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Polarized-Light Microscope Method for Identifying and Quantitating Asbestos in Non-Friable Organically Bound Bulk Samples	11/22/06	1 of 25	198.6

1. **Introduction.** This is the method for the analysis of asbestos in Non-Friable Organically Bound (NOB) bulk materials by Polarized-Light Microscopy (PLM).

1.1. **Background**. Vinyl asbestos tiles (VAT) have been categorized by NESHAP (1990) as a Category I non-friable asbestos-containing material. As such, VAT's potential to release fibers under certain conditions was recognized. Recent studies reinforce this potential for releasability (McDonald 1991, Brzezowski 1990, Marxhausen and Shaffer 1991). VATs present one of the most difficult challenges to laboratories that analyze bulk samples for asbestos. Fiber sizes used in manufacture were often the "shorts" that were too small for other applications (Rajhans and Sullivan 1981, Cossette and Delvaux 1979). These were often further comminuted during mixing (Mullin 1988) so that many of the fibers were below the resolution capability of the light microscope. The addition of organic matrix materials, usually vinyl or asphalt, compounded the detection problem by coating these small fibers with opaque matter. Finally, mechanical abrasion during sample preparation can further reduce fiber size and inhibit detection.

1.2. **Objective**. Item 198.6 allows PLM analysis of Non-Friable Organically Bound Materials (NOB) with certain limitations. Laboratories that are certified to analyze friable bulk samples are not required to have the capability of analyzing NOBs. A laboratory may be certified to analyze NOBs by PLM, provided that they follow the gravimetric reduction preparation technique described in this method. Finally, reports of PLM analysis of NOBs that yield concentrations of 1% or less asbestos must report “Inconclusive” and include the disclaimer spelled out in Section 6.3.2.1.

1.3. **Definitions**

1.3.1. **Asbestos**. "Asbestos" refers to the asbestiform varieties of: chrysotile (serpentine); crocidolite (riebeckite); amosite (cummingtonite-grunerite); anthophyllite; tremolite; and actinolite (AHERA, 1987).

1.3.2. **Asbestos-Containing Materials**. "Asbestos-containing materials" (ACM) means any material or product that contains more than 1 percent asbestos (AHERA, 1987; NESHAP, 1990).

1.3.3. **Friable**. "Friable" materials are those materials that, when dry, may be crumbled, pulverized, or reduced to powder by hand pressure, and includes previously nonfriable material after such previously nonfriable material becomes damaged to the extent that when dry it may be crumbled, pulverized, or reduced to powder by hand pressure (AHERA, 1987).

1.3.4. **Vinyl Asbestos Tile**. This term (VAT) has been widely used in the asbestos analysis and abatement industry to refer to a variety of flooring products such as vinyl or asphalt asbestos floor tiles and resilient floor coverings.

1.3.5. **Non-Friable Organically Bound Materials**. This term (NOB) refers to a wide variety non-friable building materials embedded in flexible-to-rigid asphalt or vinyl matrices. This includes VAT, mastic, asphalt shingles, roofing materials, caulking and glazing, etc.

2. **Application**. The method outlined herein is applicable to Non-Friable bulk materials. NOBs shall be prepared by the gravimetric matrix reduction method. Asbestos content in gravimetrically reduced NOB samples is determined point-counting. Quantitative TEM is the **only** NOB method that can be used to report non-ACM results to clients (ELAP Item 198.4, Section 6.5).

3. **Equipment and Supplies** The following items shall be available for sample preparation and analysis in laboratories that analyze NOBs:

3.1. HEPA-ventilated, negative-pressure sample preparation work area. This can be a laminar-flow safety cabinet or a similar enclosure that draws all air from the enclosure through a HEPA filter. This should minimize cross contamination and maintain a safe work environment. A flow rate of at least 75 fpm shall be maintained at the opening.

3.2. Low-power (10-45X) stereobinocular microscope with external light source for gross examination.

3.3. Forceps, dissecting needles, probes, scalpel or razor blades, etc. for manipulating bulk sample.

3.4. Homogenization equipment:

3.4.1. Mortar and pestle.

3.4.2. At least one of the following:

3.4.2.1. Mini-blender (approximately 30 mL capacity).

3.4.2.2. Liquid-nitrogen mill.

3.4.2.3. Wiley mill.

3.5. Centrifuge.

3.6. Filtration apparatus for polycarbonate filters.

3.6.1. 0.4-µm pore polycarbonate filters.

3.6.2. Petri dishes and covers.

3.7. Muffle furnace capable of sustained operation at 500^{o}C.

3.7.1. Crucibles (bottom and lid) that can withstand 500^{o}C.

3.7.2. Instrument or materials capable of calibrating muffle furnace at 480^{o}C:

3.7.2.1. High-temperature thermometer with range to at least 500^{o}C and with readable subdivisions of 5^{o}C or less ***or***

3.7.2.2. Melting-point solids with capability of differentiating 5^{o}C differences between 400^{o}C and 500^{o}C ***or***

3.7.2.3. Independent potentiometer capable of differentiating 5^{o}C differences between 400^{o}C and 500^{o}C.

3.8. Concentrated HCl, reagent grade.

3.9. Surfactant (sodium metaphosphate or Aerosol OT).

3.10. Heat lamp, slide warmer or drying oven.

3.11. Ultrasonic bath.

3.12. Filtered (0.1-µm) distilled water.

3.13. Textbook or reference book on mineralogy or crystallography, e.g., McCrone 1980, McCrone 1988, Deer, Howie and Zussman, 1966.

