



EARTHRES
ENGINEERING FOR SUCCESS™

April 3, 2019

Michael J. Menghini, District Mining Manager
Department of Environmental Protection
Pottsville District Mining Office
5 West Laurel Boulevard
Pottsville, PA 17901-2454

**SUBJECT: Qualitative Geologic Survey Sampling Plan
Rock Hill Quarry
SMP No. 7974SM1
East Rockhill Township, Bucks County
EARTHRES Project No. 061003.051**

Dear Mr. Menghini:

On behalf of Hanson Aggregates Pennsylvania, LLC (Hanson), Earthres Group, Inc. is providing the following Qualitative Geologic Survey Sampling Plan for continued assessment of Naturally Occurring Asbestos (NOA) at the Rock Hill Quarry Operation.

The sampling plan was prepared in follow-up to your letter of December 19, 2018, and our joint meeting on February 21, 2019 with the Pennsylvania Department of Environmental Protection (PA DEP), Hanson, Kelly Bailey, CIH, Drew R. Van Orden, P.E., and R.E. Pierson personnel.

This sampling plan has been designed to collect sufficient information for the preparation of a detailed Qualitative Geologic Survey of the site so that, based upon the results, the PA DEP will be able to lift the stop work order and allow Hanson and R.E. Pierson Materials to resume active operation of the Quarry.

CURRENT AND PROPOSED SAMPLING

Prior investigation, qualitative geological surveying and sampling conducted at the site during 2018 included the collection and analysis of thirty-three (33) samples including: 5 mineral vein rock samples, 21 drill hole cutting samples, 5 aggregate storage pile samples and 2 crusher run (fines) samples. All samples yielded non-detectable results for NOA, except for one biased rock sample that found a trace amount of asbestiform actinolite in a mineral vein.

The following work plan elements were developed in consultation with Hanson’s asbestos experts: Kelly Bailey, CIH, of Kelly Bailey Consulting, LLC and Drew R. Van Orden, P.E. of RJ Lee Group, Inc. The proposed work efforts include:

- 1) Aggregate storage pile sampling;
- 2) Rock coring and sampling;
- 3) Surficial geological analysis of the existing boulder field on the southern and eastern sides of the Quarry pit; and,
- 4) Sampling of water from the NPDES discharge, Sediment Basin 1, Sediment Basin 2, and the Quarry pit.

A minimum of two (2) days’ notice will be given to the PA DEP to allow for coordination of each field sampling event.

AGGREGATE STORAGE PILE SAMPLING

Per the Department’s letter of December 19, 2018, aggregate storage pile sampling is to include “...one test per 1,000 tons of material or any fraction thereof...” Hanson mapped the existing aggregate storage piles and determined the following tonnages and proposed samples as indicated in the below table. The relative locations of the various storage piles are also indicated by compass direction. The aggregate storage piles proposed for sampling are identified on Figure 1A.

Proposed Aggregate Storage Pile Sampling

Stone Type	Location	Tonnage	Proposed Samples
2B Stone	Northwest	9,946	10
1B Stone Pile	Northeast	1,695	2
2A Stone Pile	East	1,585	2
Screenings	South	1,983	2

Aggregate samples will be collected as material composites using AASHTO R90. The samples will be mixed and reduced in size prior to transmittal to the laboratory per AASHTO T248. Samples will be transmitted to the laboratory in one gallon sealed plastic bags. To accomplish the AASHTO R90 sampling, the 2B storage pile will be surveyed and marked into ten radial sections for subsequent collection of ten samples. Samples from the remaining storage piles will be collected from opposite sides of the piles. The exact sample locations will be determined and marked in the field at the time of sampling. Samples will be collected from storage pile locations that were not previously sampled.

Upon reduction of the sample sizes per AASHTO T248, the samples will be transmitted under chain-of-custody to RJ Lee Group, Inc. in Monroeville, PA for microscopic analysis and Polarized Light Microscopy (PLM). If asbestiform minerals are found via PLM analysis, the sample will be further analyzed by Transmission Electron Microscopy (TEM) to confirm mineral identification and morphology. Analytical methods to be employed for the proposed testing are included in Attachment 1. The Pennsylvania and national laboratory accreditation certificates for RJ Lee Group, Inc. are included in Attachment 2.

ROCK CORING

Four rock cores will be advanced in the planned mining area to an elevation of approximately 585 feet above mean sea level (MSL), which is approximately equivalent to the current water level in the Quarry pit. The approximate coring locations are shown on Figure 1B. The cores will be drilled on an approximately 30-degree angle from vertical with an azimuth of approximately N45W. This azimuth is projected perpendicular to the geologic structure (ridgeline trend and geological strike) and was chosen to intercept as many potential features as practical. Where safely accessible, the highwalls below the coring locations will be examined prior to drilling to assess the presence and trend (dip and azimuth) of mineral veining. Based upon what is observed, the azimuth and location of the core drilling locations may be modified to better intercept found mineral vein trends. The exact core locations will be determined and marked in the field at the time of drilling. Continuous wetting of the core will be accomplished during drilling to minimize dust formation. Retrieved cores will be boxed, labeled and stored onsite for logging and analysis.

A professional geologist will visually log the cores to identify and record the following:

- 1) Geological description;
- 2) Mineralogy and grain size;
- 3) The percentage of core recovered;
- 4) Bedding observations;
- 5) Fracture occurrence;
- 6) Mineral veining; and,
- 7) Other pertinent geological features.

Found mineral veins will be examined using a hand lens and fine steel pick to assess the presence of fibrous mineral morphology. If potentially suspect mineral morphology is identified, the mineral veining will be photographed and sampled in the following manner:

- 1) The section of the core containing the suspect mineral vein will be isolated using a wet saw or core splitter;
- 2) The portion of the core to be sampled will be split by wet sawing. Half of the sample will be saved, while the remainder of the sample will be transmitted for laboratory analysis.

The PA DEP will have the opportunity to collect a split sample from the sampled portion of the core;

- 3) The sample will be sent to RJ Lee Group, Inc. in Monroeville, PA under chain-of-custody for microscopic and PLM analysis; and,
- 4) If asbestiform minerals are found via PLM analysis, the sample will be further analyzed by TEM to confirm mineral identification and morphology.

Additionally, one (1) diabase sample from each rock core (4 samples total) will be collected from a representative core portion where veining is absent. Sample preparation and analysis will be in accordance with the above procedures.

BOULDER FIELD ANALYSIS

A boulder field exists on the southern and eastern sides of the quarry pit. Examinations of at least 30 mineral veins on randomly selected boulders located in the boulder field will be completed. A professional geologist will examine the found mineral veining with a hand lens and fine steel pick to assess the presence of fibrous mineral morphology. If potentially suspect mineral morphology is identified, the mineral veining will be photographed and sampled. The exact boulder and vein locations will be determined and marked in the field at the time of sampling. The samples will be sent to RJ Lee Group, Inc. in Monroeville, PA under chain-of-custody for microscopic and PLM analysis. If asbestiform minerals are found via PLM analysis, the sample will be further analyzed by TEM to confirm mineral identification and morphology.

WATER SAMPLING

Water samples will be collected from the NPDES discharge, Sediment Basin 1 (southern basin), Sediment Basin 2, and the Quarry pit (4 samples total). The water samples will be collected as grab samples using a telescopic pole sampler containing an attached sampling cup. The sampling apparatus will be washed with Alconox® and rinsed with distilled water initially and between each sampling location. Sample collection will proceed from downgradient to upgradient to minimize potential for sample cross-contamination. Proposed water sampling locations are shown on Figure 1A. The water samples will be collected in clean laboratory-provided one liter plastic bottles. The samples will be placed on ice and transmitted under chain-of-custody to RJ Lee Group, Inc. in Monroeville, PA for analysis per EPA Method 100.2.

SAMPLING AND REPORTING

Upon approval of the work plan, Hanson is prepared to commence sampling at the site. Results of the investigation should be available approximately three (3) weeks after submittal of all samples to the laboratory. Status reports will be provided to the PA DEP weekly once sampling has

commenced. The results of the investigation will be provided in a Qualitative Geologic Survey Report.

If you have any questions or concerns regarding the proposed investigation and sampling, please feel free to contact me at (215) 766-1211.

Sincerely,
Earthres Group, Inc.



Louis F. Vittorio, Jr., P.G.
Vice President

Figure 1A – Water & Aggregate Sampling Locations
Figure 1B – Proposed Coring Locations
Attachment 1 – Sample Analysis Procedures and Methods
Attachment 2 – Laboratory Certifications

cc: Mark E. Kendrick, Hanson*
Andrew J. Gutshall, P.G., Hanson*
Matthew S. Burns, Esq, Hanson*
Curt Mitchell, R.E. Pierson
Mike Logan, CPS*
Kelly F. Bailey, CIH, KBC, LLC*
Drew R. Van Orden, P.E., RJ Lee Group, Inc*
Michael P. Kutney, P.G., PA DEP*
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Marianne Morano, East Rockhill Township*
David J. Raphael, Esq., K&L Gates*
Robert W. Gundlach, Esq., Fox Rothschild*
(*via electronic mail)

FIGURES

Figure 1A

Water and Aggregate Sampling Locations

Legend




-  Aggregate Storage Pile
-  Water Sampling Location



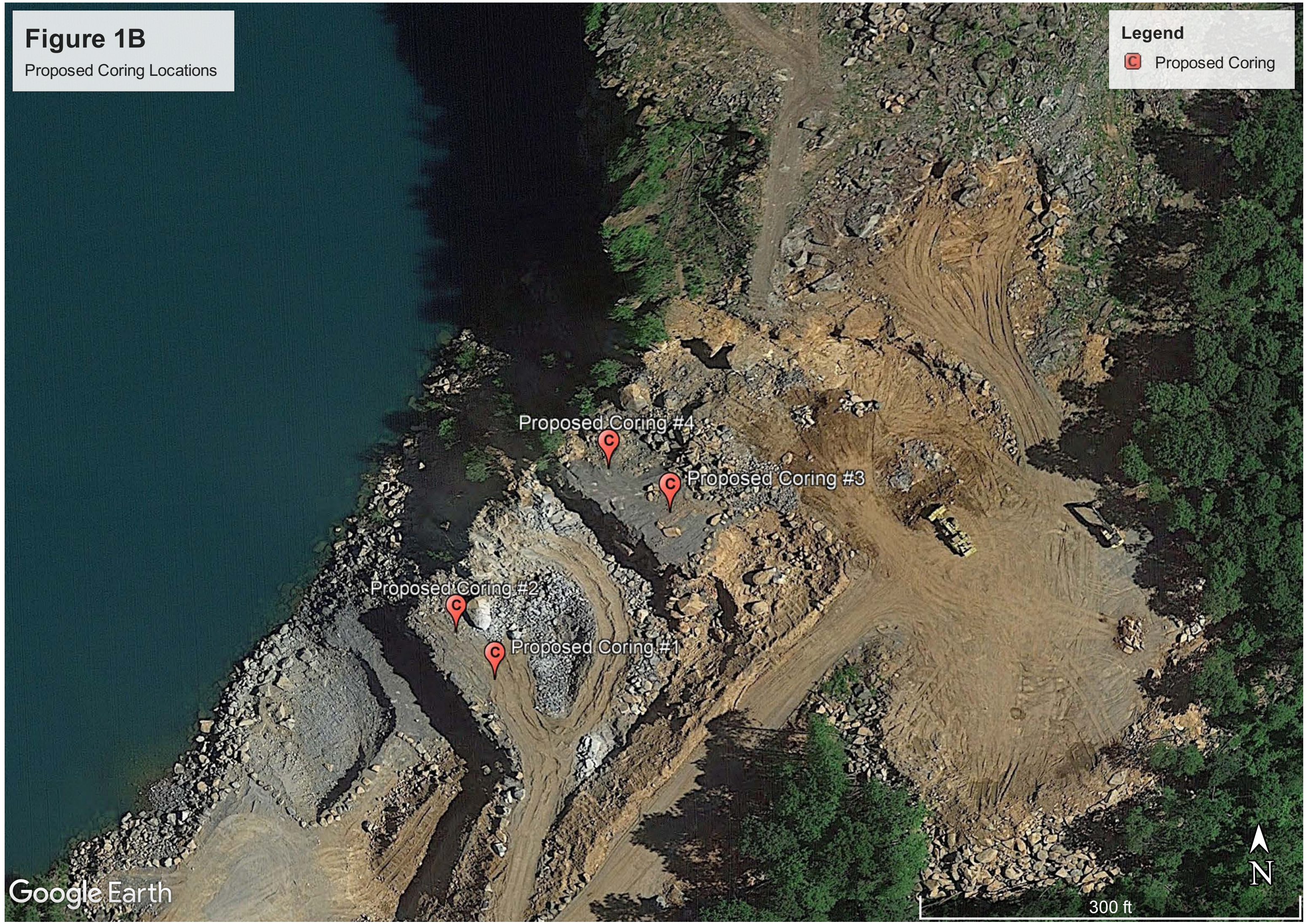
Figure 1B

Proposed Coring Locations

Legend

 Proposed Coring

Google Earth



300 ft

ATTACHMENT 1
Sample Analysis Methods

Attachment 1

Sample Analysis Procedures and Methods

For obtaining a representative sample from a large bulk sample, the AASHTO procedures for reducing the sample should be used. The subsequent analyses of the submitted samples will follow a three step procedure: 1) Basic microscopic analysis to assess the presence of asbestiform mineral habitat; 2) Polarized Light Microscopy (PLM) to determine the presence and asbestos mineral type, if present; and, 3) Should positive results be indicated by PLM, follow-up Transmission Electron Microscopy (TEM) analysis will be completed to confirm the minerals present and their morphology. The techniques and methods to be employed in sample analysis are provided below:

