Received 11/21/05
MSHA/OSRV

Regulations (Standards - 29 CFR)

OSHA Reference Method - Mandatory - 1910.1001 App A

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• **Part Number:**	1910
• **Part Title:**	Occupational Safety and Health Standards
• **Subpart:**	Z
• **Subpart Title:**	Toxic and Hazardous Substances
• **Standard Number:**	1910.1001 App A
• **Title:**	OSHA Reference Method - Mandatory

This mandatory appendix specifies the procedure for analyzing air samples for asbestos and specifies quality control procedures that must be implemented by laboratories performing the analysis. The sampling and analytical methods described below represent the elements of the available monitoring methods (such as Appendix B of thier regulation, the most current version of the OSHA method ID-160, or the most current version of the NIOSH Method 7400). All employers who are required to conduct air monitoring under paragraph (d) of the standard are required to utilize analytical laboratories that use this procedure, or an equivalent method, for collecting and analyzing samples.

pling and Analytical Procedure

1. The sampling medium for air samples shall be mixed cellulose ester filter membranes. These shall be designated by the manufacturer as suitable for asbestos counting. See below for rejection of blanks.

2. The preferred collection device shall be the 25-mm diameter cassette with an open-faced 50-mm electrically conductive extension cowl. The 37-mm cassette may be used if necessary but only if written justification for the need to use the 37-mm filter cassette accompanies the sample results in the employee's exposure monitoring record. Do not reuse or reload cassettes for asbestos sample collection.

3. An air flow rate between 0.5 liter/min and 2.5 liters/min shall be selected for the 25-mm cassette. If the 37-mm cassette is used, an air flow rate between 1 liter/min and 2.5 liters/min shall be selected.

4. Where possible, a sufficient air volume for each air sample shall be collected to yield between 100 and 1,300 fibers per square millimeter on the membrane filter. If a filter darkens in appearance or if loose dust is seen on the filter, a second sample shall be started.

5. Ship the samples in a rigid container with sufficient packing material to prevent dislodging the collected fibers. Packing material that has a high electrostatic charge on its surface (e.g., expanded polystyrene) cannot be used because such material can cause loss of fibers to the s of the cassette.

6. Calibrate each personal sampling pump before and after use with a representative filter cassette installed between the pump and the calibration devices.

AB24-COMM-107-3

7. Personal samples shall be taken in the "breathing zone" of the employee (i.e., attached to or near the collar or lapel near the worker's face).

Fiber counts shall be made by positive phase contrast using a microscope with an 8 to 10 X eyepiece and a 40 to 45 X objective for a total magnification of approximately 400 X and a numerical aperture of 0.65 to 0.75. The microscope shall also be fitted with a green or blue filter.

9. The microscope shall be fitted with a Walton-Beckett eyepiece graticule calibrated for a field diameter of 100 micrometers (+/-2 micrometers).

10. The phase-shift detection limit of the microscope shall be about 3 degrees measured using the HSE phase shift test slide as outlined below.

a. Place the test slide on the microscope stage and center it under the phase objective.

b. Bring the blocks of grooved lines into focus.

NOTE: The slide consists of seven sets of grooved lines (ca. 20 grooves to each block) in descending order of visibility from sets 1 to 7, seven being the least visible. The requirements for asbestos counting are that the microscope optics must resolve the grooved lines in set 3 completely, although they may appear somewhat faint, and that the grooved lines in sets 6 and 7 must be invisible. Sets 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope that fails to meet these requirements has either too low or too high a resolution to be used for asbestos counting.

If the image deteriorates, clean and adjust the microscope optics. If the problem persists, consult the microscope manufacturer.

11. Each set of samples taken will include 10 percent blanks or a minimum of 2 field blanks. These blanks must come from the same lot as the filters used for sample collection. The field blank results shall be averaged and subtracted from the analytical results before reporting. A set consists of any sample or group of samples for which an evaluation for this standard must be made. Any samples represented by a field blank having a fiber count in excess of the detection limit of the method being used shall be rejected.

