

ENVIRONMENTAL LABORATORY APPROVAL PROGRAM CERTIFICATION MANUAL

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1. Introduction The Stratified Point-Counting Method has been approved by the New York State Department of Health and promulgated Environmental Laboratory Approval Program (ELAP) as Item 198.1 in the ELAP Certification Manual.

1.1. Objective. For samples containing high concentrations of asbestos, the stratified point-count technique invokes labor-saving semi-quantitative counting rules. Samples initially judged (based on stereobinocular microscope examination) to be negative may be analyzed by a visual estimation option.

For samples with asbestos concentrations near the 1% level, the stratified point-count method requires the full 400-point count regimen. To minimize the potential for false negatives, the stratified method requires non-quantitative notation of asbestos not directly under points using the term "trace". Such reports of "trace" asbestos warrant additional sampling and/or analysis of the material.

1.2. Definitions

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

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

1.2.3. 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).

1.2.4. 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.

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

2. Application. Friable bulk materials shall be analyzed by the point-counting methods in this Item (198.1) or by the gravimetrically tracked matrix reduction and point-counting method for NOB materials in Item 198.6. Textiles and ceiling tiles, for example, could be prepared and analyzed through application of Item 198.6. The method in this Item (198.1) shall not be used for VAT, resilient floor tiles, mastic, asphalt shingles, roofing materials, paint chips, caulking, glazing and other NOB materials. Rather, NOB materials must be analyzed by Item 198.4 (quantitative TEM with no disclaimers) or Item 198.6 (NOB

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materials by PLM, with disclaimers required for negative samples).

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

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 (optional).

3.6.1. 0.4- μ m pore polycarbonate filters (optional).

3.6.2. Petri dishes and covers (optional).

3.7. Muffle furnace capable of sustained operation at 500°C.

3.7.1. Crucibles (bottom and lid) that can withstand 500°C.

3.7.2. Instrument or materials capable of calibrating muffle furnace at 480°C:

3.7.2.1. High-temperature thermometer with range to at least 500°C and with readable subdivisions of 5°C or less or

3.7.2.2. Melting-point solids with capability of differentiating 5°C differences between 400°C and 500°C or

3.7.2.3. Independent potentiometer capable of differentiating 5°C differences between 400°C and 500°C.

3.8. Concentrated HCl, reagent grade.

3.9. Surfactant (sodium metaphosphate or Aerosol OT).

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3.10. Heat lamp, slide warmer or drying oven.

3.11. Ultrasonic bath (optional).

3.12. Filtered (0.1- μ m) distilled water (optional).

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. 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 $\pm 1^\circ$ C.

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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° to analyzer for wave retardation plate.

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

3.19.5. 360° graduated (in 1° 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: (see Example 1 at end of Item 198.1).

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 (if any). This should include solvent or ashing steps used and amount of matrix removed during each step.

3.21.7. Entries for the first four asbestos fibers identified:

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3.21.7.1. Morphology.

3.21.7.2. Refractive index (at $\lambda_0=589.5$ nm) parallel to fiber length in specified n_D medium.

3.21.7.3. Refractive index (at $\lambda_0=589.5$ 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 recording asbestos points counted and calculation of asbestos percentage.

3.21.9. Final results including:

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

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

3.21.9.3. Percentage of non-fibrous material present.

4. Sample Preparation. 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 Analysis. Before homogenization, each sample shall be examined by stereobinocular microscope for tentative identification of any fibers present. This observation, along with the general characteristics of the bulk material describing (color, homogeneity, texture) shall be recorded on the analysis sheet. For a sample with distinctly different layers that are separable, each layer shall be prepared and analyzed separately.

4.2. Homogenization. Not all samples will require homogenization. Many are already homogeneous on a macroscopic scale and others may simply require homogenization with a mortar and pestle. Homogenization shall be performed on dry samples within the HEPA-filtered sample preparation area to prevent contamination of the overall work area. Samples containing small percentages of fibers requires the most extensive homogenization to ensure uniform distribution of these fibers. Special care should be taken when homogenizing samples containing vermiculite or non-asbestiform amphiboles; pulverization with a mortar and pestle or mill may produce asbestos-like fragments with aspect ratios greater than 3:1. Precautions shall be taken to rinse and dry homogenization equipment between sample preparations to prevent cross contamination.

