Sampling Density	Notes
1	Paved Roads (with curbs & gutters)	Paved or concrete driving lane surface with finished curb & gutters.	Not sampled	Not sampled. Visual inspection for visible vermiculite only.	No soil exposed.
2	Paved Roads (no curbs & gutters)	Paved or concrete driving lane; unfinished edges in right-of-way.	30-aliquot composite surface soil (0-to 6-inches) samples collected at regular spacing along both sides of paved areas.	In City – 1 sample per city block or every 500 feet. Outside City – 1 sample for every 1,320 linear feet.	Unbiased sampling. City blocks assumed to be less than 500 feet long.
3	Gravel Roads	Gravel or road mix driving lane (not paved or not concrete) with finished or unfinished edges in right-of-way.	30-aliquot composite surface soil (0-to 6-inches) samples collected at regular spacing from the middle and each side of the road.	In City – 1 sample per city block or every 500 feet. Outside City – 1 sample for every 1,320 linear feet.	Unbiased sampling. City blocks assumed to be less than 500 feet long.
4	Alleys	Alleys that are not paved or surfaced with concrete.	30-aliquot composite surface soil (0-to 6-inches) samples collected at regular spacing from the middle and each side of the alley.	In City – 1 sample per city block or every 500 feet.	Unbiased sampling. City blocks assumed to be less than 500 feet long. No alleys outside of the City.
5	Dirt Roads	Dirt roads (may be similar to alleys) that are not paved or surfaced with concrete.	30-aliquot composite surface soil (0-to 6-inches) samples collected at regular spacing from the middle and each side of the road.	Outside City – 1 sample every 500 to 1,320 feet.	Unbiased sampling. Rural residential dirt roads may be very similar to alleys and will be sampled every 500 feet. Dirt roads in undeveloped areas will be sampled every 1,320 feet.
6	Unplatted or Undeveloped	Unique characteristics not applicable to Classes 1 through 5.	To be determined in field. Any samples collected will be 30-aliquot composite surface soil from 0 to 6 inches deep.	To be determined in field.	Will classify road prior to sampling.

TETRA TECH, INC.