- A geologist will inspect hand and core samples initially using a stereo binocular microscope, with magnification ranging from 10x to 60x. Using a fine steel pick (dental pick) the geologist will scrape the surface of the suspect mineralization to determine if any of the minerals display typical asbestiform habit and characteristics such as fiber bundles, splayed ends, or matted or fibrous masses.
- Further examination of the sample will then be conducted using the Polarized Light Microscope (PLM) using EPA 600/R-93/116.
- If asbestiform minerals are found, representative samples will be further analyzed by Transmission Electron Microscopy per EPA 600/R-93/116 to confirm mineral identification and morphology.
- Where appropriate, the microscopic PLM and/or TEM analyses will include a count of the asbestiform fibers, representative digital images, and measurements of the width and length dimensions of found fibers counted.

Water samples will be collected as grab samples and will be analyzed by TEM per EPA 100.2.

The samples will be analyzed using the above procedures by RJ Lee Group, which is accredited by the American Industrial Hygiene Association and is in the NIST National Voluntary Laboratory Accreditation Program for asbestos analysis. RJ Lee Group has mineralogical expertise and has vast experience to detect asbestos fibers in the natural environment (e.g. rocks, soils, water, etc.).

ATTACHMENT 2

Laboratory Accreditation Certificates

COMMONWEALTH OF PENNSYLVANIA
DEPARTMENT OF ENVIRONMENTAL PROTECTION

BUREAU OF LABORATORIES
LABORATORY ACCREDITATION PROGRAM



Certifies That

02-00396

RJ Lee Group, Inc.

350 Hochberg Road, Monroeville, PA 15146



Having duly met the requirement of
The act of June 29, 2002 (P.L. 596, No. 90)
dealing with Environmental Laboratories Accreditation
(27 Pa. C.S. §§4104-4113) and the
National Environmental Laboratory Accreditation Program Standard

is hereby approved as an

Accredited Laboratory

to conduct analysis within the fields of accreditations more fully described in the attached Scope of Accreditation

Expiration Date: **04/30/2019**

Certificate Number: **014**

A handwritten signature in black ink that reads "Aaren Alger".

Aaren S. Alger, Chief
Laboratory Accreditation Program
Bureau of Laboratories



AIHA Laboratory Accreditation Programs, LLC

acknowledges that

RJ Lee Group, Inc.

350 Hochberg Road, Monroeville, PA 15146

Laboratory ID: 100364

along with all premises from which key activities are performed, as listed above, has fulfilled the requirements of the AIHA Laboratory Accreditation Programs (AIHA-LAP), LLC accreditation to the ISO/IEC 17025:2005 international standard, *General Requirements for the Competence of Testing and Calibration Laboratories* in the following:

LABORATORY ACCREDITATION PROGRAMS

- | | |
|---|---------------------------------------|
| <input checked="" type="checkbox"/> INDUSTRIAL HYGIENE | Accreditation Expires: April 01, 2020 |
| <input checked="" type="checkbox"/> ENVIRONMENTAL LEAD | Accreditation Expires: April 01, 2020 |
| <input type="checkbox"/> ENVIRONMENTAL MICROBIOLOGY | Accreditation Expires: |
| <input type="checkbox"/> FOOD | Accreditation Expires: |
| <input type="checkbox"/> UNIQUE SCOPES | Accreditation Expires: |

Specific Field(s) of Testing (FoT)/Method(s) within each Accreditation Program for which the above named laboratory maintains accreditation is outlined on the attached **Scope of Accreditation**. Continued accreditation is contingent upon successful on-going compliance with ISO/IEC 17025:2005 and AIHA-LAP, LLC requirements. This certificate is not valid without the attached **Scope of Accreditation**. Please review the AIHA-LAP, LLC website (www.aihaaccreditedlabs.org) for the most current Scope.

Elizabeth Bair

Elizabeth Bair
Chairperson, Analytical Accreditation Board

Cheryl O. Morton

Cheryl O. Morton
Managing Director, AIHA Laboratory Accreditation Programs, LLC



AIHA Laboratory Accreditation Programs, LLC SCOPE OF ACCREDITATION

RJ Lee Group, Inc.
350 Hochberg Road, Monroeville, PA 15146

Laboratory ID: **100364**
Issue Date: 03/30/2018

The laboratory is approved for those specific field(s) of testing/methods listed in the table below. Clients are urged to verify the laboratory's current accreditation status for the particular field(s) of testing/Methods, since these can change due to proficiency status, suspension and/or withdrawal of accreditation.

Industrial Hygiene Laboratory Accreditation Program (IHLAP)

Initial Accreditation Date: 09/01/1991

IHLAP Scope Category	Field of Testing (FoT) (FoTs cover all relevant IH matrices)	Technology sub-type/ Detector	Published Reference Method/ Title of In-house Method	Method Description or Analyte <i>(for internal methods only)</i>
Chromatography Core	Gas Chromatography	GC/FID	NIOSH 1003	
			NIOSH 1005	
			NIOSH 1007	
			NIOSH 1022	
			NIOSH 1300	
			NIOSH 1301	
			NIOSH 1400	
			NIOSH 1401	
			NIOSH 1402	
			NIOSH 1403	
			NIOSH 1450	
			NIOSH 1453	
			NIOSH 1457	
			NIOSH 1458	
			NIOSH 1459	
			NIOSH 1500	
			NIOSH 1501	
			NIOSH 1550	
			NIOSH 1615	
			NIOSH 2000	
NIOSH 2500				
NIOSH 2537				
NIOSH 2546				
NIOSH 2553				
NIOSH 2554				



IHLAP Scope Category	Field of Testing (FoT) (FoTs cover all relevant IH matrices)	Technology sub-type/ Detector	Published Reference Method/Title of In-house Method	Method Description or Analyte <i>(for internal methods only)</i>
Chromatography Core	Gas Chromatography	GC/FID	NIOSH 2555	
			NIOSH 4000	
			NIOSH 5020	
			NIOSH 5515	
			NIOSH 5523	
	Gas Chromatography (Diffusive Samplers)		NIOSH 1003	
			NIOSH 1005	
			NIOSH 1007	
			NIOSH 1022	
			NIOSH 1300	
			NIOSH 1301	
			NIOSH 1400	
			NIOSH 1401	
			NIOSH 1402	
			NIOSH 1403	
			NIOSH 1450	
			NIOSH 1453	
			NIOSH 1457	
			NIOSH 1458	
			NIOSH 1459	
			NIOSH 1500	
			NIOSH 1501	
			NIOSH 1550	
			NIOSH 1615	
			NIOSH 2000	
			NIOSH 2500	
			NIOSH 2537	
			NIOSH 2546	
			NIOSH 2553	
			NIOSH 2554	
			NIOSH 2555	
			NIOSH 4000	
			NIOSH 5020	
NIOSH 5515				
NIOSH 5523				
Ion Chromatography (IC)		NIOSH 7903		
Liquid Chromatography	HPLC/UV	OSHA ID - 215		
		NIOSH 2016		
		NIOSH 5506		



IHLAP Scope Category	Field of Testing (FoT) (FoTs cover all relevant IH matrices)	Technology sub-type/ Detector	Published Reference Method/Title of In-house Method	Method Description or Analyte <i>(for internal methods only)</i>
Chromatography Core	Liquid Chromatography	HPLC/UV	OSHA 42	
			OSHA 47	
			OSHA 58	
Spectrometry Core	Atomic Absorption	CVAA	NIOSH 6009	
	Inductively-Coupled Plasma	ICP/MS	NIOSH 7300 Modified	
		ICP/AES	NIOSH 7300	
			NIOSH 7300 Modified	
	X-ray Diffraction (XRD)		NIOSH 7500	
	Infrared		NIOSH 5026	
Asbestos/Fiber Microscopy Core	Polarized Light Microscopy (PLM)		40 CFR Part 763, Sub. E., Appendix E	Interim Method of the Determination of Asbestos in Bulk Insulation Samples
			EPA 600/R-93/116	
	Phase Contrast Microscopy (PCM)		NIOSH 7400A	
	Transmission Electron Microscopy (TEM)		40 CFR Part 763, Sub. E., Appendix A	
NIOSH 7402				
Miscellaneous Core	Gravimetric		NIOSH 0500	
			NIOSH 0600	
			NIOSH 5042	
			OSHA 58	
	Thermo-optical Analysis (TOA)		NIOSH 5040	
Beryllium Testing	Inductively-Coupled Plasma	ICP/MS	NIOSH 7303 Modified	
		ICP/AES	NIOSH 7300	
			NIOSH 7300 Modified	

A complete listing of currently accredited Industrial Hygiene laboratories is available on the AIHA-LAP, LLC website at: <http://www.aihaaccreditedlabs.org>



AIHA Laboratory Accreditation Programs, LLC SCOPE OF ACCREDITATION

RJ Lee Group, Inc.

350 Hochberg Road, Monroeville, PA 15146

Laboratory ID: **100364**

Issue Date: 04/10/2018

The laboratory is approved for those specific field(s) of testing/methods listed in the table below. Clients are urged to verify the laboratory's current accreditation status for the particular field(s) of testing/Methods, since these can change due to proficiency status, suspension and/or withdrawal of accreditation.

The EPA recognizes the AIHA-LAP, LLC ELLAP program as meeting the requirements of the National Lead Laboratory Accreditation Program (NLLAP) established under Title X of the Residential Lead-Based Paint Hazard Reduction Act of 1992 and includes paint, soil and dust wipe analysis. Air and composited wipes analyses are not included as part of the NLLAP.