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4.3. Matrix Modification. Some samples may require additional steps to dislodge problem matrices. These steps range from simple surfactant treatment to complete removal. Matrix removal is required for NOBs but is also recommended when matrices interfere with optical properties, e.g., "milky way effect", or when asbestos concentrations are near the one percent level. Care shall be taken when using acid or a muffle furnace to minimize damage or alterations to chrysotile. In instances where the matrix is completely removed, the sample shall be weighed before and after matrix removal so that the resultant asbestos percentage can be corrected to reflect its percentage in the original material.

4.4. Slide Preparation. After the sample is adequately homogenized, at least four subsamples shall be mounted on clearly labeled microscope slides under separate whole (>250 mm²) coverslips. Additional subsamples may have to be prepared to meet the counting rules outlined in Section 5.2. To conserve microscope slides and storage space, two coverslips may be mounted on the same slide. For each subsample, a small drop of mounting medium (appropriate to the type of fiber tentatively identified in the stereobinocular observation) is placed on the slide. For samples with no apparent fibers, $n_D=1.550$ is usually appropriate. A small pinch sample from the homogenized material is removed with forceps. For each additional subsample, pinch samples should be removed from different areas of the homogeneous material. These pinch samples are transferred to the mounting medium on the slide and dispersed evenly throughout the drop with forceps or needles. A coverslip is placed on the preparation and more medium is added at the coverslip edge as necessary. If the coverslip is raised obliquely because of large grains, the sample requires further homogenization or milling to reduce grain sizes.

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 $\lambda_0=589.5$ 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

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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" (Section 1.7.2.2). 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 U.S.E.P.A. Test Method (Perkins and Harvey, 1993) discusses a means for performing visual estimation of asbestos percentage in friable bulk samples. The analyst calibrates him/herself using formulated-weight standards. While a limited selection of these standards is available from the authors, these are only a small start of an immense series of standards that would be needed to mimic the myriad matrix and fiber compositions encountered in real-world samples. Since this is impractical to dictate in a regulatory sense, ELAP will continue to require the use of point counting for quantitation of asbestos in friable bulk samples. The superior accuracy and precision of point-counting versus visual-estimation has been verified through ELAP's proficiency-testing program (Webber *et al.*, 1997). Prior to point counting, two slide preparations shall be scanned between crossed polars to characterize components. This shall be performed both with the 530-nm compensator inserted so that signs of elongation and isotropic particles can be detected and also with the compensator retracted so that very thin fibers can be detected.

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

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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° 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. If no asbestos is detected on any of these slides, the sample is non-ACM and percentages of 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

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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:

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

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:

$$\text{TP} = 15 + 30 + 50 + 10 + 42 = 147$$

$$\text{AP for chrysotile} = 3$$

$$\text{thus } (3 \times 100\%) / 147 = 2.0\% \text{ Chrysotile}$$

$$\text{AP for amosite} = 1$$

$$\text{thus } (1 \times 100\%) / 147 = 0.68\% \text{ Amosite}$$

$$\text{AP for total asbestos} = 1 \text{ (amosite)} + 3 \text{ (chrysotile)} = 4 \text{ (total)}$$

$$\text{thus } (4 \times 100\%) / 147 = 2.7\% \text{ Asbestos}$$

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

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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 one of the following:

6.3.2.1. "No asbestos detected" - for samples that contained no asbestos points (Section 5.2.2) and no asbestos traces (Section 5.2.4) as confirmed by PLM.

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

6.3.2.3. " (fill in type) asbestos detected at ___%" - for each type of asbestos for which asbestos point(s) were counted. Percentage should be rounded off to two digits.

6.3.2.4. " ___% Total Asbestos" - sum from all types reported in Section 6.3.2.3. 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.** Theoretically, the point-count method should yield a relative standard deviation no better than 50% at a composition of 1% asbestos when performed with 400 points. However, depending on matrices within a bulk sample, the point-count method (when performed with 300 or more points) has yielded relative standard deviations of 25% or less at compositions between 1 and 3 percent asbestos (Perkins 1989). Similar results were derived from synthetic bulk samples with formulated weight compositions; relative standard deviations ranged from 24 to 45% for replicate samples containing 5 to 7% chrysotile and amosite (Webber *et al.* 1990). Certain matrices (e.g., chrysotile in vermiculite or cellulose) tend to increase analytical variability. Although

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intralaboratory precision with the 400-point method improves with increasing asbestos concentrations, precision in a stratified scheme will be reduced as the number of points counted are reduced. This reduction in precision is not critical, however, since only samples with high asbestos concentrations will have substantially decreased numbers of points counted. These samples have asbestos concentrations well in excess of 1% and thus are categorically ACM.