Legend

- PROPOSED ALIQUOTS FOR TYPICAL ROAD ROW SAMPLE (30 ALIQUOTS = 1 SAMPLE)
- ROAD AND ALLEY ROW PARCELS

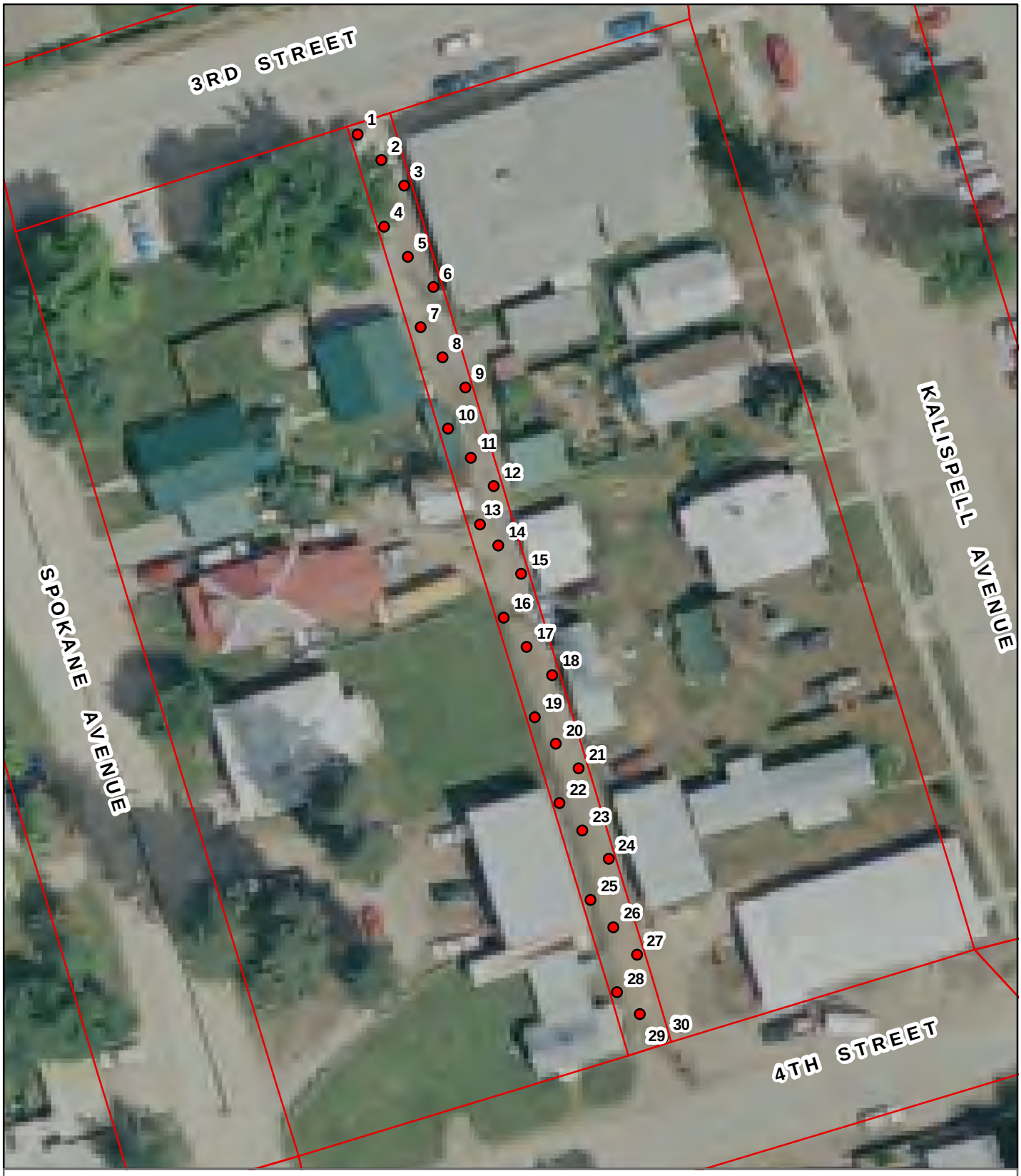
0 40 Feet

N

TROY OPERABLE UNIT
LIBBY ASBESTOS SUPERFUND SITE

FIGURE 1
PROPOSED SAMPLING SCHEME FOR ROADS WITH NO CURBS OR GUTTERS

GIS map by Ed Madej TTEMI-HE
FIG1_TROY_TYPICAL_ROAD_061509.mxd



TETRA TECH, INC.

Legend

- PROPOSED ALIQUOTS FOR TYPICAL ALLEY ROW SAMPLE (30 ALIQUOTS = 1 SAMPLE)
- ROAD AND ALLEY ROW PARCELS

0 40 Feet

N

TROY OPERABLE UNIT
LIBBY ASBESTOS SUPERFUND SITE

FIGURE 2
PROPOSED SAMPLING SCHEME FOR ALLEYS

GIS map by Ed Madej TTEMI-HE
FIG2_TROY_TYPICAL_ALLEY_061509.mxd

3.4.1 Collect Soil Samples

Soil samples will be collected from road and alley parcels in OU7 following the procedures in the TAPE WP. Stainless steel scoops will be used to collect approximately 20 grams of soil from the 0- to 6-inch depth interval at each aliquot location for a total of approximately 600 grams of soil. Due to the presence of compacted materials on the surfaces of roads and alleys, a metal "chisel" may be employed to loosen the soil prior to sampling. If the metal chisel is used, it will be thoroughly cleaned and decontaminated after each sample using procedures outlined in the TAPE WP. Each aliquot will be examined for the presence of visible vermiculite. The amount of vermiculite will be categorized as none, low, intermediate, or high using the procedures defined in the TAPE WP. Aliquots will then be placed into a stainless steel bowl and mixed. After the sample has been homogenized, approximately 600 grams of soil will be placed in one re-closable plastic bag and mixed. During sample collection and mixing, the field team will attempt to shield the soil samples from the wind to avoid potentially losing lighter fractions of the soil to the ambient air. At the conclusion of sampling the stainless steel scoop and bowl will be thoroughly cleaned and decontaminated using procedures outlined in the TAPE WP.