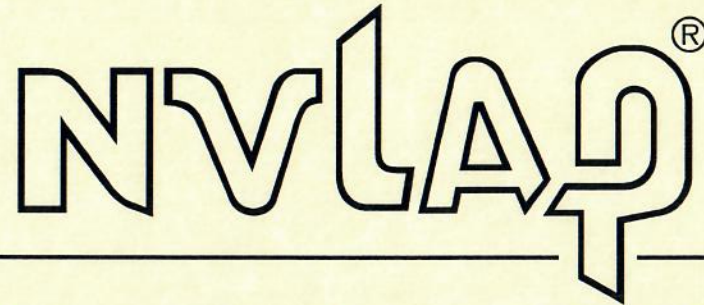
Environmental Lead Laboratory Accreditation Program (ELLAP)

Initial Accreditation Date: 12/05/1995

Field of Testing (FoT)	Technology sub-type/ Detector	Method	Method Description (for internal methods only)
Settled Dust by Wipe		EPA SW-846 3050B	
		EPA SW-846 7000B	

A complete listing of currently accredited Environmental Lead laboratories is available on the AIHA-LAP, LLC website at: <http://www.aihaaccreditedlabs.org>

United States Department of Commerce
National Institute of Standards and Technology



Certificate of Accreditation to ISO/IEC 17025:2005

NVLAP LAB CODE: 101208-0

RJ Lee Group, Inc.
Monroeville, PA

*is accredited by the National Voluntary Laboratory Accreditation Program for specific services,
listed on the Scope of Accreditation, for:*

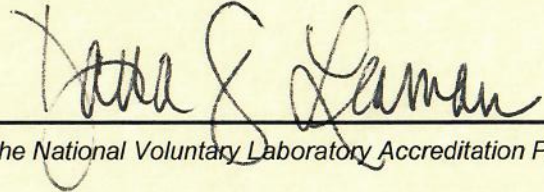
Asbestos Fiber Analysis

*This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005.
This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality
management system (refer to joint ISO-ILAC-IAF Communique dated January 2009).*

2018-07-01 through 2019-06-30

Effective Dates




For the National Voluntary Laboratory Accreditation Program



SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

RJ Lee Group, Inc.
350 Hochberg Road
Monroeville, PA 15146-1516
Ms. Tammie Mussitsch
Phone: 724-325-1776 Fax: 724-733-1799
Email: accreditations@rjlg.com
<http://www.RJLG.COM>

ASBESTOS FIBER ANALYSIS

NVLAP LAB CODE 101208-0

Bulk Asbestos Analysis

<u>Code</u>	<u>Description</u>
18/A01	EPA -- 40 CFR Appendix E to Subpart E of Part 763, Interim Method of the Determination of Asbestos in Bulk Insulation Samples
18/A03	EPA 600/R-93/116: Method for the Determination of Asbestos in Bulk Building Materials

Airborne Asbestos Analysis

<u>Code</u>	<u>Description</u>
18/A02	U.S. EPA's "Interim Transmission Electron Microscopy Analytical Methods-Mandatory and Nonmandatory-and Mandatory Section to Determine Completion of Response Actions" as found in 40 CFR, Part 763, Subpart E, Appendix A.

For the National Voluntary Laboratory Accreditation Program

METHOD 100.2

**DETERMINATION OF ASBESTOS STRUCTURES OVER
10 μm IN LENGTH IN DRINKING WATER**

Kim A. Brackett, Ph.D. (IT Corp.)

Patrick J. Clark (Risk Reduction Engineering Laboratory, USEPA)

James R. Millette, Ph.D. (MVA, Inc.)

June 1994

OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

METHOD 100.2

DETERMINATION OF ASBESTOS STRUCTURES OVER
10 µm IN LENGTH IN DRINKING WATER

1.0 SCOPE AND APPLICATION

- 1.1 This test method is recommended for the determination of the presence and quantitation of asbestos structures in drinking water samples. The method allows for the quantitation of structures greater than 10 µm in length.
- 1.2 This test method describes the equipment and procedures necessary for the sampling and analysis of drinking water by transmission electron microscopy (TEM).
- 1.3 The identification of asbestos by TEM is based on (a) morphology, (b) selected area electron diffraction (SAED) and (c) energy dispersive x-ray analysis (EDXA).
- 1.4 Applicable analytes and Chemical Abstract Service (CAS) Numbers:

Asbestos	CAS Number
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Amosite (Grunerite)	12001-73-5
Anthophyllite	77536-67-5
Tremolite	77536-68-6
Actinolite	77536-66-4

1.5 Data Quality Objectives

Method	Accuracy ^a	Precision ^a	Completeness
TEM	95%	95%	100%

^a Confidence coefficient of a confidence interval for a Poisson variable within which count ranges are expected to fall.

- 1.6 Analytical Sensitivity. A sensitivity of 200,000 fibers per liter (0.2 MFL) is required unless filter loading satisfies the stopping rules in Section 11.31. See TABLE 1.

1.7 Only asbestos structures meeting the definitions set forth in the Chatfield protocol are counted (1).

TABLE 1. Limitation of Analytical Sensitivity by Volume of Water Sample Filtered (1)

Volume of Sample Filtered (mL)		Analytical Sensitivity (f/L)
25 mm Diam. Filter ^b	47 mm Diam. Filter ^c	
0.1	0.6	3.0×10^7
0.5	2.8	6.0×10^6
1.0	5.7	3.0×10^6
2.0	11	1.6×10^6
5.0	28	6.0×10^5
10	57	3.0×10^5
15	85	2.0×10^5
25	142	1.2×10^3
50	285	6.0×10^4
100	570	3.0×10^4

^a Concentration corresponding to 1 structure detected in 10 grid openings of approximately 0.008 mm².

^b Assuming active filter area of 1.99 cm².

^c Assuming active filter area of 11.34 cm².

2.0 SUMMARY OF METHOD

Water is collected in a polyethylene or glass container and shipped to the laboratory. Known aliquots of the sample are filtered through a 0.1 to 0.22 µm pore mixed cellulose ester (MCE). A carbon extraction replica is prepared from a portion of the filter and is examined in the TEM at a magnification of 10,000 to 20,000X. Asbestos structures are identified by morphology, selected area electron diffraction (SAED) and energy dispersive x-ray analysis (EDXA). Structures are classified according to the counting rules specified in the Chatfield polycarbonate filter protocol (1). Only asbestos structures greater than 10 µm in length are counted. Some **states** may require identification and measurement of all asbestos fibers,

regardless of size. In this case the use of a 0.1 μm pore-size polycarbonate or MCE filter membrane is necessary to prevent loss of small fibers during filtration.

3.0 DEFINITION OF TERMS

3.1 Analytical Sensitivity -- The waterborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the fraction of the sample filtered and the dilution factor (if applicable).

3.2 Asbestiform -- A specific type of fibrous habit which has greater flexibility and higher tensile strength than other habits of the same mineral.

3.3 Asbestos -- generic term for a group of hydrated mineral silicates.

3.4 Aspect Ratio -- The ratio of the length of a fibrous particulate to its apparent width (equivalent diameter).

3.5 Bundle -- A structure composed of three or more fibers in a parallel arrangement with each fiber lying less than one diameter apart.

3.6 Cluster -- A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group.

3.7 EDXA -- Energy dispersive X-ray analysis.

3.8 Fiber -- For the purposes of this method, a structure having a minimum length of 10 μm and an aspect ratio (length to width) of 3:1 or greater with substantially parallel sides.

3.9 Fibril -- The smallest crystalline fiber that can be separated from a fiber bundle which cannot be subdivided without losing its fibrous properties.

3.10 Grid -- a 3 mm diameter 200-mesh copper lattice used to hold the carbon extraction replica for observation in the TEM.

3.11 Intersection -- Nonparallel touching or crossing of fibers, with the projection having an aspect ratio $\geq 3:1$.

3.12 Matrix -- Fiber or fibers with one free end and the other end embedded in or hidden by a particulate.

3.13 MFL -- million fibers per liter.

3.14 SAED or ED -- Selected area electron diffraction.

3.15 Structure -- A microscopic bundle, cluster, fiber or matrix which may contain asbestos.

3.16 TEM -- transmission electron microscope.

4.0 INTERFERENCES

The minerals listed below can exhibit morphological, chemical or crystal structure similarities to the asbestos minerals. The laboratory QA/QC manual should describe routine techniques to differentiate them from asbestos. In general, these techniques should be the same as those required for accreditation by the National Institute of Standards and Technology/National Voluntary Laboratory Accreditation Program (NIST/NVLAP) for airborne asbestos.

4.1 Antigorite

4.2 Attapulgite (Palygorskite)

4.3 Halloysite

4.4 Horneblende

4.5 Pyroxenes

4.6 Sepiolite

4.7 Vermiculite scrolls

5.0 SAFETY

This test method may involve hazardous materials, operations and equipment, and does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Sample filtration take place in a clean HEPA filtered positive pressure hood to avoid possible contamination of the preparation. Collapsing of the filter should be performed in an exhaust hood.

6.0 EQUIPMENT AND SUPPLIES

6.1 Transmission Electron Microscope capable of performing electron diffraction, with a fluorescent screen inscribed with a calibrated measuring scale. The TEM must have EDXA and be able to produce a spot size, at crossover, less than 250 nm in diameter.

6.2 Energy dispersive X-ray analyzer

6.3 High vacuum carbon evaporator with rotating stage

6.4 Positive pressure HEPA filtered hood

6.5 Fume hood

6.6 Table-top low power ultrasonic bath

6.7 Ozone generator capable of generating at least 400g of ozone per day at a concentration of 1% by weight when supplied with dry oxygen.

6.8 Quartz pipets

6.9 Submersible UV lamp (254 μm wavelength)

6.10 Waterproof marker

6.11 Forceps (tweezers)

6.12 Graduated pipettes (1, 5, 10 ml sizes), disposable glass

6.13 25 or 47 mm diameter filter funnel assembly (either glass or disposable plastic)

6.14 1000 mL side arm vacuum filtration flask

6.15 25 or 47 mm diameter mixed cellulose ester (MCE) membrane filters ($\leq 0.22 \mu\text{m}$ and $5 \mu\text{m}$ pore size)

6.16 Disposable petri dishes (or suitable equivalent) for storage of filtration membranes

6.17 Glass microscope slides

6.18 Curved scalpel blades

6.19 Low temperature oven or cabinet-type desiccator

- 6.20 Low temperature plasma asher
- 6.21 Jaffe washer
- 6.22 200 mesh copper TEM finder grids
- 6.23 Carbon rods
- 6.24 1000 mL glass or polyethylene sample bottles with screw-on caps

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (2).

- 7.2 Deionized particle-free water
- 7.3 Acetone
- 7.4 Dimethylformamide (DMF)
- 7.5 Glacial acetic acid
- 7.6 Chloroform
- 7.7 1-methyl-2-pyrrolidone also called 1-methyl-2-pyrrolidinone or n-methyl-2-pyrrolidone (CAS 872-50-4)
- 7.8 NIST traceable asbestos standards
- 7.9 Laboratory standards for the interference minerals listed in Section 4.0.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 The sample container will be an unused, pre-cleaned, screw-capped bottle of glass or low density (conventional) polyethylene of at least 1 liter capacity. It is recommended that the use of polypropylene bottles be avoided since problems of particulate being released into water samples have been observed. Before use, the bottles should be rinsed twice by filling approximately one-third full with fiber-free water and shaking vigorously for thirty seconds. After discarding the rinse water, the bottles should then be filled with fiber-free water and treated in an ultrasonic bath for 15 minutes, followed by several rinses with fiber-free water.

8.2 Blank determinations should be made prior to sample collection. When using polyethylene bottles, one bottle from each batch, or a minimum of one from each 24, should be tested for background level. When using glass bottles, four bottles from each 24 should be tested. Additional blanks may be desirable when sampling waters suspected of containing very low levels of asbestos, or when additional confidence in the bottle blanks are desired. An acceptable bottle blank level is defined as ≤ 0.01 MFL $> 10 \mu\text{m}$.

8.3 SAMPLE COLLECTION-- It is beyond the scope of this procedure to furnish detailed instructions for field sampling; the general principles of obtaining water samples apply. If tests are being made of drinking water in a bulk storage supply, there may be a vertical distribution of particle sizes. If a representative sample of the water supply is required, a carefully designated set of samples should be taken representing the vertical as well as the horizontal distribution and then composited for analysis. Compositing must be done in the laboratory, and not in the field.

When sampling from a distribution system a commonly used faucet should be chosen. Remove all hoses or fittings from the faucet and allow the water to run to waste for a sufficiently long period to ensure that the sample collected is representative of the fresh water supply. For most buildings this may be indicated by a change in temperature of the water at the faucet. Faucets or valves should not be adjusted until all samples have been collected. Samples should not be taken from hydrants or other faucets at the deadend of a distribution system.

As an additional precaution against contamination, each bottle may be rinsed several times in the source water being tested before the final sample is taken. This procedure is not suitable when taking depth samples from a storage tank, however.

8.4 QUANTITY OF SAMPLE-- Two samples of approximately 800 mL should be collected from each site. No preservatives should be added during sampling. This sample volume will leave an air space to allow efficient redispersal of settled material, by shaking, before analysis. Each bottle should be labeled with the date, time, place, field sample number and sampler's name using a waterproof marker.

8.5 SAMPLE SHIPMENT-- Samples must be transported to the analytical laboratory as soon as possible after collection. The samples should be shipped in a sealed container, separate from any bulk or air samples. Samples should be shipped in a cooler with ice to retard bacterial or algal growth in the samples. Do not freeze the samples. Samples should be received and filtered in the laboratory within 48 hours of collection. Samples must be accompanied by a properly executed chain of custody document.