3.14. Reference materials.

3.14.1. National Institute of Standards and Technology (NIST) SRM 1866a and SRM 1867:

3.14.1.1. Chrysotile.

3.14.1.2. Grunerite (Amosite).

3.14.1.3. Riebeckite (Crocidolite).

3.14.1.4. Glass Fiber.

3.14.1.5. Anthophyllite.

3.14.1.6. Tremolite.

3.14.1.7. Actinolite.

3.14.2. Verified quantitative standards. All laboratories must have at least 10 different VAT specimens that have been analyzed by an ELAP-certified TEM laboratory (own lab or outside lab) using the quantitative method outlined

herein. Raw gravimetric data and calculations used to determine percentages of each component (organic, carbonate, insoluble inorganic, and asbestos) must be provided by the outside laboratories and kept on file. Each of these reference specimens should initially exceed 50 cm^2 in area.

Sections of these VATs must be submitted blindly as routine samples as outlined in Section 8.2.3 to determine the accuracy and precision of the laboratory's current analytical capabilities.

3.14.2.1. Negative (non-ACM) standards. At least two negative standards shall be kept.

3.14.2.2. Positive (ACM) standards (low concentration). At least two positive standards with concentrations less than 10% shall be kept.

3.14.2.3. Positive (ACM) standards (high concentration). At least two positive standards with concentrations greater than 10% shall be kept.

3.14.2.4. At least four other different standards shall be kept.

3.14.3. Permanent mount of NIST amosite in n_D=1.680.

3.15. Microscope slides.

3.16. Cover slips.

3.17. Refractive index liquids:

3.17.1. n_D=1.550 high dispersion.

3.17.2. n_D=1.605 high dispersion.

3.17.3. n_D=1.630 high dispersion.

3.17.4. n_D=1.680.

3.17.5. n_D=1.700.

3.17.6. Series of n_D=1.49 through 1.72 in intervals less than or equal to 0.005. This full series is required because of the range of refractive indices exhibited by the different asbestos types in both their natural and altered (heated or acid-stressed) states. High-dispersion liquids may be substituted in the 1.49 through 1.63 range.

3.17.7. Calibration accessory for measuring refractive indices of refractive index liquids. These can be calibrated solids, e.g., glasses, or a refractometer capable of an accuracy of ±0.004.

3.17.8. Laboratory thermometer with range of 0° to 50° C and readability of

±1° C.

3.18. Marker for labeling slides.

3.19. Polarizing-light microscope equipped with the following:

3.19.1. Substage polarizer.

3.19.2. Analyzer capable of producing a completely black field when privileged direction is oriented perpendicular to that of the substage polarizer. Either the polarizer or analyzer shall be rotatable so that polars can be slightly uncrossed when necessary.

3.19.3. Port @ 45^{o} to analyzer for wave retardation plate.

3.19.4. 550 nm (first-order red) compensator plate.

3.19.5. 360^{o} graduated (in 1^{o} increments) rotating stage.

3.19.6. Illuminator and adjustable diaphragm.

3.19.7. The following objective lenses:

3.19.7.1. Dispersion-staining objective capable of central stop illumination with magnification of approximately 10X (optional).

3.19.7.2. Low-magnification objective (3.2 to 10X).

3.19.7.3. High-magnification, dry objective (30 to 50X).

3.19.8. Eyepiece(s) of at least 8X magnification containing a fixed cross-hair.

3.19.8.1 Chalkley point-count reticle (optional).

3.19.9. Focusable condenser with centerable iris diaphragm capable of completely eclipsing the back-focal-plane image of the central stop.

3.20. Beam balance with readability of 1 mg or less. (e.g., Fisher Model 711).

3.21. Analysis sheet with space for the following entries:

3.21.1. Analyst's signature.

3.21.2. Date of analysis.

3.21.3. Sample identification number.

3.21.4. Gross description of bulk sample (color, homogeneity, texture) and tentative identification of fibers by stereobinocular microscope.

3.21.5. Type of homogenization (if any).

3.21.6. Matrix reduction. This shall include ashing and acid steps used and amount of matrix removed during each step to the nearest mg.

3.21.6.1. Mass of original subsample.

3.21.6.2. Mass of post-ashed subsample.

3.21.6.3. Mass of post-acid subsample.

3.21.7. Entries for each asbestos type identified:

3.21.7.1. Morphology.

3.21.7.2. Refractive index (at $?_o$=589.5 nm) parallel to fiber length in specified n_D medium.

3.21.7.3. Refractive index (at $?_o$=589.5 nm) perpendicular to fiber length in specified n_D medium.

3.21.7.4. Sign of elongation.

3.21.7.5. Angle of fiber length extinction.

3.21.7.6. Pleochroism and color.

3.21.7.7. Birefringence.

3.21.7.8. Other observations.

3.21.8. Space for estimation of asbestos percentage. This shall include the number of asbestos vs. non-asbestos points counted.