The initial re-closable plastic bag will be placed inside a second bag as a precaution. A pre-printed sample label will be affixed to the outside of the inner re-closable bag, and the sample ID number written on the outside of the inner bag. The outer re-closable plastic bag will be labeled and marked similarly using the pre-printed sample ID numbers. Soil samples will be labeled with a unique sample identification number "TT-XXXXX" where "TT" indicates a "Troy TAPE" sample. Samples will remain under chain-of-custody procedures as described in Section 5.5 of the TAPE WP.

The TAPE field team will attempt to restore the road or alley surface to its prior condition after sampling. For most sample locations, the small excavation can be filled with soil from the immediately surrounding area, and lightly tamped down. Since the required samples are small, sampling is not expected to cause noticeable disturbance to a road or alley.

3.4.2 Record Sample Location on Field Sketch and with GPS

A Trimble Geo XT GPS will be used to record the midpoint latitude and longitude for each composite soil sample. The GPS location coordinates will be recorded on the Trimble unit and will be associated with the unique AD identification number for the road or alley parcel. A field sketch of each road and

alley parcel will also be prepared and will show the approximate locations of the aliquots of the composite sample.

3.4.3 Photography

Each TAPE field team will have a camera for photo-documenting the conditions at the road or alley parcel if the conditions are not readily describable in writing or, if the field team decides that photographs may assist in development of a remedial action plan for that parcel. Permission to photograph the roads and alleys will be obtained from the City of Troy and Lincoln County.

All photographs will be recorded in the field logbook. All photographs will be taken using digital cameras, will be downloaded the same day at the Tetra Tech Troy field office, and will be saved into the TAPE project Scribe database. The photographs will become part of the electronic record for each road and alley parcel.

4.0 REFERENCES

Tetra Tech EM Inc. 2007. Troy Asbestos Property Evaluation Work Plan (Field Sampling Plan and Quality Assurance Project Plan). Prepared for: Montana Department of Environmental Quality, Remediation Division. July 9.

U.S. Environmental Protection Agency (EPA). 2000a. "Data Quality Objectives Process for Hazardous Waste Site Investigations (EPA QA/ G-4HW)." Office of Environmental Information. Washington, D.C. EPA/600/R-00/007. January.

EPA. 2000b. "Guidance for the Data Quality Objectives Process, EPA QA/G-4." Office of Environmental Information. Washington, DC. EPA/600/R-96/055. August.

APPENDIX C

OUTDOOR AMBIENT AIR TROY FIELD OFFICE (TFO) 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 2, 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: Catherine LeCours Date: March 3, 2010
(DEQ Project Manager or designate)

EPA Review and Approval: N/A Date:_____
(USEPA RPM or designate)

REMEDIAL INVESTIGATION WORK PLAN
OUTDOOR AMBIENT AIR STUDY

Operable Unit Number 7
of the Libby Asbestos Superfund Site

Tetra Tech, Update March 2010

DOCUMENT REVISION LOG

Revision	Date	Primary Changes
TFO-00001	March 3, 2010	Changed filter size from 0.45 µm to 0.80 µm
TFO-00002	March 15, 2010	Flow rate change

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.