8.6 SAMPLE PRESERVATION-- Samples should be filtered immediately after arrival at the laboratory or stored in a refrigerator until filtered.

8.7 SAMPLE COMPOSITING-- Up to five samples may be composited after receipt in the laboratory. The composite sample must be prepared from the individual samples within 48 hours of collection, or, if the samples have been stored for more than 48 hours, they must be individually treated with O₃-UV in the original containers. Samples should be sonicated in the original container and equal amounts extracted to make up the composite. It may also be prudent to filter an aliquot of each sample for analysis in case the composite sample exceeds 1/5th the MCL (1.4 MFL >10 μm long). If, later, the original samples are to be filtered separately, they must be treated again with O₃-UV in the original containers and re-sonicated.

9.0 QUALITY CONTROL

9.1 The quality control checks required for this method generally follow those specified in the Federal Register for AHERA analysis of air samples (3) and the NISTIR document relating to airborne asbestos analysis (4). These requirements are summarized in Table 2. The criterion for acceptability of bottle and process blanks is ≤0.01 MFL >10 μm in length.

10.0 CALIBRATION AND STANDARDIZATION

10.1 MAGNIFICATION CALIBRATION-- Magnification calibration must be done at the fluorescent screen and must be performed at the magnification used for fiber counting, generally 10,000 and 20,000X. Calibration is performed using a grating replica (e.g., one containing at least 2,160 lines/mm). Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric). If the instrument contains a tilting stage (goniometer), the z-axis must be adjusted to ensure that the stage is in the eucentric position prior to performing any measurements. A logbook must be maintained with the dates of the calibration recorded. Frequency of calibration will depend on the service history of the instrument. It is recommended that calibrations be performed monthly to establish the stability of the magnification. Also, the calibration should be checked following any maintenance of the microscope involving adjustment of the lens or high voltage power supplies or the disassembly of the electron optical column apart from filament exchange.

10.2 CAMERA CONSTANT-- The camera length of the TEM in the electron diffraction (ED) mode must be calibrated before ED patterns of unknown samples are observed. This can be achieved by using a carbon-coated grid on which a thin film of gold has

been sputtered or evaporated. A thin film of gold can also be evaporated on the specimen grids to obtain ED patterns superimposed on the ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thicker gold films will tend to mask weaker diffraction spots from the fibrous particulates. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings from thicker films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant on a regular basis for each TEM in the laboratory. The stage must be at the eucentric position for this calibration. The camera constant calculated for that particular instrument can then be used for ED patterns of unknowns taken during the corresponding period.

10.3 SPOT SIZE-- The diameter of the smallest beam spot at crossover must be measured regularly. Photograph the beam at crossover at 20,000 to 25,000X at a short exposure setting (to avoid spreading of the exposed spot on the film). Measure the diameter on the negative and divide by the magnification used.

TABLE 2. SUMMARY OF LABORATORY DATA QUALITY OBJECTIVES

<u>Unit Operation</u>	<u>QC Check</u>	<u>Frequency Expectation</u>	<u>Conformance</u>
Sample Receiving	Review chain-of-custody and sampling data	Each sample	95% complete
Sample Prep	Supplies and reagents	On Receipt	Meet specs.
	Grid opening size	20/20 grids/lot of 1000 or 1/sample	100%
	Clean area monitoring	After service	Meet specs.
	Lab blank	1/series or 10%	Meet specs.
	Etcher blank	1/20 samples	75%
	>3 grids/sample	Each sample	≥70% intact openings
Sample Analysis	System check	Each day	Each day
	Alignment check	Each day	Each day
	Mag calibration	Monthly or after service	95%
	Camera constant	Weekly	95%
	EDS Cu K" line	Each day	95%
Performance	Lab blank	1/prep series or 10%	Meet specs.
	Replicate count	≥1/100 samples	1.5XPoisson std dev.
	Duplicate count	≥1/100 samples	2XPoisson std. dev.
	Analysis of standards	Training and comparison with unknowns	100%
	Analysis of SRM	1 per analyst per year	1.5XPoisson std dev.
	Data entry	Each sample	95%
	Record and verify SAED patterns	1/5 samples	80% accuracy
Calculations	Hand calculation of automated data reductions or 2 nd analyst check of manual calcs.	1/100 samples	85%

The resulting figure must be <250 nm.

10.4 EDXA SYSTEM-- The resolution and calibration of the EDXA must be checked at least monthly and after service. Initially, the system is calibrated by using two reference elements to calibrate the energy scale of the instrument according to the manufacturer's instructions. This can easily be done using a carbon-coated copper grid upon which a thin film of aluminum has been evaporated. The Al and Cu K^α peaks should be centered at 1.48 KeV and 8.04 KeV respectively. The deviation from these energies should be no more than ±10eV. The ability of the system to resolve the sodium K^α line from the copper L line should be confirmed by obtaining a spectrum from a standard crocidolite sample on a copper grid. Additional resolution checks are usually found in the manufacturer's instruction manual. K-factors relative to silicon should be calculated for Na, Mg, Al, Si, Ca and Fe using NIST SRM 2063. The k factor for Mg to Fe must be calculated; a value of 1.5 or less is required. EDXA spectra should be obtained from NIST traceable standards and kept on file in the laboratory for comparison with published standard spectra and unknown spectra.

11.0 PREPARATION AND ANALYSIS

11.1 If the water samples are suspected to contain high levels of organic contaminants, or have been stored for periods longer than 48 hours, oxidation of the organics by the ozone-ultraviolet technique (1) may be necessary. Details for this procedure appear in Appendix 1.

11.2 Wet-wipe the exterior of the sample bottle before entering the clean area used for specimen preparation. The sample prep area should be separate from the areas used for bulk sample or air sample preparation to avoid possible cross-contamination. Sample filtration should take place in a positive pressure HEPA filtered hood.

11.3 The use of vertical sided 25 or 47 mm diameter glass filtration systems with a sintered glass frit support is recommended to avoid loss of fibers by settling on tapered sides of the reservoir. Disposable plastic funnel assemblies may be substituted for glass apparatus. A few precautions must be taken with re-usable glassware to ensure optimum sample preparations. All glassware should be carefully washed in a detergent solution with a brush before each use and rinsed several times in fiber-free water. Any glassware that has contained asbestos in solution should be placed in soapy water and scrubbed before it has had the opportunity to dry. Sonication in a detergent solution is also recommended. Frequent blanks should be run with fiber-free water to check cleanliness of the apparatus.

11.4 Unwrap an unused disposable plastic filter funnel unit and remove the tape around the base of the funnel. Remove the funnel and discard the top filter membrane supplied with the

unit. Do not remove the coarse polypropylene support pad. Assemble the unit with the adapter and a properly sized neoprene one-hole stopper, and attach the assembly to the 1000 mL vacuum flask.

11.5 Whether using a glass or plastic filtration unit, care must be taken to ensure that the filter support and the mating surface of the filtration base not be damp when the backing filter is placed on the support. If this should occur, the vacuum across the filtration surface will be uneven, resulting in uneven distribution of the filtered particulates. Either the filtration base must be thoroughly dried before use, or completely wetted so that the backing filter and filtration membrane are uniformly wet before filtration is started.

11.6 DISPOSABLE FILTER UNIT-- Wet the support pad with distilled water, if desired, and place a 5 μm pore-size MCE backing filter on the pad. Place a ≤ 0.22 μm pore-size MCE filter membrane on the backing filter. Ensure that both filter membranes are completely wet, or dry depending upon the technique preferred. Apply a vacuum to the flask and ensure that the filters are centered and pulled flat with no entrapped air bubbles between the membranes. If any irregularities are seen on the filter surface, discard the filters and try again. Replace the funnel and seal the assembly with tape.

11.7 REUSABLE GLASS FILTER UNIT

a. (Dry Filter) Apply vacuum to the flask and leave the vacuum on until the filtration process is completed. Place a ≤ 5 μm pore-size MCE filter on the glass frit to serve as a backing filter. Be sure that the filter is not creased during installation. If the filter appears to absorb water, it should be discarded and the frit rinsed with methanol to speed drying. Place a ≤ 0.22 μm pore-size MCE filter membrane on the backing filter. Make sure that the mating surface of the filter reservoir is dry, then place it on the filters and clamp in place.

b. (Wet Filter) Wet the glass frit and place a 5 μm pore-size MCE filter on the frit to serve as a backing filter. Place a ≤ 0.22 μm pore-size MCE filter membrane on the backing filter. Make sure that both membranes are thoroughly soaked with water. Install the filter reservoir and clamp in place.

11.8 A process blank sample consisting of fiber-free water should be run before the first field sample. The quantity of water should be ≥ 10 mL for a 25 mm diameter filter and ≥ 50 mL for a 47 mm diameter filter.

11.9 Shake the capped sample bottle vigorously by hand and place in a low power ultrasonic bath for 15 minutes. Shake the sample by hand again before removal of any aliquots.

11.10 Remove the desired aliquot from the original sample. Large volumes may be measured with a graduated cylinder, smaller volumes should be taken with disposable glass pipettes. Samples taken by pipette should be taken from the vertical center of the original sample. No aliquot less than 1 mL should be taken from the original sample. The minimum volume that should be filtered is 10 mL for a 25 mm diameter filter, or 50 mL for a 47 mm diameter filter. If it is necessary to filter aliquots less than these volumes, the aliquots should be brought up to these levels with fiber-free water and shaken vigorously before filtration. Obtaining a filter with the proper loading is a matter of trial and error. It is best to filter several volumes of the sample. Samples with high particulate or asbestos content may require volumes less than 1 mL to be filtered. In this case, preparation of serial dilutions must be performed.

11.11 Agitate the aliquot and pour into the filter reservoir. Apply vacuum to the flask. When volumes larger than the capacity of the reservoir have to be filtered, the additional solution should be carefully added while the reservoir is over half full to avoid disturbing the particulates already deposited on the filter. **Do not rinse the sides of the reservoir.**

11.12 If a re-usable glass reservoir is used, **immediately** remove the reservoir and place in soapy water.

11.13 Disassemble the filtration apparatus and remove the filters with clean forceps. Carefully separate the working filter from the backing filter and discard the backing filter. Place the working filter in a pre-cleaned disposable petri dish and cover.

11.14 Allow filter to dry. Drying may take place in a HEPA filtered hood, an asbestos-free oven, or a cabinet type desiccator. The cover of the petri dish should be opened slightly to allow water vapor to escape.

11.15 Using a clean scalpel remove a portion of the dry filters for preparation of TEM grids by the direct transfer technique. Be sure to avoid the outer ring of the filter that was covered by the mating surface of the reservoir. Transfer the removed portion to an unused petri dish.

11.16 A portion of an unused filter should also be prepared as a lot blank.

11.17 MCE filters must be collapsed prior to low temperature etching. Use of either the DMF-Acetic acid method or the acetone

method is acceptable. Samples should be transferred to an exhaust hood for this step.

a. DMF-ACETIC ACID METHOD-- Place a drop of the clearing solution (35% dimethylformamide [DMF], 15% glacial acetic acid and 50% water [V/V] on a clean microscope slide. Use just enough solution to saturate the filter. DMF is a relatively toxic solvent and should be used in a fume hood. Carefully lay the filter segment, sample surface upward, on top of the solution, bringing the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any solution not absorbed by the filter with lens paper. Label the slide with a glass scribing tool or a permanent marker. Place the slide into an oven, or on the surface of a hot plate, at 65-70°C for 10 to 30 minutes.

b. ACETONE METHOD-- Place the filter section on a clean microscope slide. Affix the filter section to the slide with tape around the edges. Label the slide with a glass scribing tool or permanent marker. Place the slide in a petri dish containing several paper filters soaked with acetone. Cover the dish and wait for the sample to fuse and clear (approximately 5 minutes).

c. Plasma etching of the collapsed filter is required if 0.22 µm pore-size membrane filters are used. Plasma etching is optional (but recommended) with 0.1 µm pore-size filters. The microscope slide to which the collapsed filter pieces are attached is placed in a low temperature plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher barrel, it is difficult to specify the conditions that should be used. Insufficient etching will result in a failure to expose embedded fibrils, and too much etching may result in loss of particulate from the surface. It is recommended that conditions be used which will remove about 10% of the filter mass. Additional information about calibration of the plasma asher can be found in the AHERA (3) and NISTIR (4) documents.

