

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.<sup>5,13,14</sup> 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<sup>7,8</sup> or filter substrate (Ag) peak.<sup>5,6</sup>

#### 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  $\mu\text{m}$ .<sup>15</sup> 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.<sup>7,8,15</sup> 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.<sup>12,14</sup> 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.<sup>18</sup> have observed that in insulating matrices, chrysotile tends to break open into bundles more frequently than amphiboles. This results in a line-broadening effect with a resultant decrease in sensitivity. Unless this effect is the same for both standard and sample materials, the amount of chrysotile in the sample will be under-estimated by XRD analysis. To minimize this problem, it is recommended that standardized matrix reduction procedures be used for both sample and standard materials.

#### 2.4.4 Precision and Accuracy

Neither the precision nor accuracy of this method has been determined. The individual laboratory should obtain or prepare a set of calibration materials containing a range of asbestos weight percent concentrations in combination with a variety of matrix/binder materials. Calibration curves may be constructed for use in semi-quantitative analysis of bulk materials.

#### 2.4.5 Procedure

##### 2.4.5.1 Sampling

Samples taken for analysis of asbestos content should be collected as specified by EPA<sup>19</sup>

##### 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.<sup>20-23</sup> 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  $\mu\text{m}$ . 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<sup>®</sup>, 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.<sup>5</sup> Bulk samples of very large particle size may require grinding in two stages for full matrix reduction to  $< 10 \mu\text{m}$ .<sup>8,16</sup>

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  $550^\circ\text{C}$  and  $650^\circ\text{C}$ , with subsequent transformation to forsterite,<sup>24,25</sup> ashing temperatures should be kept below  $500^\circ\text{C}$ . Use of a muffle furnace is recommended. In all cases, calibration of the furnace is essential to ensure that a maximum ashing temperature of  $500^\circ\text{C}$  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 method<sup>26</sup> 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 data<sup>3</sup> 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  $2\theta$  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.<sup>6</sup> A thick-layer bulk method involving pelletizing the sample may be used for semi-quantitative analysis;<sup>7,8</sup> 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 Å silver peak following the procedure in step 12. Remove the filter from the holder, reverse it, and reattach it to the holder. Determine the net count for the unattenuated silver peak,  $I_{Ag}^\circ$ . Scan times may be less for measurement of silver peaks than for sample peaks; however, they should be constant throughout the analysis.
14. Normalize all raw, net intensities (to correct for instrument instabilities) by referencing them to an external standard (e.g., the 3.34 Å peak of an  $\alpha$ -quartz reference crystal). After each unknown is scanned, determine the net count,  $I_r^\circ$ , of the reference specimen following the procedure in step 12. Determine the normalized intensities by dividing the peak intensities by  $I_r^\circ$ :

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

## 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 ( $\pm 15$  percent RSD) at any given level indicates problems in the sample preparation technique, and a need for new standards. The data should fit a straight-line equation.

Determine the slope,  $m$ , of the calibration curve in counts/microgram. The intercept,  $b$ , of the line with the  $\hat{I}_{\text{std}}^{\circ}$  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,  $\hat{I}_{\text{Ag}}$  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:<sup>27,28</sup>

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

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

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

where

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

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

$\theta_a$  = 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  $\mu\text{g}$ ;

$W_T$  = total sample weight on filter, in  $\mu\text{g}$ ;