3.21.9. Final results including:

3.21.9.1. Type(s) and percentage(s) of each asbestos type detected.

3.21.9.2. Total percentage of asbestos.

3.21.9.3. Type(s) and percentage(s) of non-asbestos fibers detected.

3.21.9.4. Percentage of non-fibrous material present.

3.21.9.5. Percentage of non-asbestos insoluble inorganic fraction.

3.21.9.6. Percentage of soluble inorganic fraction.

3.21.9.7. Percentage of organic fraction.

4. **Sample Preparation**. Careful sample preparation is a critical step in the analysis of NOBs. Detection of asbestos fibers used in VATs is often extremely difficult because of the small fibers used during manufacture, their subsequent mixing and coating with organic matrix (vinyl, asphalt, etc.), and potential comminution during

sample preparation. In general, the more material that can be removed and tracked gravimetrically, the fewer interferences will remain and the results should be more accurate and reliable. The steps outlined below are based on the Chatfield (1991) method and must be followed to maximize detection and accurate quantitation of asbestos. All mass determinations must be made to the nearest 1 mg or less. Filter residue prepared for PLM analysis of NOBs according to this Item (198.6 - Section 4.1 through 4.4) may be used for TEM grid preparation so that steps in Sections 4.1 through 4.4 of Item 198.4 do not have to be repeated.

The United States Environmental Protection Agency has clarified how bulk samples that contain multiple layers are to be analyzed and reported (U.S.E.P.A., 1995; U.S.E.P.A., 1994a; U.S.E.P.A., 1994b). Layered samples should be handled according to these guidelines.

4.1. **Preliminary Examination.** Each sample must be examined in its entirety by stereobinocular microscopy. (Preliminary PLM analysis is not mandatory as the NOB matrix materials will likely dissolve in RI liquid, making it impossible to measure fiber RI and to accurately measure asbestos quantity). This mandatory stereobinocular microscopical examination will serve three purposes.

4.1.1. Determine the existence of different materials or layers: Each discrete material must be prepared and analyzed separately.

4.1.2. Determine the overall homogeneity of the material: If the material is homogeneous, subsampling of sections for preparation and analysis can be at random.

4.1.3. Search for protruding fibers: Fibers protruding from the matrix may be removed for PLM analysis. These fibers must be matrix-free because dissolution of NOB matrix in RI liquids will change the liquid's RI and lead to incorrect measurements of fiber RI.

If the protruding fiber(s) is identified as asbestos, the laboratory may notify their client that the NOB contains asbestos at an *unquantified percentage* as detailed in 6.3.2.1 and that full gravimetric reduction will be necessary to determine a specific percentage. *This step cannot be used to report that the sample is an ACM or a non-ACM.*

The identification of asbestos by PLM in this step must be noted on the analysis sheet. If full gravimetric reduction and TEM analysis of this sample (Item 198.4) yields 1% or less asbestos, the report to the client will state that the results are *Conflicting*, as detailed in Item 198.4 Section 6.3.2.3.

4.2. **Preliminary Preparation**. All extraneous materials (mastic, wax, polish)

should be removed before preparation is initiated. Mastic must be considered a separate sample and must be removed for independent analysis. For a sample with distinctly different layers that are separable, each layer must be prepared, analyzed, and reported separately (U.S.E.P.A 1994a, 1994b).

4.3. **Organic Reduction**. Shave off approximately 100-500 mg NOB into a tared crucible and weigh. The mass of the subsample must be at least 200 times greater than the readability of the laboratory's analytical balance. Place in muffle furnace at 480^{o}C until mass stabilizes (1-12 hr). Cool in a desiccator, reweigh and calculate percent organic loss.

4.4. **Acid Digestion**. Grind residue in crucible with 0.5 mL 0.1-µm-filtered distilled water and add 2-5 mL concentrated HCl. After 15 minutes, dilute with 0.1-µm-filtered distilled water and pour into filtration apparatus holding a tared 0.4-µm polycarbonate filter and apply vacuum. Rinse the crucible a second time and pour this into the filtration apparatus and rinse down the sides of the apparatus. When filtration is complete, transfer filter and residue carefully to a clean, tared plastic petri dish and allow filter to dry to stable mass under a heat lamp. Weigh filter and petri dish and calculate percent mineral carbonate loss. *If* the residue mass is less than 1% of the subsample's original mass, analysis may be terminated and the sample reported as a non-ACM (Section 6.3.2.1).

4.5. **Slide Preparation**. At least four subsamples from the residue on the filter shall be mounted on clearly labeled microscope slides under separate whole (>250 mm^2) coverslips. To conserve microscope slides and storage space, two coverslips may be mounted on the same slide. A section (4-9mm^2) of residue should be detached from the polycarbonate filter and placed in a droplet of appropriate refractive-index liquid on a clean microscope slide. This residue should be disaggregated by a shearing rubout procedure or similar technique. A coverslip is placed on the preparation and more medium is added at the coverslip edge as necessary.

5. **Sample Analysis**

5.1. **Identification**. It is expected that analysts using this method are competent in the identification of asbestos by PLM and can refer to texts such as McCrone (1980, 1988) for assistance in identification. All fibrous components in each sample shall be positively identified. Typical properties of asbestos are outlined in Table I at the end of Item 198.1. Deviations from these properties are sometimes seen for asbestos from atypical ores or, more frequently, for asbestos that has been altered chemically or thermally (Laughlin and McCrone, 1989). Materials that commonly interfere with the identification of asbestos are

detailed in Section 2 of the U.S.E.P.A.'s Test Method (Perkins and Harvey 1993). At least the first four fibers of asbestos in each sample shall be positively identified by each of the criteria required on the analysis sheet:

5.1.1. Morphology.

5.1.2. Refractive index (measured at $?_o$=589.5 nm) fiber length *and* fiber width. This shall be a numerical value (±0.004) that can be determined by the Becke line method or by use of dispersion staining tables (e.g., McCrone 1989, Su 1994). Laboratory temperature must be measured using the calibrated laboratory thermometer and used in calculating refractive index.