APPENDIX D

SUMMARY OF TAPE INVESTIGATION RESULTS

Description	Parcel	Primary Building	Secondary Building	Use Area	Dust Samples	Dust Sample (Primary Building)
Total Inspections	1,239					
Total Parcels	1,578					
Inspections outside of OU7	28					
Parcels outside of OU7	31					
Road and Alley Inspections	302					
Inspected parcel ERS actions	30					
2007 AA Mailing - geographic count	1,092					
2007 AA Mailing - owner sent AA	1,078					
2007 AA Mailing - access granted	586					
2007 AA Mailing - limited access	8					
2007 AA Mailing - denied access	12					
2007 TAPE Inspection	536	508	730	1,987	1,211	678
2007 TAPE Inspection - No attics		111	442			
2007 TAPE Inspection - Inaccessible attics		25	16			
2007 TAPE Inspection - VCI in attic	56					
2007 TAPE Inspection - VV in indoor living space	28					
2007 TAPE Inspection - Common Use Areas	502					
2007 TAPE Inspection - Limited Use Areas	145					
2007 TAPE Inspection - Specific Use Areas	1,146					
2007 TAPE Inspection - Non-Use Areas	194					
2007 TAPE Inspection - Interior Soil						
2007 TAPE Inspection - ERS Action	10					
2008 AA Mailing - owner sent AA	538					
2008 AA Mailing - access granted	391					
2008 AA Mailing - limited access	8					
2008 AA Mailing - denied access	24					
2008 TAPE Inspection	261	226	349	1,098	573	313
2008 TAPE Inspection - No attics		82	307			
2008 TAPE Inspection - Inaccessible attics		13	10			
2008 TAPE Inspection - VCI in attic	21	21				
2008 TAPE Inspection - VV in indoor living space	20	20				
2008 TAPE Inspection - Common Use Areas	257					
2008 TAPE Inspection - Limited Use Areas	83					
2008 TAPE Inspection - Specific Use Areas	655					
2008 TAPE Inspection - Non-Use Areas	103					
2008 TAPE Inspection - Interior Soil						
2008 VV Reinspection	101			527		
2008 VV Reinspection - Common Use Areas				132		
2008 VV Reinspection - Limited Use Areas				41		
2008 VV Reinspection - Specific Use Areas				353		
2008 VV Reinspection - Non-Use Areas				1		
2008 TAPE Inspection - ERS Action	12					
2009 AA Mailing - owner sent AA	313					
2009 AA Mailing - access granted	180					
2009 AA Mailing - limited access	12					
2009 AA Mailing - denied access	18					
2009 TOAD Parcel Status - access granted	941					
2009 TOAD Parcel Status - limited access	15					
2009 TOAD Parcel Status - denied access	34					
2009 TAPE Inspection	436	120	216	648		
2009 TAPE Inspection - No attics		40	178			
2009 TAPE Inspection - Inaccessible attics		12	13			
2009 TAPE Inspection - VCI in attic		5				
2009 TAPE Inspection - VV in indoor living space		5				
2009 TAPE Attic Revisit	30					
2009 TAPE Inspection - Common Use Areas	125					
2009 TAPE Inspection - Limited Use Areas	153					
2009 TAPE Inspection - Specific Use Areas	323					
2009 TAPE Inspection - Non-Use Areas	47					
2009 TAPE Inspection - Interior Soil						
2009 TAPE Inspection - Road and Alley	302					
2009 TAPE Inspection - ERS Action	8					

Description	Dust Sample (Secondary Building)	Dust Field Duplicate	Dust Field Duplicate (Primary Building)	Dust Field Duplicate (Secondary Building)	Dust Field Blank
Total Inspections					
Total Parcels					
Inspections outside of OU7					
Parcels outside of OU7					
Road and Alley Inspections					
Inspected parcel ERS actions					
2007 AA Mailing - geographic count					
2007 AA Mailing - owner sent AA					
2007 AA Mailing - access granted					
2007 AA Mailing - limited access					
2007 AA Mailing - denied access					
2007 TAPE Inspection	533	42	24	18	195
2007 TAPE Inspection - No attics					
2007 TAPE Inspection - Inaccessible attics					
2007 TAPE Inspection - VCI in attic					
2007 TAPE Inspection - VV in indoor living space					
2007 TAPE Inspection - Common Use Areas					
2007 TAPE Inspection - Limited Use Areas					
2007 TAPE Inspection - Specific Use Areas					
2007 TAPE Inspection - Non-Use Areas					
2007 TAPE Inspection - Interior Soil					
2007 TAPE Inspection - ERS Action					
2008 AA Mailing - owner sent AA					
2008 AA Mailing - access granted					
2008 AA Mailing - limited access					
2008 AA Mailing - denied access					
2008 TAPE Inspection	260	21	13	8	97
2008 TAPE Inspection - No attics					
2008 TAPE Inspection - Inaccessible attics					
2008 TAPE Inspection - VCI in attic					
2008 TAPE Inspection - VV in indoor living space					
2008 TAPE Inspection - Common Use Areas					
2008 TAPE Inspection - Limited Use Areas					
2008 TAPE Inspection - Specific Use Areas					
2008 TAPE Inspection - Non-Use Areas					
2008 TAPE Inspection - Interior Soil					
2008 VV Reinspection					
2008 VV Reinspection - Common Use Areas					
2008 VV Reinspection - Limited Use Areas					
2008 VV Reinspection - Specific Use Areas					
2008 VV Reinspection - Non-Use Areas					
2008 TAPE Inspection - ERS Action					
2009 AA Mailing - owner sent AA					
2009 AA Mailing - access granted					
2009 AA Mailing - limited access					
2009 AA Mailing - denied access					
2009 TOAD Parcel Status - access granted					
2009 TOAD Parcel Status - limited access					
2009 TOAD Parcel Status - denied access					
2009 TAPE Inspection					
2009 TAPE Inspection - No attics					
2009 TAPE Inspection - Inaccessible attics					
2009 TAPE Inspection - VCI in attic					
2009 TAPE Inspection - VV in indoor living space					
2009 TAPE Attic Revisit					
2009 TAPE Inspection - Common Use Areas					
2009 TAPE Inspection - Limited Use Areas					
2009 TAPE Inspection - Specific Use Areas					
2009 TAPE Inspection - Non-Use Areas					
2009 TAPE Inspection - Interior Soil					
2009 TAPE Inspection - Road and Alley					
2009 TAPE Inspection - ERS Action					