$L$  = 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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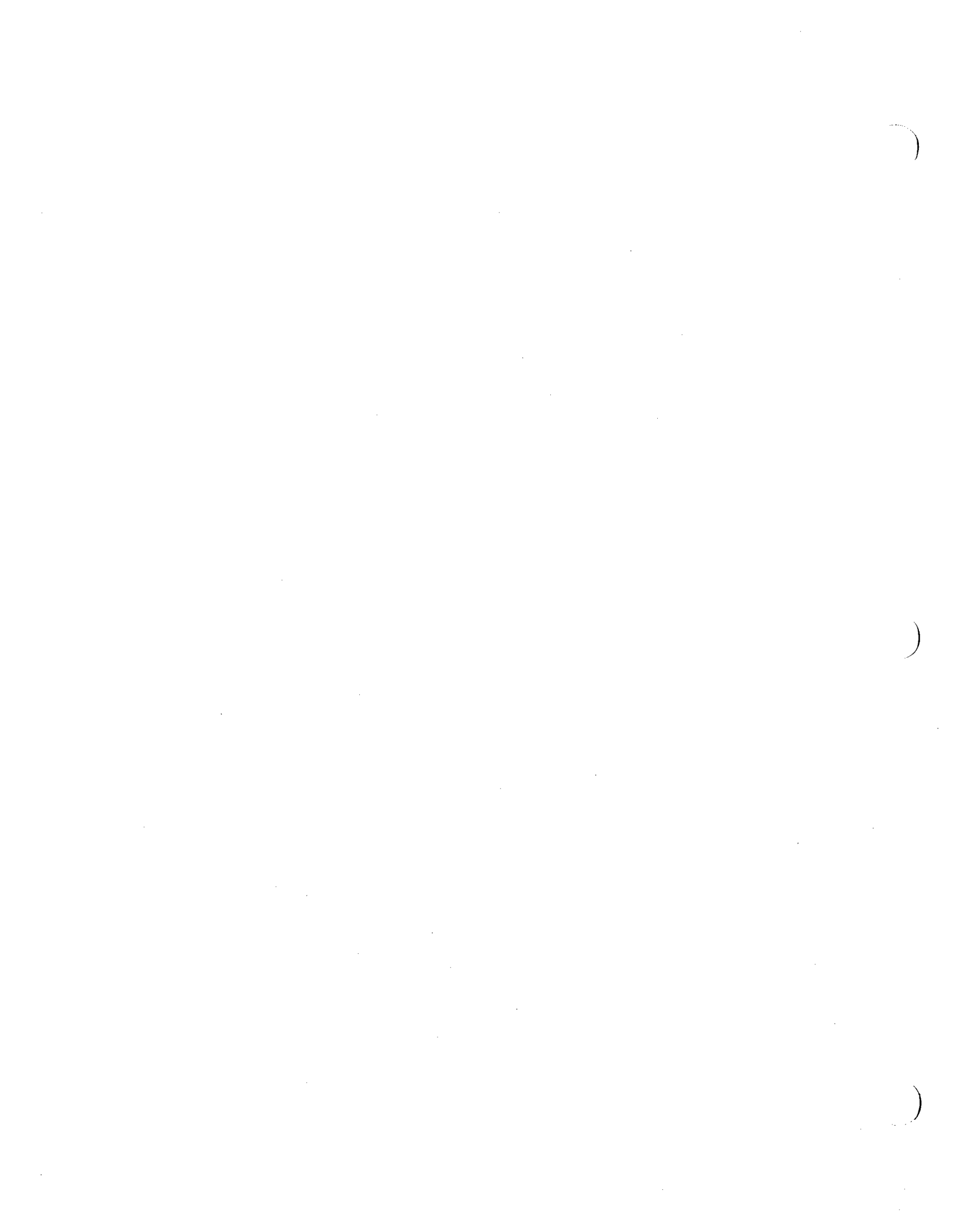
## 2.5 Analytical Electron Microscopy

### 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.<sup>1</sup> 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.<sup>2</sup>

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,<sup>1,2,3,4,5,6</sup> only several of the elements will be discussed here. Quality Assurance (QA) is comprised of Quality Control and Quality Assessment and is a system of activities designed to provide assurance that a product or service meets defined standards of quality.

The purpose of the Quality Assurance program is to minimize failures in the analysis of materials prior to submitting the results to the client. Failures in the analysis of asbestos materials include false positives, false negatives, and misidentification of asbestos types. False positives result from identification or quantitation errors. False negatives result from identification, detection, or quantitation errors.