5.1.3. Sign of elongation.

5.1.4. Pleochroism and color.

5.1.5. Extinction angle along fiber length.

5.1.6. Birefringence.

Additional observations are required for difficult samples.

5.2. **Quantitation**. Accurate quantitation is most critical at the 1 percent level, the level differentiating ACM vs non-ACM. Because the U.S.E.P.A.'s initial ACM definition was based on weight and because of PLM's limitation to determining areal percentage, the National Emission Standards for Hazardous Air Pollutants (NESHAP, 1990) rule defines "friable asbestos material" and "nonfriable ACMs" as "containing more than 1 percent asbestos as determined using" the EPA (1982) interim PLM method. While the EPA PLM method analyzes on an areal basis, it also allows removal of matrix materials and "requires a correction for percent weight loss". Thus weight percentage and area percentage determinations can be combined during analysis. The NESHAP preamble (55 *FR* 48410) includes an important discussion of quantitation of ACMs.

The analyst is encouraged to review references in Section 9 for useful strategies in the identification of asbestos in VATs. High-contrast Becke lines can enhance detection of fine fibers. Low RI liquids (solvents or Cargille oils) may be used with 400X phase-contrast microscopy to increase Becke-line contrast and aid in detection and quantitation of fibers. However, asbestos type(s) shall be positively identified in appropriate media according to all criteria in Section 5.1.

Matrix losses shall be gravimetrically tracked at each preparation step (Section 4.2 through 4.4). Estimates of asbestos percentage versus the percentage of remaining insoluble inorganic matrix material shall be made for each of the four coverslip preparations. This shall be done by the stratified point-counting

method.

5.2.1. **Point Counting Criteria**. A point is a discrete point or the intersection of two mutually perpendicular lines in the eyepiece reticle. Thus there is a single point in a cross-hair reticle and 25 points in a Chalkley reticle. A nonempty point is the visual superposition of a point over any material in the slide preparation. A nonempty point shall be categorized as a specific asbestos variety, as a specific non-asbestos fiber type or as nonfibrous material (see Section 5.1 for identification criteria), while empty points are those points that lie over areas containing no materials. Ideally, slide preparations should contain approximately 50% nonempty points. Moving to new fields of view shall be done at random, with the analyst looking away temporarily while moving the slide. The slide shall never be deliberately moved to preferred fields of view under the reticle. If the point(s) lie over an area where particles are heavily clumped, the analyst should move the slide to a new field to avoid attempting to count multiple layers under a point. For the occasional superposition of a point over two particles, the analyst should count both particles as separate nonempty points.

5.2.2. **Counting Rules**. Point counting shall be done on the PLM, usually with the slide between crossed polars and with a first-order red compensator inserted in the 45^{o} port above the slide. In some situations where extremely fine asbestos fibers are present, it may be preferable to analyze the sample between *slightly* uncrossed polars without the compensator. Other situations may warrant point counting in a dispersion-staining mode. All point counting shall be done at 100x magnification although it will be advantageous at times to switch to higher magnification(s) for enhanced visualization of identification criteria. For each of the first four slides, counting shall be performed until *either* one asbestos point is counted *or* 50 nonempty points are counted. No more than one asbestos point may be counted per preparation. If four asbestos points have been counted after all four preparations have been analyzed, analysis should be halted and calculations based on the total points counted. If less than four asbestos points have been counted, additional coverslip preparations shall be analyzed (at the rate of 50 nonempty points per preparation) until either: a) at least four asbestos points have been counted, or b) at least 400 nonempty points from at least 8 slide preparations have been counted. When analysis is performed with a multi-point eyepiece, a uniform scan pattern shall always be followed so that an asbestos fiber is not automatically the first point counted in a field. For

example, the top left point is always the first point counted, the bottom right point is always the last point counted and all points between are counted in a systematic pattern. Non-asbestos fibers may be counted separately to produce point-count quantitation or they may be counted as part of a larger "non-asbestos" category and then quantitation done by visual estimation similar to the Scanning Option (Section 5.2.3). Sample composition is calculated based on the nonempty points counted as detailed in Section 5.2.5.

5.2.3. **Scanning Option for Negative Samples**. If, based on the stereobinocular microscopical observation, the analyst is confident that the sample contains no asbestos, a scanning option may be substituted for point counting. This option requires the analyst to scan the entire area of all 4 mandatory slide preparations by PLM at 100x magnification. Percentages of non-asbestos fibrous components may be determined by visual estimation. If asbestos is detected during this scan, stratified point-counting shall be initiated. Starting with the slide on which the asbestos was detected, the analyst returns to the normal starting position on the coverslip and begins counting the 50 points (or up to the first asbestos point) as required on that slide and any remaining slides. Slides from that particular sample which were already scanned in their entirety and contained no asbestos will be considered to contain 50 non-asbestos points each.