11.18 CARBON-COATING FILTER SEGEMENTS

a. Coating must be performed with a high-vacuum evaporation unit equipped with a rotating tilting stage. Units based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and must not be used. The carbon rods should be sharpened by a carbon rod sharpener to necks of about 4 mm long and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 10 cm from the surface of the microscope slide.

b. Affix the glass slide to the rotating tilting table and evacuate the chamber to a pressure of ≤ 0.013 Pa. The evaporation must be performed in very short bursts, separated by some seconds to allow the electrodes to cool to avoid overheating the surface of the filter. An experienced analyst can judge the thickness of the carbon film to be applied, and some tests should be made first on unused filters. If the film is too thin, there will be few complete, undamaged grid openings on the specimen and large particles may be lost. If the coating is too thick, the TEM image will lack contrast and the ability to obtain selected area electron diffraction (SAED) patterns will be compromised. A carbon film that appears similar to a 15% gray scale is usually adequate.

11.19 Prepare the Jaffe washer. The precise design of the Jaffe washer is not important, so any one of the published designs may be used (3,5). Place the washer in a fume hood and fill with DMF, acetone or 1-methyl-2-pyrrolidone to the level of the screen on which the samples will be placed.

11.20 Place calibrated TEM grids in the Jaffe washer. Indexed ("Finder") grids or grids with a unique center mark should be used. The area of the grid square openings must be determined either by using the TEM at a calibrated magnification low enough to measure the sides of the opening, or with phase contrast microscopy at a calibrated magnification (usually 400X). If the measurements are made by TEM, one grid opening per grid is measured. For measurement by phase contrast, 20 grid openings are measured on each of 20 grids and the average size calculated. The TEM grids are first placed on a piece of lens tissue or filter paper so that individual grids can be picked up with forceps. Grids should be placed on pieces of filter paper or on individual screens and placed in the Jaffe washer. Three or more grids should be prepared from each sample.

11.21 Using a clean curved scalpel blade, cut 3 mm square pieces of the carbon coated filter from the glass slide. The point of the scalpel should be placed on the filter and a rocking motion used to cut the 3 mm square segments. Squares should be selected from the center of the filter and at two points between the outer periphery of the active surface and the center. The excised filter segments are placed carbon-side up on the grids. A map of the Jaffe washer should be drawn to keep track of the samples. Place the lid on the Jaffe washer and allow to stand until the filter is adequately dissolved (several hours).

11.22 Remove the grids from the Jaffe washer and allow to dry thoroughly before placing them in marked grid storage boxes (or other suitable containers).

11.23 Analyze the samples by TEM at an accelerating voltage of 80 to 120 kV and a screen magnification of 10,000 to 20,000X.

a. Use at least three grids from each filter to obtain the necessary number of grid openings or structures to reach the required analytical sensitivity.

b. Carefully load the grid into the sample holder. Orient the grid so that the grid bars are parallel and perpendicular to the long axis of the holder. This orientation will align the grid bars with the X and Y axes of the specimen translation controls.

c. Scan the grid at a magnification of 250 to 1000X to determine its suitability for analysis. Reject the grid if:

1. Less than 70% of the grid openings covered by the replica are intact.

2. The replica is folded or doubled.

3. The replica is too dark or has obviously visible filter structure because of incomplete dissolution.

4. The grid is too heavily loaded to obtain an accurate count.

5. The distribution of structures on the grid is obviously not uniform.

d. Reject individual grid openings with greater than 5% of their areas occupied by holes or tears in the carbon film. Reject openings with $\geq 25\%$ covered by particulates.

11.24 Examine the grid openings at 10-20,000X magnification.

11.25 Record the length and width of any grouping of particles in which an asbestos fiber with an aspect ratio $\geq 3:1$ and a length greater than 10 μm is detected.

11.26 Asbestos structures will be classified as fibers according to the following rules.

a. Fiber. A structure having a minimum length greater than 10 μm and an aspect ratio (length to width) of 3:1 or greater and substantially parallel sides without rounded ends.

b. Count an asbestos bundle $>10 \mu\text{m}$ long as a single fiber. Assign a length equal to the maximum length of any fiber within the bundle. If the bundle has stepped sides assign a width equal to an estimate of the mean width of the bundle.

c. Count a matrix as a single fiber if it contains a fiber or fibers, meeting the length and aspect ratio requirements, with one free end and the other end embedded in or hidden by a

particulate. If two ends are visible which appear to be the ends of a single fiber, the distance between the two ends is measured. If only one end of the fiber is visible, the fiber will be assigned a length equal to twice its visible length, except where this would place the concealed end outside of the particle. In this case, the length will be recorded as the visible length plus the diameter of the portion of the particle at the point of the fiber intersection. If the structure is too complex to be dealt with in this manner, record the overall dimensions of the structure but do not include it in the fiber count.

d. Count and record as single fibers the individual fibers visible with a cluster as long as they meet the fiber definition. If the aggregate is too complex, record the overall dimensions but do not include it in the fiber count.

e. Fibers which intersect the top and left sides of the grid opening are counted and recorded as twice their visible length. Fibers intersecting the bottom and right sides of the grid opening are not recorded.

f. Count only one end of the fiber to avoid the possibility of counting a single fiber more than once.

11.27 Structures classified as chrysotile must first be examined by SAED. If the characteristic chrysotile ED pattern is observed, the fiber will be counted. If no pattern is observed, or the pattern is not distinctive, the fiber must be examined by quantitative EDXA. If EDXA is characteristic of chrysotile, the fiber will be counted. Chrysotile fibers identified by morphology alone can be recorded but not counted towards the regulatory limit. The analysts's count sheet must indicate the method used to verify identity. A modified version of the AHERA count sheet may be used, which has columns to check off the method of identification.

11.28 Structures which are suspected to be an amphibole must first be examined by SAED. If a random orientation ED pattern with a 0.53 nm layer spacing is obtained, the fiber should be analyzed by EDXA. If the elements and peak ratios of the spectra correspond to those of a known amphibole, the fiber will be counted. If the random orientation ED pattern cannot be obtained, is incomplete or is not recognizable as a **non**-amphibole pattern, but an EDXA spectrum corresponding to a known amphibole is obtained, the fiber will be counted. Only fibers classified in this manner or by single or double zone axis SAED can be included in the regulated fiber count. The count sheet should indicate method used for identification.

11.29 Record both a typical SAED pattern and x-ray spectrum for each type of asbestos observed for each set of samples from the same source, or a minimum of every fifth sample analyzed.

Record the micrograph number on the count sheet and attach the x-ray spectrum to the back of the count sheet. If the x-ray spectrum is stored on disk, record the file and disk number on the count sheet.

11.30 Record NSD when no structures are detected in the grid opening.

11.31 Stopping Rules. Counting can be stopped at the completion of the grid opening in which an analytical sensitivity of 0.2 MFL is reached, or at the completion of the grid opening which contains the 100th structure, whichever occurs first. A minimum of 4 grid openings must be analyzed even if this results in the counting of more than 100 asbestos fibers over 10 μ m in length.

11.32 The grid openings examined must be drawn approximately equally from the three grids used in the analysis.

11.33 After completion of analysis, remove the grid from the microscope and replace in the labelled specimen storage box. Sample grids must be stored for a minimum of three years from the date of analysis. Sample filters may also be archived if desired.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Calculation of results. The concentration of asbestos in a given sample is calculated using the following formula:

$$\frac{\text{no str} \times \text{efa} \times R_D}{GO \times GOA \times V \times 1000}$$

where--

no str = number of asbestos fibers counted

efa = effective filter area of the sampling filter in mm^2

R_D = dilution ratio of original sample (if applicable)

GO = number of grid openings counted

GOA = area of grid openings in μm^2

V = original volume of sample filtered in ml.

12.2 The following information must be reported for each sample analyzed:

- a. Mean concentration of asbestos in million fibers per liter.
- b. Upper and lower 95% confidence limits on the mean concentration.
- c. Aliquot used for analysis and dilution factor (if any).
- d. Effective filter area.
- e. Total area of filter examined.
- f. Number of asbestos structures counted.
- g. Analytical sensitivity.
- h. Copies of the TEM count sheet, if requested.
- i. Number of structures which were too complex to classify, and number of suspected chrysotile and amphibole fibers which could not be positively identified.

13.0 METHOD PERFORMANCE

13.1 Limitations of accuracy can result from errors in identification of asbestos structures. Complete identification of every fiber is not possible due to both instrumental limitations and the nature of some of the fibers.

a. The requirement for a calibrated SAED pattern eliminates the possibility of an incorrect identification of chrysotile fibers. However, it is not always possible to obtain a satisfactory diffraction pattern from every fiber. 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 SAED pattern (1).

b. Complete identification of all amphibole fibers is not practical due to instrumental factors, the nature of some of the fibers, and limitations of time and cost. Particles of a number of other minerals having compositions similar to those of some amphiboles could be mistakenly classified as amphibole when zone axis SAED is not used. However, quantitative EDXA measurements on all fibers as support for the random orientation SAED patterns makes misidentification unlikely. The possibility of

misidentification is further reduced with increasing aspect ratio, since many of the minerals with which amphibole may be confused do not display its prominent cleavage parallel to the c-axis (1).

13.2 Limitations of accuracy can also result from the overlapping of structures by other (nonasbestos) particulates.

13.3 Inadequate dispersion of fibers can occur if enough organic contaminants were present in the original sample to cause adhesion of the fibers to the container walls or each other.

13.4 Contamination of the aliquots by asbestos during preparation in the laboratory can lead to inaccuracy of results. This is a particular problem with chrysotile, and should be carefully monitored by preparation of blank samples.

13.5 This method has not yet been subjected to an inter-laboratory test round. Precision measurements for EPA intra-laboratory comparisons of results from multiple operators using three TEMs produced a relative standard deviation (RSD) of 26.5% for MCE filters and 25.5% for PC filters for fibers over 10µm in length in standard samples. For similar methods, precision measurements for intra-laboratory comparisons have been found to have an RSD of 13 to 22 percent for standard and environmental water samples, with an RSD of 8.4 to 29 percent for inter-laboratory comparisons (1). Statistical formulae for the establishment of confidence limits on the laboratory results can be found in Chatfield and Dillon (1). An earlier study found an inter-laboratory reproducibility of 25 to 50 percent in standard samples (6). Accuracy measurements from inter- and intra-laboratory studies have demonstrated an RSD of 17 percent for standard chrysotile suspensions and an RSD of 16 percent for standard crocidolite suspensions (1).

13.6 The detection limit will depend upon the concentration of asbestos in the original sample and the constraints of time and cost of analysis. The detection limit can be improved by increasing the amount of water filtered, increasing the number of grid openings counted or decreasing the size of the filter used (when practical). Samples containing a high level of particulates will necessarily have a higher detection limit.

14.0 POLLUTION PREVENTION

14.1 It is the laboratory's responsibility to comply with all federal, state and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Also, compliance is required with any sewage discharge permits and regulations. For further

information on waste management, consult "The Waste Management Manual for Laboratory Personnel", available from the American Chemical Society.

15.0 REFERENCES

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APPENDIX 1

OZONE-ULTRAVIOLET OXIDATION TECHNIQUE

1. Assemble and operate the oxidation apparatus under an exhaust hood.
2. Place each sample bottle in the ultrasonic bath for 15 minutes.
3. Mark the level of liquid in each sample bottle using a waterproof marker.
4. Wash a quartz pipet thoroughly and attach to the ozone supply.
5. Suspend the pipet in the sample so that the tip is close to the bottom of the bottle.
6. Wash the UV lamp and immerse in the sample prior to turning the power supply on.
7. At an ozone concentration of 4% in oxygen, adjust flow to approximately 1 liter per minute. Treat for 3 hours. At other ozone concentrations, adjust the oxidation time so that each sample receives about 10g of ozone. Gas flow should be sufficient to produce a mixing action without splashing sample out of the bottle.
8. When oxidation is complete, remove the UV lamp and quartz pipet, recap the bottle and sonicate for 15 minutes.
9. If the water level has fallen, add known fiber-free water to bring it back to the original level marked on the bottle. Sonicate for 15 minutes.
10. Proceed with removal of aliquot and filtration immediately.