For the stereomicroscopic and PLM techniques, the quality control procedures should characterize the accuracy and precision of both individual analysts and the techniques. Analysts should demonstrate their abilities on calibration materials, and also be checked routinely on the analysis of unknowns by comparison with results of a second analyst. The limitations of the stereomicroscopic and PLM techniques can be determined by using a second analytical technique, such as gravimetry, XRD, or AEM. For example, stereomicroscopic and PLM techniques can fail in the analysis of floor tiles because the asbestos fibers in the sample may be too small to be resolved by light microscopy. An XRD or AEM analysis is not subject to the same limitations, and may indicate the presence of asbestos in the sample.

The accuracy, precision, and detection limits of all analytical techniques described in this method are dependent on the type of sample (matrix components, texture, etc.), on the preparation of the sample (homogeneity, grain size, etc.), and the specifics of the method (number of point counts for PLM, mass of sample for gravimetry, counting time for XRD,

etc.). These should be kept in mind when designing quality control procedures and characterizing performance, and are variables that must be tracked in the quality assurance system.

### 3.1 General Considerations

#### 3.1.1 Training

Of paramount importance in the successful use of this or any other analytical method is the well-trained analyst. It is highly recommended that the analyst have completed course work in optical mineralogy on the collegiate level. That is not to say that others cannot successfully use this method, but the classification error rate<sup>7</sup> 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.<sup>1,2,3,4,5</sup> Several cautions are in order:

- Control charts are based on the assumption that the data are distributed normally. Using rational subgrouping, the means of the subgroups are approximately normally distributed, irrespective of the distribution of the individual values in the subgroups. Control charts for asbestos analysis are probably going to be based on individual measurements, not rational subgroups. Check the data for normality before proceeding with the use of control charts. Ryan<sup>8</sup> suggests a minimum of 50 analyses before an attempt is made to establish control limits. However, for this analysis, consider setting "temporary" limits after accumulating 20-30 analyses of the sample.
- Include both prepared slides as well as bulk samples in your reference inventory.
- Make certain that sample quantities are sufficient to last, and that the act of sampling will not alter the composition of the reference sample.

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

### 3.3 Interlaboratory Quality Control

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

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

### 3.4 Performance Audits

Performance audits are independent quantitative assessments of laboratory performance. These audits are similar to the interlaboratory QC programs established between several laboratories, but with a much larger cohort (the EPA Asbestos Bulk Sample Analysis Quality Assurance Program had as many as 1100 participating laboratories). Participation in this type of program permitted assessment of performance through the use of "consensus" test materials, and served to assist in assessing the bias relative to individual interlaboratory, as well as intralaboratory programs. Caution should be exercised in the use of "consensus" quantitation results, as they are likely to be significantly responsible for the propagation of high bias in visual estimates. The current NIST/NVLAP<sup>9</sup> 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)<sup>10</sup> also conducts a proficiency testing program for bulk asbestos laboratories. Quantitation results for this program are derived from analyses by two reference laboratories and PLM, XRD and gravimetric analysis performed by Research Triangle Institute.

### 3.5 Systems Audits

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

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

### 3.6 References

1. **Quality Assurance for Air Pollution Measurement Systems. Volume I, Principles.** EPA-600/9-76-005, March, 1976.
2. Juran, J. and F. Gryna, **Quality Planning Analysis**, 2nd edition, McGraw-Hill, Inc., 1980.
3. Taylor, J.R., **Quality Control Systems**, McGraw Hill, Inc., 1989.
4. Ratliff, T.A., **The Laboratory Quality Assurance System**, Van Nostrand Reinhold, 1990.
5. Taylor, J.K., **Quality Assurance of Chemical Measurements**, Lewis Publishers, 1987.
6. **Bulk Asbestos Handbook**, National Institute of Standards and Technology, National Voluntary Laboratory Accreditation Program, NISTIR 88-3879, October 1988.
7. Harvey, B.W., "Classification and Identification Error Tendencies in Bulk Insulation Proficiency Testing Materials," **American Environmental Laboratory**, 2(2), 4/90, pp. 8-14.
8. Ryan, T.P., **Statistical Techniques for Quality Improvement**, John Wiley & Sons, Inc., New York, 1989.
9. National Institute of Standards & Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP), Building 411, Room A124, Gaithersburg, MD 20899, telephone (301) 975-4016.
10. American Industrial Hygiene Association (AIHA), 2700 Prosperity Avenue, Suite 250, Fairfax, VA 22031, (703) 849-8888.