5.2.4. **Trace Levels of Asbestos**. If asbestos appears in a field of view but does not lie directly under a point, the analyst shall note this on the analysis sheet. If the analyst suspects that, based on the stereobinocular examination, asbestos is present but none is detected during the point-count analysis, the analyst shall retrieve the original bulk material, remove any suspicious fibers, mount them in an appropriate medium, and determine their identity. If the fibers are confirmed as asbestos, this should be noted on the analysis sheet. Although these observations will not be used for quantitation, they will be incorporated into the final report to warn about potential false negatives.

5.2.5. **Calculations**. Calculations are performed in the same manner as for the EPA point-count method. The percentage of each asbestos type, each non-asbestos fiber type, and nonfibrous components are calculated by dividing the number of nonempty points of that component by the total nonempty points counted for that sample. Thus:

$$\% \text{ Asbestos} = (AP \times 100\%)/TP$$

where

AP = number of points counted for a specific asbestos type

TP = total number of nonempty points counted

For example, if point counting yielded a chrysotile point as the fifteenth nonempty point on the first slide and as the thirtieth nonempty point on the second slide, no asbestos was detected in 50 nonempty points on the third slide, chrysotile was counted as the tenth nonempty point on the fourth slide and amosite was counted as the forty-second nonempty point on the fifth slide, then:

TP = 15 + 30 + 50 + 10 + 42 = 147

AP for chrysotile = 3

thus (3 X 100%)/147 = 2.0% Chrysotile

AP for amosite = 1

thus (1 X 100%)/147 = 0.68% Amosite

AP for total asbestos = 1 (amosite) + 3 (chrysotile) = 4 (total)

thus (4 X 100%)/147 = 2.7% Asbestos

The percentage of asbestos versus the remaining inorganic residue (AOS below) is the mean of the four slide estimations.

The amount of asbestos is then calculated by:

% Asbestos = (PAM/OM)X(AP)

where:

PAM = Mass of residue after furnace and acid treatments (mg)

OM = Mass of original subsample (mg)

AP = Mean percentage of asbestos (versus inorganic residue) in final slide preparations

5.3. **Analytical Records**. Detailed records shall be kept of all phases of analysis. An analysis sheet that includes all the data required in Section 3.21 shall be filled out completely, signed and dated by analyst.

6. **Test Reports**. Reports to clients shall include at least the following:

6.1. **Client**. Identify name and address.

6.2. **Sample Identity**. The identification number (Section 8.1.5.1.3) assigned by the laboratory shall be clearly cross-referenced to information provided by the client or collector (field identification number, location - Section 8.1.5.1.2) for each sample.

6.3. **Analytical Results**. The following information shall be reported for each sample:

6.3.1. Color.

6.3.2. Presence or absence of asbestos, total percentage of asbestos, type(s) of asbestos present, and percentage of each asbestos type. Asbestos quantities shall be reported as follows:

6.3.2.1. For samples that have yielded asbestos fibers during preliminary analysis (4.1.3) but have not received full gravimetric reduction, report results as “Asbestos Detected at Unquantified Percentage”. Report cannot classify sample as ACM or non-ACM.

6.3.2.2. **Disclaimers**. Because quantitative TEM (ELAP Item 198.4) is the only consistently reliable method for detection of asbestos in NOBs, a disclaimer for PLM is required for each negative (1% or less asbestos) NOB sample that contained more than 1% residue in Section 4.4. This disclaimer **shall** be cited along with each negative result reported to clients:

"Polarized-light microscopy is not consistently reliable in detecting asbestos in floor coverings and similar non-friable organically bound materials. Quantitative transmission electron microscopy is currently the only method that can be used to determine if this material can be considered or treated as non-asbestos containing."

6.3.2.3. "Inconclusive - No asbestos detected" - for samples that contained no PLM-detectable asbestos.

6.3.2.4 "Inconclusive - Trace **(fill in type(s))** asbestos detected at 1% or less" - for samples that contained 1% or less asbestos and for samples that contained 0 asbestos points out of 400 (or more) nonempty points but did contain asbestos positively identified by PLM (Section 4.1.3 and/or Section 5.2.4).

6.3.2.5. " **(fill in type)** asbestos detected at %" - for each type of asbestos for samples that yielded one or more asbestos points.

6.3.2.6. " % Total Asbestos" - sum from all types reported in Section

6.3.2. 5. Percentage should be rounded off to two digits.

6.3.3. Type(s) and estimated percentage(s) of other fibrous materials present.

6.3.4. Percentage of nonfibrous matrix material.

6.4. **Homogeneity**. For samples with obvious layers, the summary shall include results as specified in Section 6.3 for each layer. Compositing of layers into a single result is no longer allowed, except for joint compounds in certain cases (U.S.E.P.A. 1994b).

7. **Precision and Accuracy**.

7.1. **Precision**. The New York State Department of Health sent out floor tiles to more than a dozen PLM laboratories, requesting that the laboratories use their own PLM method for analysis. Relative standard deviations for VATs in the 15 to 30% ranges (as determined by quantitative TEM) were about 60 to 100%. It is expected that precision will not be as good by PLM because of its inability to detect thin asbestos fibers.