Test Method

Method for the Determination of Asbestos in Bulk Building Materials



EPA/600/R-93/116
July 1993

TEST METHOD
METHOD FOR THE DETERMINATION OF ASBESTOS
IN BULK BUILDING MATERIALS

by

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DISCLAIMER

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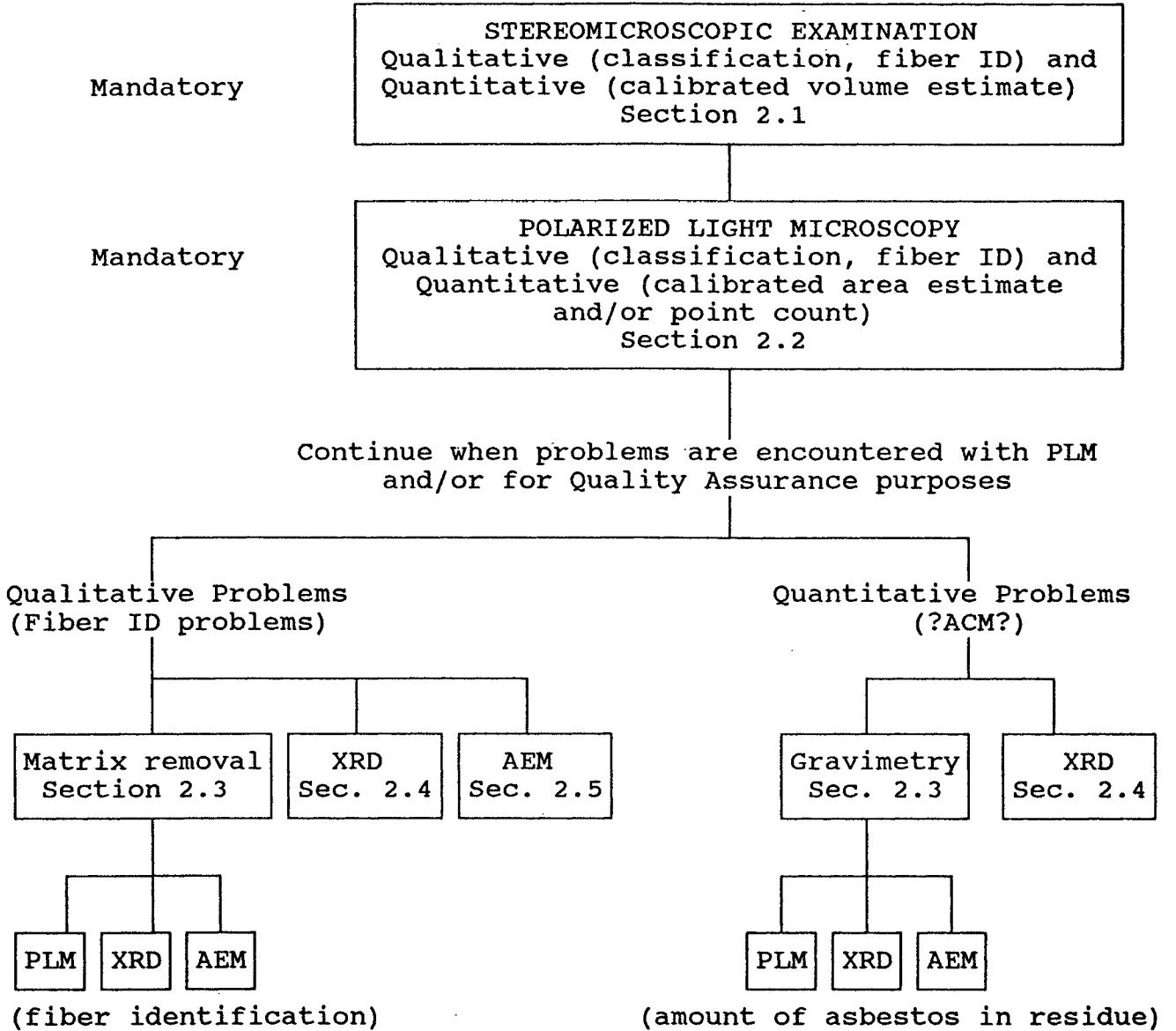
1.0 INTRODUCTION

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 counting³ and the interpretation of point count data⁵ 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 ($< 0.25 \mu\text{m}$ 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 Users¹⁰ 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, $\alpha(n \perp)$, 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 are not measured 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 the true extinction angle; true α will be the minimum 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 maximum 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 (~ 15 seconds) if using the blender technique so as to preclude a significant reduction in fiber size. The use of large cover slips (22x30mm) allows for large subsamples to be analyzed. Each slide should be checked to ensure that the subsample is representative, uniformly dispersed, and loaded in a way so as not to be dominated by superimposed (overlapping) particles.

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

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

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

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

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

TABLE 2-2. OPTICAL PROPERTIES OF ASBESTOS FIBERS

Mineral	Morphology and Color ¹	Refractive Indices ²		Birefringence ⁶	Extinction	Sign of Elongation
		α	γ^5			
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 1.625-1.655 1.604-1.612 1.627-1.635 1.599-1.612 1.625-1.637 1.6063 ⁷ 1.6343 ⁷ Actinolite 1.600-1.628 1.625-1.655 1.612-1.668 1.635-1.688 1.613-1.628 1.638-1.655 1.6126 ⁷ 1.6393 ⁷		0.017-0.028 0.017-0.028	Parallel and oblique (up to 21°); Composite fibers show parallel extinction.	+ (length slow)

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

⁵|| to fiber length, except ⊥ to fiber length for crocidolite only.

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

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

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

⁷± 0.0007

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

TABLE 2-3. TYPICAL CENTRAL STOP DISPERSION STAINING COLORS¹

Mineral	Cargille® RI Liquid	n_{\parallel}	n_{\perp}
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

¹Modified from reference 16

TABLE 2-4. OPTICAL PROPERTIES OF MAN-MADE TEXTILE FIBERS^{1,2}

Fiber Type	n_{\parallel}	n_{\perp}	$n_{\parallel} - n_{\perp}$	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	+

¹Modified from reference 17

²Refractive indices for specific fibers; other fibers may vary

TABLE 2-5. OPTICAL PROPERTIES OF SELECTED FIBERS¹

FIBER TYPE	MORPHOLOGY	REFRACTIVE INDICES	BIREFRINGENCE (n - n _⊥)	EXTINCTION ANGLE	SIGN OF ELONGATION	DISPERSION STAINING COLORS
Paper (Cellulose)	Tapered, flat ribbons	n ~ 1.580 n _⊥ ~ 1.530	High (0.05)	Parallel and incomplete	+	in 1.550HD n : yellow (λ ₀ 's < 400nm) n _⊥ : pale blue (λ ₀ 's > 700nm)
Olefin (polyethylene)	Filaments or shredded like chrysotile	n ~ 1.556 n _⊥ ~ 1.512	Moderate (0.044)	Parallel	+	in 1.550HD n : yellow to magenta (λ ₀ 's = 440-540nm) n _⊥ : pale blue (λ ₀ 's > 700nm)
Brucite (nematite)	Straight fibers	n ~ 1.560-1.590 n _⊥ ~ 1.580-1.600	Moderate (0.012-0.020)	Usually parallel	- occasionally +	in 1.550HD n : golden yellow (λ ₀ 's 440-460nm) n _⊥ : yellow (λ ₀ 's 400-440nm)
Heated amosite	Similar to unheated, (brittle and shorter) pleochroic: n -dark brown n _⊥ yellow	n and n _⊥ > 1.700 ²	High (> 0.05)	Usually parallel	+	in 1.680HD n & n _⊥ : 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 ~ 1.630 n _⊥ ~ 1.632 n _⊥ also 1.610	Moderate to low (0.018 to 0.002)	Parallel and oblique	+ and -	in 1.605HD n & n _⊥ : yellow to pale yellow (λ ₀ 's < 460nm)
Fibrous talc	Thin cleavage ribbons and wavy fibers	n ~ 1.60 n _⊥ ~ 1.54	High (0.06)	Parallel and oblique	+	in 1.550HD n : pale yellow (λ ₀ 's < 400nm) n _⊥ : pale blue (λ ₀ 's > 660nm)

¹From reference 19

²From references 13, 14, and 15

2.2.5.2.3 Microscope Alignment

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

- 1) the privileged directions of the substage polarizer and the analyzer are at 90° to one another and are represented by the ocular cross-lines;
- 2) the compensator plate's privileged vibration directions are 45° to the privileged directions of the polarizer and analyzer;
- 3) the objectives are centered with respect to stage rotation; and,
- 4) the substage condenser and iris diaphragm are centered in the optic axis.

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

2.2.6 References

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2.3 Gravimetry

2.3.1 Principle and Applicability

Many components of bulk building materials, specifically binder components, can be selectively removed using appropriate solvents or, in the case of some organics, by ashing. The removal of these components serves the following purposes:

- 1) to isolate asbestos from the sample, allowing its weight to be determined;
- 2) to concentrate asbestos and therefore lower the detection limit in the total sample;
- 3) to aid in the detection and identification of fibrous components; and,
- 4) to remove organic (ashable) fibers which are optically similar to asbestos.

Common binder materials which are removed easily using the techniques described include: 1) calcite, gypsum, magnesite, brucite, bassanite, portlandite, and dolomite, using hydrochloric acid, and 2) vinyl, cellulose, and other organic components, by ashing. The removal of the binder components results in a residue containing asbestos, if initially present, and any other non-soluble or non-ashable components which were present in the original sample. Unless the procedures employed result in the loss of some asbestos, the weight percent of the residue is the upper limit for the weight percent of asbestos in the sample.

This section describes the procedure for removing acid-soluble and ashable components, and for determining the weight percent of the residue. However, the acid dissolution and ashing techniques can be used without the accompanying weight measurements to either liberate or clean fibers to aid in qualitative PLM or AEM analyses.

This technique is not an identification technique. Other methods, such as PLM, XRD, or AEM must be used to determine the identity of the components. A description of the suggested apparatus, reagents, etc. needed for the techniques described is included in Appendix B.

2.3.2 Interferences

Any components which cannot be 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) **Ultrasonicate 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.

$$\text{wt\% asbestos in sample} = \% \text{ asbestos in residue} \times \text{wt\% residue} \div 100$$

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:

$$A = \text{wt\% residue from ashing} = 70\%$$

$$B = \text{wt\% residue from HCl} = 20\%$$

$$C = \text{wt\% of asbestos in HCl residue} = 50\%$$

$$\text{wt\% asbestos after HCl dissolution} = B \times C \div 100 = 20 \times 50 \div 100 = 10\%$$

$$\text{wt\% asbestos in floor tile} = (B \times C \div 100) \times A \div 100 = 10 \times 70 \div 100 = 7\%$$

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.

$$\text{wt\% asbestos after HCl dissolution} = B \times C = 0.2 \times 0.5 = 0.1 \text{ (x 100 = 10\%)}$$

$$\text{wt\% asbestos in floor tile} = (B \times C) \times A = 0.1 \times 0.7 = 0.07 \text{ (x 100 = 7\%)}$$

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° 2 θ ; 1° 2 θ /min) qualitative XRD scan, and their diffraction patterns should be compared with standard reference powder

diffraction patterns³ 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 $\sim 10 \mu\text{m}$ 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 MINERALS¹

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)

<p>A. Insulation Materials</p> <p>Chrysotile Amosite Crocidolite *Rock wool *Slag wool *Fiber glass Gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) 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</p>	<p>B. Flooring Materials</p> <p>Calcite Dolomite Titanium Oxide Quartz Antigorite Chrysotile Anthophyllite</p> <p>Tremolite *Organic binders Talc Wollastonite</p>	<p>C. Spray Finishes or Paints</p> <p>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")</p>	<p>D. Cementitious Materials</p> <p>Chrysotile Amosite Crocidolite Micas Fiber glass Cellulose Animal hair Quartz Gypsum Calcite Dolomite Calcium silicates</p> <p>E. Roofing Materials</p> <p>Chrysotile Cellulose Fiber glass Mineral Wool Asphalt Quartz Talc Micas</p>
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* 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 (CaCO₃) 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 EPA¹⁹