Description	Dust Field Blank (Primary Building)	Dust Field Blank (Secondary Building)	Soil Samples	Soil Field Duplicate	Soil Split
Total Inspections					
Total Parcels					
Inspections outside of OU7					
Parcels outside of OU7					
Road and Alley Inspections					
Inspected parcel ERS actions					
2007 AA Mailing - geographic count					
2007 AA Mailing - owner sent AA					
2007 AA Mailing - access granted					
2007 AA Mailing - limited access					
2007 AA Mailing - denied access					
2007 TAPE Inspection	159	36	1,608	54	
2007 TAPE Inspection - No attics					
2007 TAPE Inspection - Inaccessible attics					
2007 TAPE Inspection - VCI in attic					
2007 TAPE Inspection - VV in indoor living space					
2007 TAPE Inspection - Common Use Areas					
2007 TAPE Inspection - Limited Use Areas					
2007 TAPE Inspection - Specific Use Areas					
2007 TAPE Inspection - Non-Use Areas					
2007 TAPE Inspection - Interior Soil			316	4	
2007 TAPE Inspection - ERS Action					
2008 AA Mailing - owner sent AA					
2008 AA Mailing - access granted					
2008 AA Mailing - limited access					
2008 AA Mailing - denied access					
2008 TAPE Inspection	82	15	1,000	44	152
2008 TAPE Inspection - No attics					
2008 TAPE Inspection - Inaccessible attics					
2008 TAPE Inspection - VCI in attic					
2008 TAPE Inspection - VV in indoor living space					
2008 TAPE Inspection - Common Use Areas					
2008 TAPE Inspection - Limited Use Areas					
2008 TAPE Inspection - Specific Use Areas					
2008 TAPE Inspection - Non-Use Areas					
2008 TAPE Inspection - Interior Soil			95	2	12
2008 VV Reinspection					
2008 VV Reinspection - Common Use Areas					
2008 VV Reinspection - Limited Use Areas					
2008 VV Reinspection - Specific Use Areas			131		
2008 VV Reinspection - Non-Use Areas					
2008 TAPE Inspection - ERS Action					
2009 AA Mailing - owner sent AA					
2009 AA Mailing - access granted					
2009 AA Mailing - limited access					
2009 AA Mailing - denied access					
2009 TOAD Parcel Status - access granted					
2009 TOAD Parcel Status - limited access					
2009 TOAD Parcel Status - denied access					
2009 TAPE Inspection			581	30	
2009 TAPE Inspection - No attics					
2009 TAPE Inspection - Inaccessible attics					
2009 TAPE Inspection - VCI in attic					
2009 TAPE Inspection - VV in indoor living space					
2009 TAPE Attic Revisit					
2009 TAPE Inspection - Common Use Areas					
2009 TAPE Inspection - Limited Use Areas					
2009 TAPE Inspection - Specific Use Areas					
2009 TAPE Inspection - Non-Use Areas					
2009 TAPE Inspection - Interior Soil			75	2	
2009 TAPE Inspection - Road and Alley			271	8	
2009 TAPE Inspection - ERS Action					