7.2. **Accuracy**. The NYSDOH study revealed that PLM often underestimated asbestos concentrations in VATs when compared to quantitative transmission electron microscopy. Results for a sample containing 15% chrysotile ranged from 0 to 7.5%, with a mean of 2.5% while results from a 25% sample ranged from 1.5 to 35%, with a mean of 14%.

8. **Quality Assurance**

8.1. **Quality-Assurance Manual**. The laboratory's QA manual can be devoted to asbestos analysis or it can be a larger manual comprising many types of analyses. In either case, the QA manual shall include at least the following and shall be in conformance with the general ELAP requirements for quality manuals:

8.1.1. **Quality Assurance Responsibility**. A single individual shall be designated as responsible for overseeing quality assurance. This includes updating and controlling distribution of the laboratory's quality-assurance manual, performing at least monthly reviews of analytical quality control and contamination control and resolving any deficiencies.

8.1.2. **Analytical Method**. The laboratory's implementation of the NOB method shall be explicitly detailed in this manual. If a copy of an externally published method, e.g., this document or the U.S.E.P.A. Test Method, is used then it shall be customized to include only those options actually utilized in the laboratory.

8.1.3. **Analytical Quality Control**. The manual shall describe a systematic method of submitting quality-control samples including intra-analyst, inter-analyst, standards, proficiency-testing and interlaboratory samples so that analysts are unaware of the sample's true identity.

8.1.4. **Sample Control**. The manual shall describe all aspects of NOB sample handling from sample receipt to sample disposal. Criteria for acceptance and rejection of received samples (e.g. broken containers, too-small samples) and for safe handling shall be defined. Samples shall be retained in secure areas (similar to areas used to store evidentiary material) for at least 60 days after a final report of results is sent to the client. Samples may be returned to the client at the client's request at any time. Procedures for safe disposal of asbestos (in compliance with federal and local regulations) shall be detailed and records of such disposals shall be kept.

8.1.5. **Recordkeeping**. The laboratory shall maintain a recordkeeping system as specified in its QA manual. This shall define provisions to ensure the secure storage of records for at least five years. Records, whether they be hardcopy or computer files, shall be easily accessible and shall include:

8.1.5.1. **Sample Accessioning**. Each sample shall pass through an accessioning process that documents:

8.1.5.1.1. **Client**. This should include name, address, phone number and name of contact person.

8.1.5.1.2. **Client Sample Identification**. This should include the identification characteristics provided by the client, e.g., identification number, collection site, etc.

8.1.5.1.3. **Laboratory Sample Identification**. A unique laboratory sample identification number shall be assigned to each sample.

8.1.5.1.4. **Date of Receipt**

8.1.5.1.5. **Chain of Custody**

8.1.5.1.6. **Condition of Sample (Accept/Reject)**

8.1.5.1.7. **Type of Sample**. This should place the sample in one of several bulk sample categories, e.g., friable, floor tile, etc.

8.1.5.2. **Analytical Quality Control**. All results of analytical quality control activities shall be recorded in an orderly fashion.

8.1.5.3. **Equipment and Supply Records**. Records shall be kept for maintenance, calibration, replacement and repair of pertinent equipment and supplies. For major pieces of equipment (microscopes, hoods, muffle furnaces, analytical balances) these records shall include manufacturer, model and serial numbers, major components, calibration and maintenance/service information and location of manuals.

8.1.5.4. **Contamination Control**. See Section 8.3.

8.1.5.5. **Calibration**. See Section 8.4.

8.1.5.6. **Personnel**. See Section 8.5.

8.1.5.7. **Test Reports**. See Section 6.

8.1.6. **Staff Training Programs**. The Laboratory Director is responsible for continued in-house training of analysts. Each analyst shall receive formal training in proper identification and quantitation of asbestos in bulk samples. This can be achieved by sending the analyst to a 5-day course at a recognized PLM institute or by an in-house training course with a detailed and extensive curriculum equivalent to that at recognized institutes. The course shall include formal training in the theory of mineral analysis by PLM and hands-on analysis of all asbestos types and common fiber types. This formal training shall be followed by an in-house apprenticeship during which performance is carefully monitored and documented to show increasing competence to the point where the analyst can work independently within the laboratory's QA framework.

8.2. **Analytical Quality Control**. At least 10% of a laboratory's PLM analyses shall be re-analyzed as part of the laboratory's QC program. Selection of samples for quality-control (intra-analyst, inter-analyst, interlaboratory, or reference) analyses shall be semi-random so that the analyst performing the original analysis is not aware that the sample will be reanalyzed. Furthermore, the second analyst shall not know the results of the original analysis. These QC data shall be routinely assessed to evaluate the precision and accuracy of each analyst and to identify and correct areas of analytical weaknesses. These QC samples shall be routinely resubmitted for analytical quality control according to the method detailed in Section 8.1.3. QC reanalysis shall include complete re-preparation (including gravimetric reduction) of slides from the original sample. All QC results shall be documented in a QC notebook or on appropriate analysis sheets. Procedures for resolving analytical discrepancies shall be defined and details of resolved discrepancies shall be recorded. Discrepancies include classification differences (ACM vs. non-ACM),

identification differences (e.g., chrysotile vs. amosite) and substantial quantitation differences, as specified below. Monthly summaries shall be compiled for each analyst.