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 $\sim 10 \mu\text{m}$. 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 $< 10 \mu\text{m}$.^{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 ($\sim 100 \mu\text{m}$) 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 method²⁶ 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 ($\sim 7.4 \text{ \AA}$) and amphibole ($8.2\text{-}8.5 \text{ \AA}$) minerals (see Table 2-7). The x-ray diffraction equipment should be optimized for intensity. A slow scanning speed of $1^\circ 2\theta/\text{min}$ 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 ($5^\circ\text{-}60^\circ 2\theta$; $1^\circ 2\theta/\text{min}$) 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 data³ 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 $10 \mu\text{m}$ 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 $0.02^\circ 2\theta$ 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_n , 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 \AA 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 \AA 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° :

$$\hat{I}_a = \frac{I_a}{I_r^\circ}, \quad \hat{I}_{Ag} = \frac{I_{Ag}}{I_r^\circ}, \quad \text{and} \quad \hat{I}_{Ag}^\circ = \frac{I_{Ag}^\circ}{I_r^\circ}$$

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, $\hat{I}_{\text{std}}^{\circ}$, 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 $\hat{I}_{\text{std}}^{\circ}$, on w . Poor reproducibility (± 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 $\hat{I}_{\text{std}}^{\circ}$ 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 $\hat{I}_{\text{Ag}}^{\circ}$, of the sample filter, calculate the transmittance, T , for each sample as follows:^{27,28}

$$T = \frac{\hat{I}_{\text{Ag}}}{\hat{I}_{\text{Ag}}^{\circ}}$$

Determine the correction factor, $f(T)$, for each sample according to the formula:

$$f(T) = \frac{-R(\ln T)}{1 - T^R}$$

where

$$R = \frac{\sin \theta_{Ag}}{\sin \theta_a}$$

θ_{Ag} = angular position of the measured silver peak (from Bragg's Law), and

θ_a = angular position of the diagnostic asbestos peak.

Calculate the weight, W_a , in micrograms, of the asbestos material analyzed for in each sample, using the absorption corrections:

$$W_a = \frac{\hat{I}_a f(t) - b}{m}$$

Calculate the percent composition, P_a , of each asbestos mineral analyzed for in the parent material, from the total sample weight, W_T , on the filter:

$$P_a = \frac{W_a (1 - .01L)}{W_T} \times 100$$

where

P_a = percent asbestos mineral in parent material;

W_a = mass of asbestos mineral on filter, in μg ;

W_T = total sample weight on filter, in μg ;

L = percent weight loss of parent material on ashing and/or acid treatment (see Section 2.4.5.4).

2.4.8 References

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2.5 Analytical Electron Microscopy

2.5.1 Applicability

Analytical electron microscopy (AEM) can often be a reliable method for the detection and positive identification of asbestos in some bulk building materials, both friable and nonfriable. The method is particularly applicable to bulk materials that contain a large amount of interfering materials that can be removed by ashing and/or dissolution and contain asbestos fibers that are not resolved by PLM techniques. Many floor tiles and plasters would be included in this type of sample. In combination with suitable specimen preparation techniques, the AEM method can also be used to quantify asbestos concentrations.

2.5.2 Range

The range is dependent on the type of bulk material being analyzed. The upper detection limit is 100%, and the lower detection limit can be as low as 0.0001% depending on the extent to which interfering materials can be separated during the preparation of AEM

specimens, the sophistication of the AEM preparation, and the amount of labor expended on AEM examination.

2.5.3 Interferences

The presence of a large amount of binder/matrix materials associated with fibers can make it difficult to positively identify fibers as asbestos. The portion of the fiber examined by either electron diffraction or energy dispersive x-ray analysis (EDXA) must be free of binder/matrix materials.

2.5.4 Precision and Accuracy

The precision and accuracy of the method have not been determined.

2.5.5 Procedures

The procedures for AEM specimen preparation depend on the data required. In analysis of floor tiles, the weighed residue after removal of the matrix components (see Section 2.3, Gravimetry) is often mostly asbestos, and the task is primarily to identify the fibers. In this situation the proportion of asbestos in the residue can be estimated by AEM and this estimate can be used to refine the gravimetric result. For many floor tiles, the final result is not very sensitive to errors in this estimation because the proportion of asbestos in the residue is very high. For samples in which this is not the case, precise measurements can be made using a quantitative AEM preparation, in which each grid opening of the specimen grid corresponds to a known weight of the original sample or of a concentrate derived from the original sample. Asbestos fibers on these grids are then identified and measured, using a fiber counting protocol which is directed towards a precise determination of mass concentration. This latter procedure is suitable for samples of low asbestos concentration, or for those in which it is not possible to remove a large proportion of the matrix material.

2.5.5.1 AEM Specimen Preparation for Semi-Quantitative Evaluation

The residual material from any ashing or dissolution procedures (see Section 2.3) used (usually trapped on a membrane filter) should be placed in a small volume of ethanol or another solvent such as acetone or isopropyl alcohol, in a disposable beaker, and dispersed

by treatment in an ultrasonic bath. A small volume of this suspension (approximately $3\mu\text{l}$) 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 $0.22\ \mu\text{m}$ pore-size MCE or $0.2\ \mu\text{m}$ pore-size PC filters, using filtration techniques as described for analysis of water samples.¹ In order to obtain filters of appropriate particulate loading for AEM analysis, it may be necessary to perform serial dilutions of the initial dispersion. TEM grids should then be prepared from appropriately-loaded filters, using the standard methods.²

Determination of the mass concentration of asbestos on the TEM grids requires a different fiber counting protocol than that usually used for determination of numerical fiber concentrations. Initially, the grids should be scanned to determine the dimensions of the largest asbestos fiber or fiber bundle on the specimens. The volume of this fiber or bundle should be calculated. The magnification of the AEM should be set at a value for which the length of this fiber or bundle just fills the fluorescent screen. Asbestos fiber counting should then be continued at this magnification. The count should be terminated when the volume of the initial large fiber or bundle represents less than about 5% of the integrated volume of all asbestos fibers detected. This counting strategy ensures that the fiber counting effort is directed toward those fibers which contribute most to the mass, and permits a precise mass concentration value to be obtained.

2.5.5.2.1 Identification

To document the positive identification of asbestos in a sample, the analyst should record the following physical properties: morphology data, electron diffraction data, EDXA data, and any other distinguishing characteristics observed. For fibrous structures identified as nonasbestos, the unique physical property or properties that differentiate the material from asbestos should be recorded.

The purpose of the identification data collected is to prevent or limit false negatives and false positives. This can be accomplished by having a system for measuring and recording the d-spacings and symmetry of the diffraction patterns, determining the relative abundance of the elements detected by EDXA, and comparing these results to reference data. The laboratory should have a set of reference asbestos materials from which a set of reference diffraction patterns and x-ray spectra have been developed. Also, the laboratory should have available reference data on the crystallography and chemical composition of minerals that might analytically interfere with asbestos.

2.5.6 References

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2.6 Other Methodologies

Additional analytical methods (e.g. Scanning Electron Microscopy) may be applicable for some bulk materials. However, the analyst should take care to recognize the limitations of any analytical method chosen. Conventional SEM, for example, cannot detect small diameter fibers ($\sim < 0.2\mu\text{m}$), 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 rate⁷ 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 not 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. Ryan⁸ suggests a minimum of 50 analyses before an attempt is made to establish control limits. However, for this analysis, consider setting "temporary" limits after accumulating 20-30 analyses of the sample.
- Include both prepared slides as well as bulk samples in your reference inventory.
- Make certain that sample quantities are sufficient to last, and that the act of sampling will not alter the composition of the reference sample.

Data on analytical variability can be obtained by having analysts repeat their analyses of samples and also by having different analysts analyze the same samples.

3.3 Interlaboratory Quality Control

The establishment and maintenance of an interlaboratory QC program is fundamental to continued assurance that the data produced within the laboratory are of consistent high quality. Intralaboratory programs may not be as sensitive to accuracy and precision error, especially if the control charts (see Section 3.2.2) for all analysts in the laboratory indicate small percent differences. A routine interlaboratory testing program will assist in the detection of internal bias and analyses may be performed more frequently than proficiency

testing. Arrangements should be made with at least two (preferably more) other laboratories that conduct asbestos identification by PLM. Samples (the number of which is left to the participating laboratories, but at least 4-10) representing the types of samples and matrices routinely submitted to the lab for analysis should be exchanged with sufficient frequency to determine intralaboratory bias. Both reference slides and bulk samples should be used. Results of the interlaboratory testing program should be evaluated by each of the participating laboratories and corrective actions, if needed, identified and implemented. Since quantitation problems are more pronounced at low concentrations ($\leq 5\%$), it would be prudent to include approximately 30-50% from this concentration range in the sample selection process.

3.4 Performance Audits

Performance audits are independent quantitative assessments of laboratory performance. These audits are similar to the interlaboratory QC programs established between several laboratories, but with a much larger cohort (the EPA Asbestos Bulk Sample Analysis Quality Assurance Program had as many as 1100 participating laboratories). Participation in this type of program permitted assessment of performance through the use of "consensus" test materials, and served to assist in assessing the bias relative to individual interlaboratory, as well as intralaboratory programs. Caution should be exercised in the use of "consensus" quantitation results, as they are likely to be significantly responsible for the propagation of high bias in visual estimates. The current NIST/NVLAP⁹ for bulk asbestos laboratories (PLM) does not use consensus quantitation results. Results are reported in weight percent with a 95% tolerance interval. The American Industrial Hygiene Association (AIHA)¹⁰ also conducts a proficiency testing program for bulk asbestos laboratories. Quantitation results for this program are derived from analyses by two reference laboratories and PLM, XRD and gravimetric analysis performed by Research Triangle Institute.

3.5 Systems Audits

Where performance audits are quantitative in nature, systems audits are qualitative. Systems audits are assessments of the laboratory quality system as specified in the Laboratory

Quality Assurance Manual. Such an audit might consist of an evaluation of some facet of the QA Manual, or the audit may be larger in scope. For example, the auditor might request specific laboratory data sheets which will be evaluated against written procedures for data recording in the laboratory. Or, the auditor might request air monitoring or contamination control data to review for frequency of sampling, analysis methodology, and/or corrective actions taken when problems were discovered. The audit report should reflect the nature of the audit as well as the audit results. Any recommendations for improvement should also be reflected in such a report.

3.6 References

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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.
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8. Ryan, T.P., **Statistical Techniques for Quality Improvement**, John Wiley & Sons, Inc., New York, 1989.
9. National Institute of Standards & Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP), Building 411, Room A124, Gaithersburg, MD 20899, telephone (301) 975-4016.
10. American Industrial Hygiene Association (AIHA), 2700 Prosperity Avenue, Suite 250, Fairfax, VA 22031, (703) 849-8888.

APPENDIX A

Glossary Of Terms

APPENDIX A. GLOSSARY OF TERMS

Accuracy - The degree of agreement of a measured value with the true or expected value.

Anisotropic - Refers to substances that have more than one refractive index (e.g. are birefringent), such as nonisometric crystals, oriented polymers, or strained isotropic substances.

Asbestiform (morphology) - Said of a mineral that is like asbestos, i.e., crystallized with the habit of asbestos. Some asbestiform minerals may lack the properties which make asbestos commercially valuable, such as long fiber length and high tensile strength. With the light microscope, the asbestiform habit is generally recognized by the following characteristics:

- Mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than $5\mu\text{m}$. 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 $5\mu\text{m}$. 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.

<u>Qualitative</u>	<u>Quantitative(N-n)</u>
none	0.00 or isotropic
low	≤ 0.010
moderate	0.011-0.050
high	> 0.050

Bulk Sample - A sample of building material taken for identification and quantitation of asbestos. Bulk building materials may include a wide variety of friable and nonfriable materials.

Bundle - Asbestos structure consisting of several fibers having a common axis of elongation.

Calibration Materials - Materials, such as known weight % standards, that assist in the calibration of microscopists in terms of ability to quantitate the asbestos content of bulk materials.

Color - The color of a particle or fiber when observed in plane polarized light.

Compensator - A device with known, fixed or variable retardation and vibration direction used for determining the degree of retardation (hence the thickness or value of birefringence) in an anisotropic specimen. It is also used to determine the sign of elongation of elongated materials. The most common compensator is the first-order red plate (530-550nm retardation).