**APPENDIX A**

Glossary Of Terms

## APPENDIX A. GLOSSARY OF TERMS

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

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

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

- Mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than  $5\mu\text{m}$ . 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	$\leq 0.010$
moderate	0.011-0.050
high	$> 0.050$

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

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

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

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

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

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

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

**Dispersion Staining (focal masking)** - An optical means of imparting apparent or virtual color to transparent substances by the use of stops in the objective back focal plane; 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 ( $\lambda_0$ )** - The wavelength ( $\lambda_0$ ) 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.<sup>1,2,3</sup>

## **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 ( $n_p$ ), or functional equivalent. (optional)

In addition, the following equipment, materials and reagents are required or recommended.<sup>1</sup>

- 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  $\mu\text{m}$  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- $\mu$ 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:  $\alpha$ -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.<sup>3</sup>

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 $\mu$ m pore size) or 0.2 $\mu$ m pore size polycarbonate filters
- MCE backing filters (5 $\mu$ 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.<sup>1</sup> 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 $\mu$ 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.<sup>2</sup>

##### C4.1 Stereomicroscopic Analysis

Visual estimation of sample components using the stereomicroscope is in reality a comparison of the relative volumes of the components.<sup>3</sup> 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.<sup>4</sup> 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<sub>a</sub> = weight percent asbestos
- G<sub>a</sub> = specific gravity of asbestos
- W<sub>m</sub> = weight percent matrix
- G<sub>m</sub> = specific gravity of matrix
- V<sub>a</sub> = 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.