One of QC's primary functions is the timely detection and correction of deficiencies in an analytical system. QC is not an optional activity to be carried out at the convenience of the laboratory or to be postponed when sample loads are heavy. ELAP-certified laboratories **shall** perform PLM QC concurrent with sample load and **shall** evaluate these QC results before sending written reports to clients.

8.2.1. **Intra-Analyst QC**. At least 1 out of 50 samples shall be reanalyzed by the same analyst. Relative difference (R) values shall be calculated for each pair of re-analyses and shall be compiled and statistically evaluated for that analyst, comparing his/her QC result to his/her original result for that same sample using:

$$R = |(A-B)/((A+B)/2)|$$

where

A = First result from the analyst being checked

B = Second result from same analyst for same sample

(Note that these intra-analyst R values are absolute values)

Intra-analyst results will require additional reanalysis, possibly including another analyst, to resolve discrepancies when classification (ACM vs. non-ACM) errors occur, when asbestos identification errors occur, or when R is greater than 1.0.

Record: Sample, date(s) of analyses, analysts' signatures, both results, R value, reason(s) for and resolution(s) of disagreement(s). R control charts shall be updated monthly for each analyst monitoring intra-analyst precision. These charts shall include all R values from at least the three previous months.

8.2.2. **Inter-Analyst QC**. At least 1 out of 15 samples shall be reanalyzed by another analyst. R values shall be calculated for each pair of re-analyses and shall be compiled and statistically evaluated for each analyst, comparing his/her result to a QC result for that same sample from another analyst using:

$$R = (A-B)/((A+B)/2)$$

where

A = Result from the analyst being checked

B = Result from other analyst for same sample

Inter-analyst results will require additional reanalysis, possibly including another analyst, to resolve discrepancies when classification (ACM vs. non-ACM) errors occur, when asbestos identification errors occur, or when R is greater than 1.0 or less than -1.0.

Obviously single-analyst laboratories will not be able to meet this requirement. Instead, they shall perform **intra-analyst** analyses on 1 out of every **11** samples.

Record: Sample, date(s) of analyses, analysts' signatures, both results, R value, reason(s) for and resolution(s) of disagreement(s). R-bar control charts shall be updated monthly for each analyst monitoring both intra-and inter-analyst precision. These charts shall include all R values from at least the three previous months.

8.2.3. **Standard/Reference QC**. At least 1 out of 100 samples shall be a verified quantitative standard (Section 3.14.2) that has been routinely resubmitted to determine analyst's precision and accuracy. Results should be displayed on x-bar charts to keep track of each analyst's accuracy and precision.

8.2.4. **Interlaboratory QC**. The laboratory must participate in round-robin testing with at least one other ELAP-certified lab. For laboratories with more than one bulk-sample analyst, samples must be sent to this other lab at least four times per year or at the rate of 1 sample in 500 routine samples (whichever is less). For single-analyst laboratories, at least 1 sample in 500 routine samples must be sent to this lab. These samples must be samples previously analyzed as QC samples. Results of these analyses must be assessed in accordance with QC-outlier criteria detailed in the lab's QA manual. At the very least, the QA manual must address misclassifications (false positives, false negatives) and misidentification of asbestos types.

8.3. **Contamination Control**.

8.3.1. **Prevention**. The laboratory shall detail its methods for preventing cross contamination of equipment, supplies and reagents. Much of this will be careful cleaning of work area, equipment and supplies. Intensity and frequency of this effort should be based on experience gained through any contamination detected as described in 8.3.2.

8.3.2. **Monitoring**. The laboratory shall have a documented routine

procedure for monitoring contamination of laboratory equipment, supplies and work stations and for resolving contamination problems when discovered. If any asbestos is detected, the source of contamination shall be traced and the problem resolved to prevent recurrence. Any of the previous samples that may have had results affected by the contamination shall be reanalyzed and the client notified of any revisions to reported values. Detailed records of monitoring shall be maintained. At least one non-ACM NOB shall be prepared and analyzed with every 20 samples analyzed. This non-ACM shall go through the full preparation and analysis regimen for the type of analysis being performed.

8.4. **Calibration**. Written records for calibration of the following equipment and supplies shall be kept. All calibrations listed below (unless otherwise noted) shall be performed under the same analytical conditions used for routine asbestos analysis and shall be recorded in a bound notebook and include date and analyst's signature. Frequencies stated below may be reduced to "before next use" if no samples are analyzed after the last calibration period has expired. Likewise, frequencies shall be increased following non-routine maintenance or unacceptable calibration performance.

8.4.1. **Refractive Index Media**. The refractive index medium (oil or solid) used to prepare slides shall be calibrated to within 0.004 using certified refractive-index solids or a refractometer. This shall be performed when the original container is first opened for use and thereafter at three-month intervals.

Record: Date, nominal refractive index, measured refractive index, temperature.

8.4.2. **Laboratory Thermometer**. The laboratory thermometer must be calibrated to a NIST-traceable standard annually to ±1° C within a temperature range of 20° to 30°C.

Record: Date, nominal temperature from thermometer, actual temperature.

8.4.3. **PLM Alignment**. The PLM shall be aligned daily to achieve illumination as close to Köhler illumination as possible and centered through the substage condenser and iris diaphragm. The stage's rotation axis shall be centered with the appropriate objectives. Analyzer and polarizer shall be rotated to maximum extinction with each other and their privileged directions shall be oriented parallel to the lines in the fixed ocular cross hairs (or grid) and aligned at 45^{o} to the accessory port.