Control Chart - A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.

Detection Limit - The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

Dispersion Staining (focal masking) - An optical means of imparting apparent or virtual color to transparent substances by the use of stops in the objective back focal plane; 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 $\pm 10^\circ\text{C}$, temperature at sample position calibrated to $\pm 10^\circ\text{C}$
- 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- μ m 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 (0.22 μ m pore size) or 0.2 μ m pore size polycarbonate filters
- MCE backing filters (5 μ m 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, 0.4 μ m 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:

$$\frac{W_a}{G_a} \times 100 = V_a$$

$$\frac{W_a + W_m}{G_a \quad G_m}$$

where:

- W_a = weight percent asbestos
- G_a = specific gravity of asbestos
- W_m = weight percent matrix
- G_m = specific gravity of matrix
- V_a = 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?

$$\begin{array}{l} W_a = 5\% \\ G_a = 2.6 \\ W_m = 95\% \\ G_m = 0.4 \end{array} \quad V_a = \frac{\frac{5}{2.6}}{\frac{5}{2.6} + \frac{95}{0.4}} \times 100 = 0.8\%$$

Conversely, to convert volume percent asbestos to equivalent weight percent, the following formula may be used.

$$\frac{(V_a)(G_a)}{(V_a)(G_a) + (V_m)(G_m)} \times 100 = W_a$$

V_m = 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?

$$\begin{array}{l} V_a = 2\% \\ G_a = 3.2 \\ V_m = 98\% \\ G_m = 0.9 \end{array} \quad W_a = \frac{(2)(3.2)}{(2)(3.2) + (98)(0.9)} \times 100 = 6.77\%$$

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.

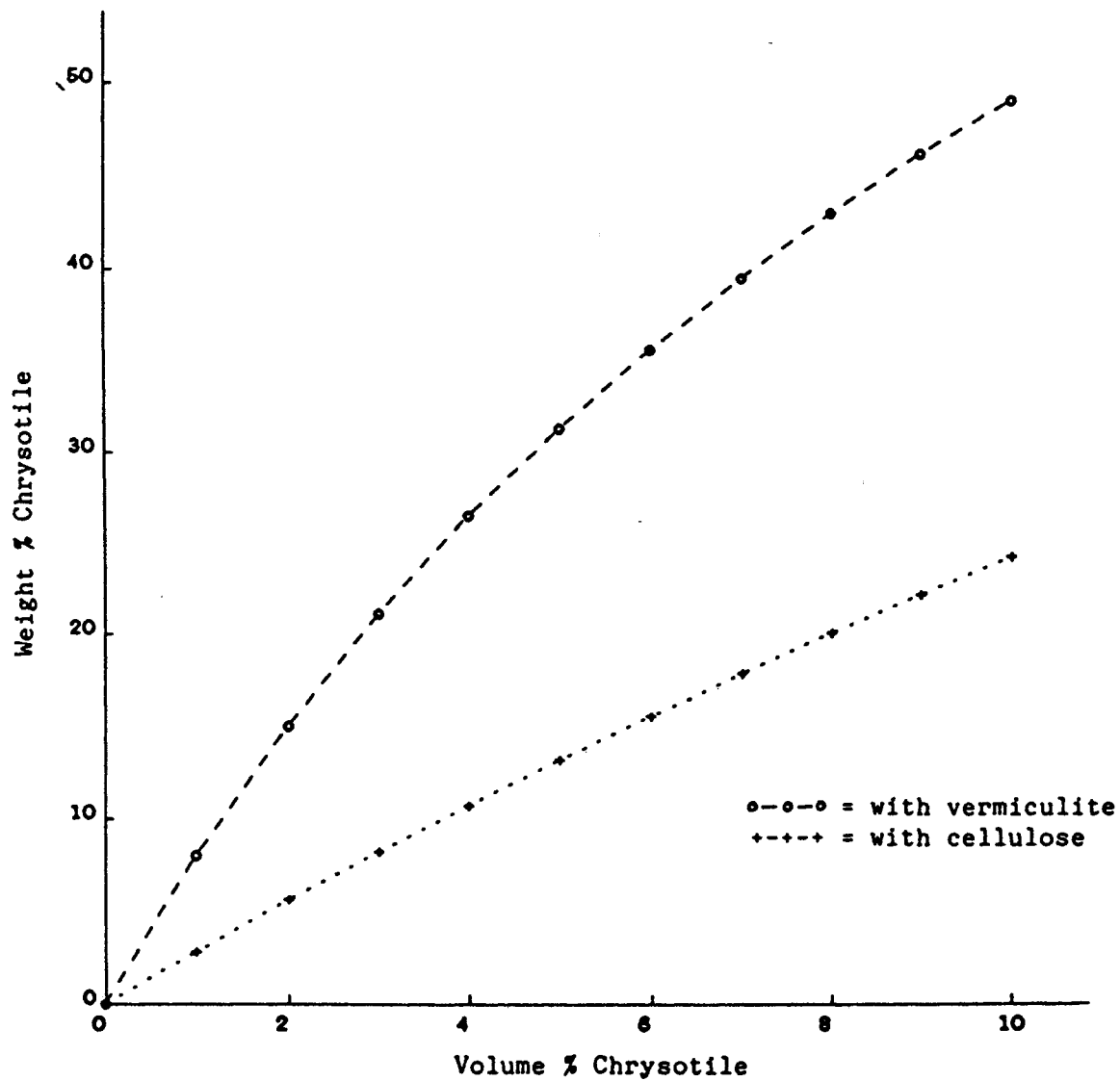


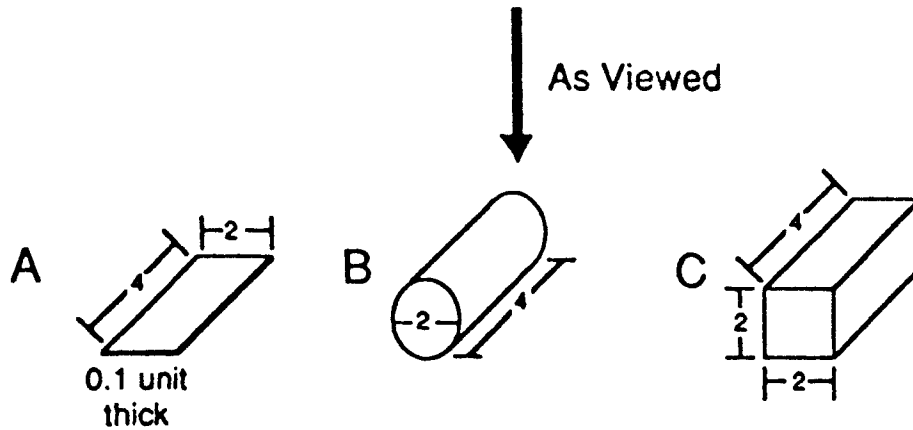
Figure C1. Relationship between volume % and weight % of chrysotile mixed with a)vermiculite and b) cellulose.

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:

$$\frac{V_a}{\frac{V_m}{T} + V_a} \times 100 = A_a$$

where:

- V_a = true volume percent asbestos
- V_m = true volume percent matrix
- T = thickness factor (mean size matrix particle/mean size asbestos fiber)
- A_a = 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?

$$\begin{array}{l} V_a = 5\% \\ V_m = 95\% \\ T = 10 \end{array} \quad A_a = \frac{5}{\frac{95}{10} + 5} \times 100 = 34.5\%$$

Conversely, to convert projected area percent asbestos to equivalent volume percent, the following formula may be used:

$$\frac{A_a}{T(A_m) + A_a} \times 100 = V_a$$

Where: A_m = 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?

$$\begin{aligned}
 A_m &= 81.4\% \\
 A_a &= 18.6\% \\
 T &= 6
 \end{aligned}
 \quad
 V_a = \frac{(18.6)}{6(81.4) + 18.6} \times 100 = 3.67\%$$

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?

$$\begin{aligned}
 V_a &= 3.67\% \\
 G_a &= 3.2 \\
 V_m &= 96.33\% \\
 G_m &= 2.5
 \end{aligned}
 \quad
 W_a = \frac{(3.67)(3.2)}{(3.67)(3.2) + (96.33)(2.5)} \times 100 = 4.7\%$$

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.)

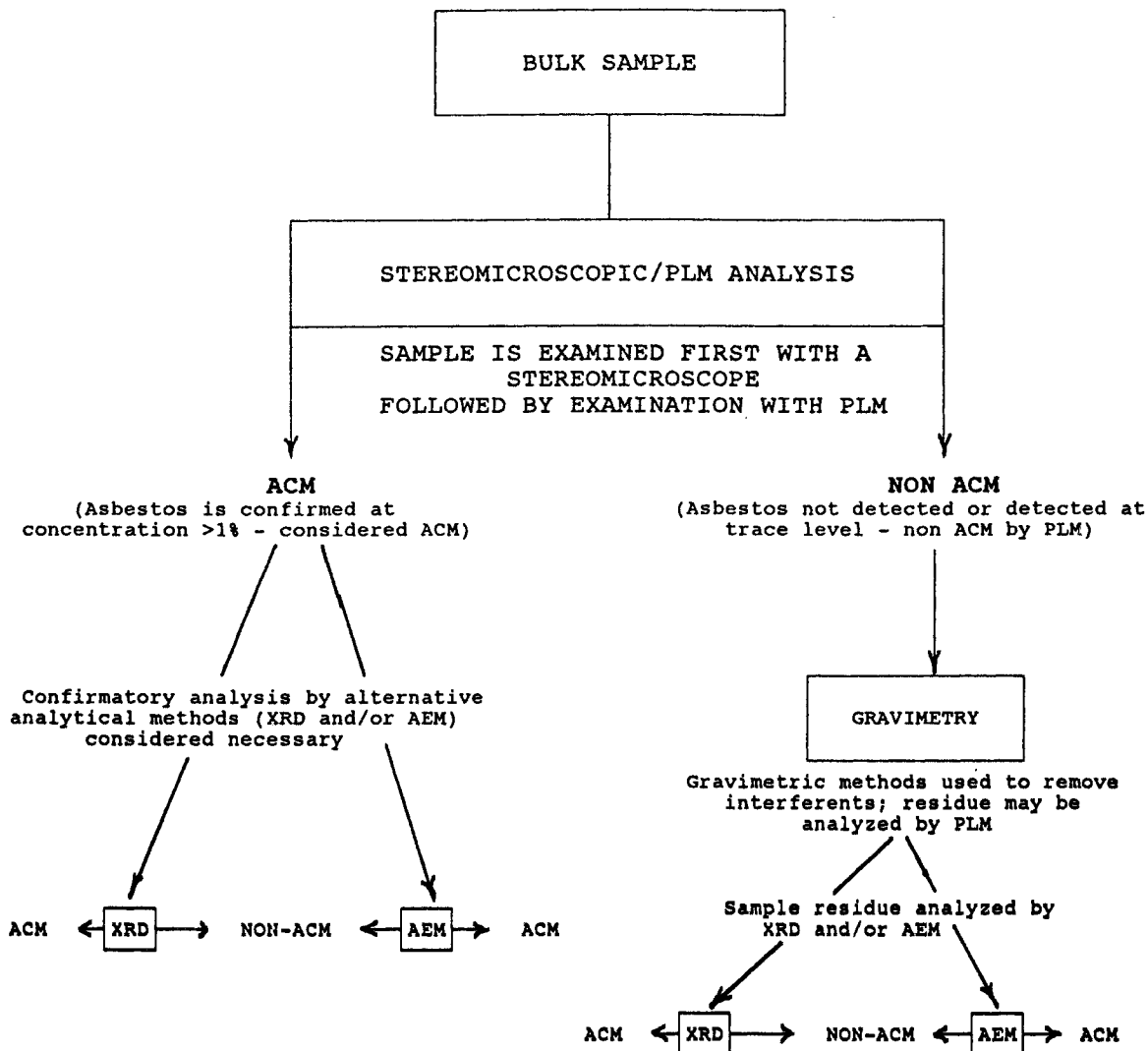
Materials characterized by interfering binder/matrix, low asbestos content, and/or small fiber size may require that additional sample treatment(s) and analysis be performed beyond routine PLM analysis. The sample treatment(s) required is(are) determined by the dominant nonasbestos sample components (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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