7.2. Accuracy.

The point-count method is usually more accurate than the visual estimation method at quantitating asbestos concentrations in bulk samples, especially at low concentrations (Perkins 1989, Perkins 1990, Webber *et al.* 1990, Harvey *et al.* 1991). Nonetheless, there are material combinations that seem to cause biases, e.g., the presence of cellulose causes underestimation of chrysotile while amosite in plaster is usually overestimated. In even these instances, however, point-count results are usually closer to actual weight percents than visual-estimation results.

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 stratified point-count 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 bulk 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 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

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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., pipe insulation, ceiling tile, plaster, 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

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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 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

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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. Because accuracy cannot be determined by reanalysis of routine field samples, at least 1 out of 100 samples shall be a standard or reference sample that has been routinely resubmitted to determine analyst's precision and accuracy. A set of these samples should be accumulated from proficiency-testing samples with predetermined weight compositions or from standards generated with weighed quantities of asbestos and other bulk components (Perkins and Harvey, 1993; Parekh *et al.*, 1992; Webber *et al.*, 1982). At least half of the reference samples submitted for this QC shall contain between 1 and 10% asbestos. Accuracy of each analyst shall be monitored by determining percentage recovery, e.g.,

$$\text{Recovery} = (\text{Analytical Result}/\text{Formulated Weight}) \times 100\%.$$

Results should be displayed on x-bar charts to keep track of each analyst's accuracy and precision.

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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 blank slide shall be prepared daily or with every 50 samples analyzed, whichever is less. This is prepared by mounting a subsample of an isotropic verified non-ACM, e.g., fiberglass, in a drop of immersion oil (n_D should reflect usage of various n_D 's) on a clean slide, rubbing preparation tools (forceps, "speedles", etc.) in the mount and placing a clean coverslip on the drop. The entire area under the coverslip shall be scanned to detect any asbestos contamination. A similar check shall be made after every 20 uses of each piece of homogenization equipment. An isotropic verified non-ACM shall be homogenized in the cleaned equipment, a slide prepared with the material and the slide scanned for asbestos contamination. (This can be substituted for the blank slide mentioned in this section).

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

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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 $\pm 1^\circ$ 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° 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 3.14.2). 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 optical 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° to 480°C. If a thermometer is used for calibration, the thermometer bulb should be immersed in a sand bath.

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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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Figure 1 - Bulk Sample Analysis Sheet - Friable

Analyst: _____ Analysis Date: _____ Sample ID: _____

STEREOBINOCULAR MICROSCOPY:

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

Probable Fibers: _____ Remarks: _____

POLARIZED-LIGHT MICROSCOPY · Condenser Focused · Condenser Centered · Rotation Centered
· Polars Extinct · Crosshair Privileged

IDENTIFICATION:

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

Remarks: _____

QUANTITATION: Method: · EPA 400 Point Count · Stratified Point Count · Scanning Option

| Slide 1 | | Slide 2 | | Slide 3 | | Slide 4 | | Slide 5 | | Slide 6 | | Slide 7 | | Slide 8 | |
|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| Type | Points- | Type | Points- | Type | Points- | Type | Points- | Type | Points- | Type | Points- | Type | Points- | Type | Points- |
| | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | |

C=Chrysotile, A=Amosite, D= Crocidolite, ___ = _____ Asbestos, ___ = _____ Asbestos
G=Fibrous Glass, L=Cellulose, W=Wollastonite, ___ = _____ Fiber, ___ = _____ Fiber
/Nonfiber, ___ = _____, ___ = _____, ___ = _____, ___ = _____

CALCULATIONS:

| | | | | |
|---------------------------|--|--|--|--|
| COMPONENT | | | | |
| Points Counted (PC) | | | | |
| Total Points Counted (TP) | | | | |
| Percentage (100*PC/TP) | | | | |

FINAL RESULTS:

| | Type | Percent | Type | Percent | Type | Percent | Type | Percent |
|--------------|------|---------|------|---------|------|---------|------|---------|
| ASBESTOS | | | | | | | | |
| Other Fibers | | | | | | | | |
| NonFibers | | | | | | | | |

Verified: _____ Date: _____ Remarks: _____

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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° 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° 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° extinction. |

^a Perkins, R.L., and Harvey, B.W. 1993.