Record: Date, check-off for rotation centering, axial illumination, full extinction and crosshair alignment fixed in the polarizer's privileged direction.

8.4.4. **Refractive-Index Colors**. Dispersion-staining or Becke-line colors shall be determined monthly from the permanent 1.680 mount of amosite (Section 3.14.3). The source of any deviations shall be located and corrected.

Record: Date, colors or wavelengths perpendicular and parallel to length.

8.4.5. **Analytical Balance**.

8.4.5.1. Analytical balances should be serviced by a qualified service organization annually.

Record: Service organization sticker with date of service.

8.4.5.2. Analytical balances shall be checked in two ranges weekly with class S weights. The ranges selected should reflect routine use of the balance and the actual class S weights used should test the scale at mid-point.

Record: Date, target and actual readings in a tabular format.

8.4.6. **Muffle Oven**. Temperatures on external meters (either direct-temperature displays or graduated potentiometers) shall be calibrated quarterly (Section 3.7.2). This shall be a three-point calibration covering a temperature range of at least 450^{o} to 480^{o}C. If a thermometer is used for calibration, the thermometer bulb should be immersed in a sand bath.

Record: Date, target temperature, measured temperature in a tabular format.

8.4.7. **HEPA-Ventilated Sample Preparation Enclosure**. Flow rate at enclosure opening shall be measured twice annually to the nearest 5 fpm. Flow rate shall not be less than 75 fpm.

Record: Date, flow rate.

8.5. **Personnel**. The laboratory shall assure that all analysts are competent to perform PLM analysis of asbestos in bulk samples. Analysts shall be familiar with the theory of dispersion staining and the measurement of refractive indices by the Becke line technique and be able to apply these. A personnel file shall be maintained for every analyst and shall include:

8.5.1. **Resume**. Each resume shall include formal education, experience and other pertinent information.

8.5.2. **Training**. Both classroom and in-house training shall be detailed to demonstrate the analyst's competence in performing independent analysis.

8.5.3. **Job Title**. A job title shall be defined that specifies responsibilities and laboratory assignments.

8.5.4. **QC Records**. Details and summaries of results of QC analyses shall be updated at least monthly. Accuracy shall be determined from the standard/reference samples while precision shall be determined from intra- and inter-analyst R values.

8.5.5. **Deficiency Resolutions**. Details of noted deficiencies and steps taken to resolve these shall be included in the personnel folder.

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Example 1 - Bulk Sample Analysis Sheet - Non-Friable Organically Bound

Analyst: _____ **Analysis Date:** _____ **Sample ID:** _____

STEREOBINOCULAR MICROSCOPY:

Color: _____ **Texture:** _____ **Homogeneity:** _____ **Homogenization:** _____

Probable Fibers: _____ **Remarks:** _____

MATRIX REDUCTION:

ASHING: g: A:(Crucible) _____ **B:(Crucible + Subsample)** _____ **C:(Crucible + Ashed Subsample)** _____

ACID DIGESTION: g: D:(Filter/Petri) _____ **E:(Filter/Petri + Residue)**

CALCULATIONS	g		Percent	
	Calculation	Result	Calculation	Result
Untreated Sample	(F = B - A)		(G = 100%)	100%
Organic Component	(H = (F - (C - A))		(I = (H X G) / F)	
Acid-Insoluble Inorganic Component	(J = E - D)		(K = (J X G) / F)	
Acid-Soluble Inorganic Component	(L = (F - (J + H)))		(M = (L X G) / F)	

Remarks: _____

POLARIZED-LIGHT MICROSCOPY:

IDENTIFICATION:

Morphology	Refractive Index		Sign of Elongation	Extinction Angle	Pleochroism /Color	Birefringence	Other	Identity
	-	¦						

Remarks: _____

QUANTITATION:	Slide 1	Slide 2	Slide 3	Slide 4	Mean (N)
Percent Asbestos					

TABLE I

ASBESTOS TYPES	Morphology and Color	Refractive Indices[a]		Sign of Elongation	Extinction Angle
		Perpendicular	Parallel		
Chrysotile	White to pale green. Very flexible with "kinks". Wavy with "knuckles" under PLM.	1.493-1.559	1.517-1.567	Positive	Parallel/Undulose.
Amosite	Tan. Moderately flexible but straight bundles. Easily splayed ends.	1.657-1.686	1.696-1.729	Positive	Parallel. Very infrequently shows 2^{o} extinction.
Crocidolite	Dark blue. Flexible. Some "kinks". Splayed ends. Strongly pleochroic.	1.654-1.701	1.668-1.717	Negative	Parallel
Anthophyllite	White to light tan. Usually stiff. Ends splayed to blunt.	1.596-1.652	1.615-1.722	Positive	Parallel
Tremolite	White to light tan. Usually stiff. Large bundles may have splayed ends.	1.599-1.628	1.625-1.655	Positive	Parallel. Very thin fibers or cleavage fragments will show up to 15^{o} extinction.
Actinolite	White to green. Usually stiff. Large bundles may have splayed ends. Often pleochroic.	1.600-1.668	1.625-1.688	Positive	Parallel. Very thin fibers or cleavage fragments will show up to 20^{o} extinction.

[a] Perkins, R.L., and Harvey, B.W. 1993.