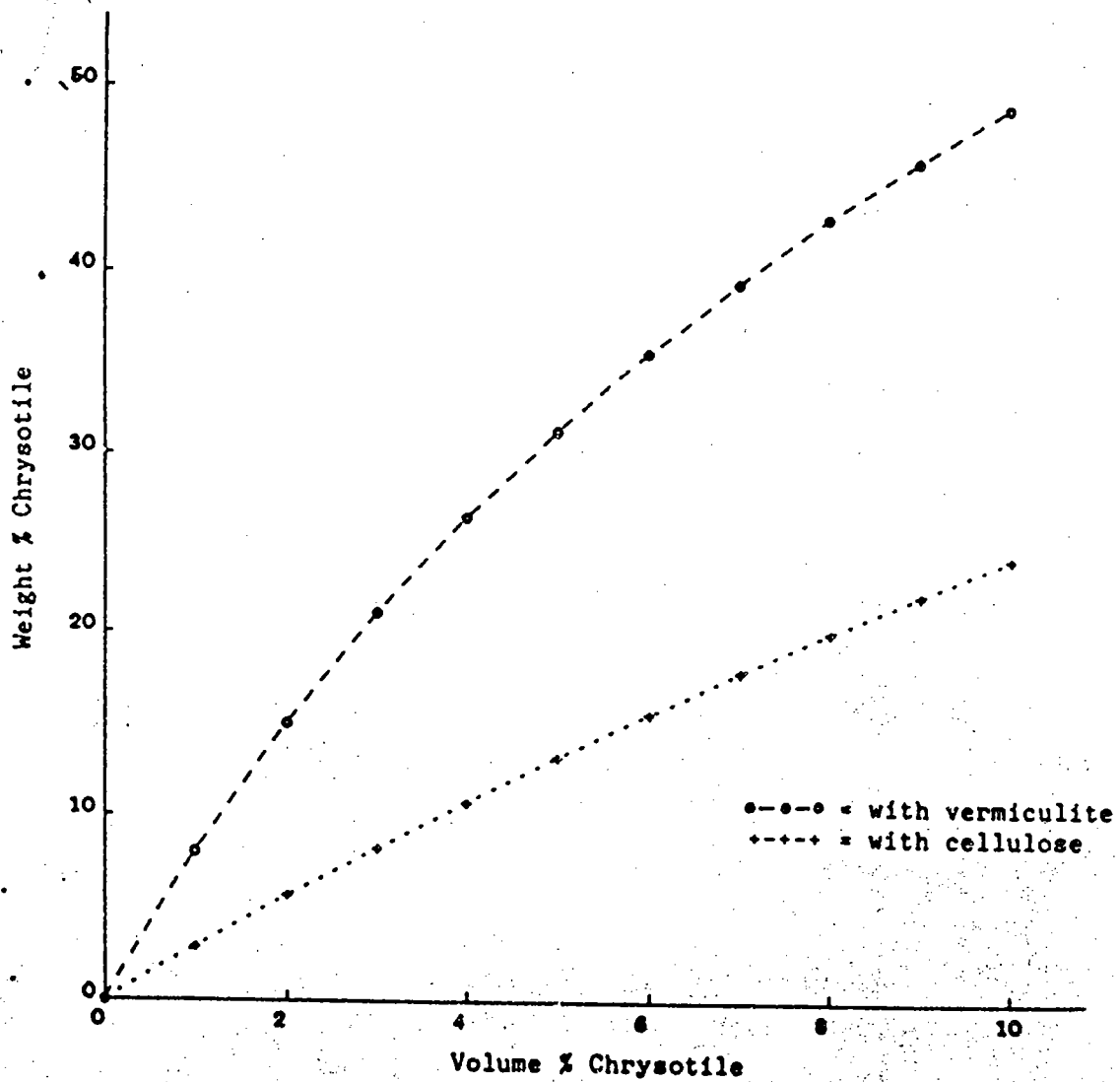


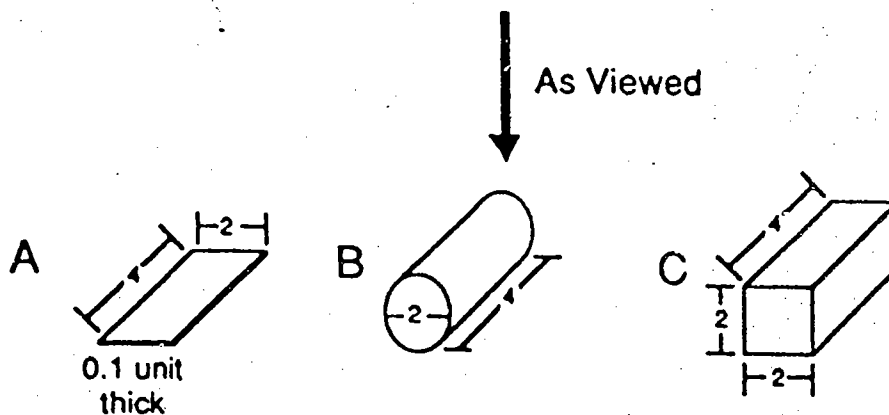
Figure C1. Relationship between volume % and weight % of chrysotile mixed with a)vermiculite and b) cellulose.

## 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<sub>a</sub> = true volume percent asbestos
- V<sub>m</sub> = true volume percent matrix
- T = thickness factor (mean size matrix particle/mean size asbestos fiber)
- A<sub>a</sub> = 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<sub>m</sub> = 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?

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

### 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.)

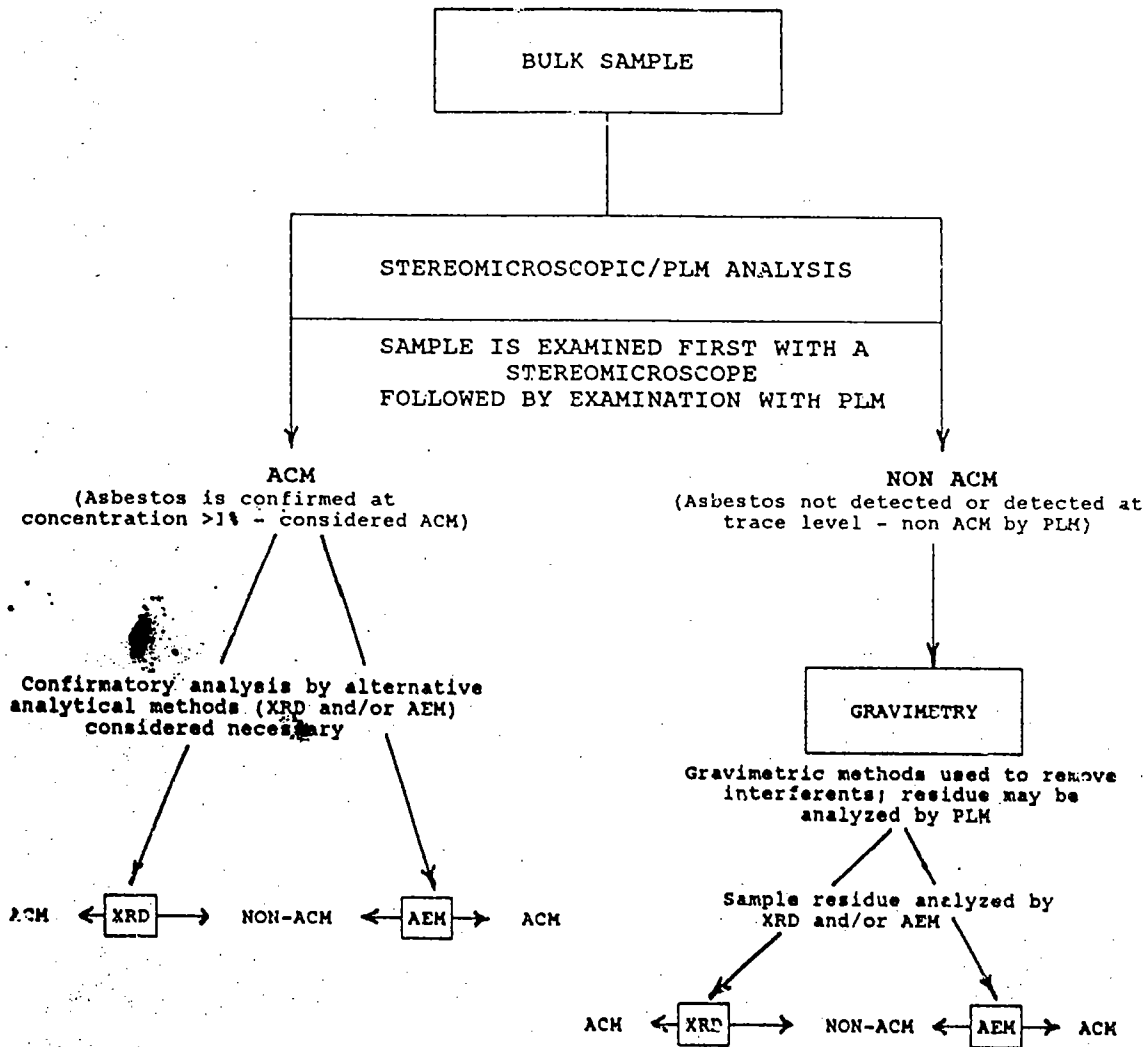
Materials characterized by interfering binder/matrix, low asbestos content, and/or small fiber size may require that additional sample treatment(s) and analysis be performed beyond routine PLM analysis. The sample treatment(s) required is(are) determined by the dominant nonasbestos sample components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