12. The samples shall be mounted by the acetone/triacetin method or a method with an equivalent index of refraction and similar clarity.

13. Observe the following counting rules.

a. Count only fibers equal to or longer than 5 micrometers. Measure the length of curved fibers along the curve.

b. In the absence of other information, count all particles as asbestos that have a length-to-width ratio (aspect ratio) of 3:1 or greater.

c. Fibers lying entirely within the boundary of the Walton-Beckett graticule field shall receive a count of 1. Fibers crossing the boundary once, having one end within the circle, shall receive the count of one half (1/2). Do not count any fiber that crosses the graticule boundary more than once. Reject and do not count any other fibers even though they may be visible outside the graticule area.

d. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of an individual fiber.

e. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields; stop counting at 100 fields regardless of fiber count.

14. Blind recounts shall be conducted at the rate of 10 percent.

Quality Control Procedures

1. Intralaboratory program. Each laboratory and/or each company with more than one microscopist counting slides shall establish a statistically designed quality assurance program involving blind recounts and comparisons between microscopists to monitor the variability of counting by each microscopist and between microscopists. In a company with more than one laboratory, the program shall include all laboratories and shall also evaluate the laboratory-to-laboratory variability.

2.a. Interlaboratory program. Each laboratory analyzing asbestos samples for compliance determination shall implement an interlaboratory quality assurance program that as a minimum includes participation of at least two other independent laboratories. Each laboratory shall participate in round robin testing at least once every 6 months with at least all the other laboratories in its interlaboratory quality assurance group. Each laboratory shall submit slides typical of its own work load for use in this program. The round robin shall be designed and results analyzed using appropriate statistical methodology.

2.b. All laboratories should also participate in a national sample testing scheme such as the Proficiency Analytical Testing Program (PAT), or the Asbestos Registry sponsored by the American Industrial Hygiene Association (AIHA).

3. All individuals performing asbestos analysis must have taken the NIOSH course for sampling and evaluating airborne asbestos dust or an equivalent course.

4. When the use of different microscopes contributes to differences between counters and laboratories, the effect of the different microscope shall be evaluated and the microscope shall be replaced, as necessary.

5. Current results of these quality assurance programs shall be posted in each laboratory to keep the microscopists informed.

[57 FR 24330, June 8, 1992; 59 FR 40964, Aug. 10, 1994]

Next Standard (1910.1001 App B)

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Regulations (Standards - 29 CFR)

Detailed procedure for asbestos sampling and analysis - Non-Mandatory - 1910.1001 App B

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• **Part Number:**	1910
• **Part Title:**	Occupational Safety and Health Standards
• **Subpart:**	Z
• **Subpart Title:**	Toxic and Hazardous Substances
• **Standard Number:**	1910.1001 App B
• **Title:**	Detailed procedure for asbestos sampling and analysis - Non-Mandatory

This OSHA method ID-160

phase-contrast

Appendix B to §1910.1001 -- Detailed Procedures for Asbestos Sampling and Analysis -- Non-Mandatory

```
Matrix:
 OSHA Permissible Exposure Limits:
 Time Weighted Average.......................  0.1 fiber/cc
 Excursion Level (30 minutes)................  1.0 fiber/cc

Collection Procedure:
A known volume of air is drawn through a 25-mm diameter cassette
 containing a mixed-cellulose ester filter. The cassette must be
 equipped with an electrically conductive 50-mm extension cowl.
 The sampling time and rate are chosen to give a fiber density of
 between 100 to 1,300 fibers/mm(2) on the filter.

Recommended Sampling Rate...................... 0.5 to 5.0 liters/
                                                   minute (L/min)
Recommended Air Volumes:
 Minimum.....................................  25 L
 Maximum.....................................  2,400 L
```

Analytical Procedure: A portion of the sample filter is cleared and prepared for asbestos fiber counting by Phase Contrast Microscopy (PCM) at 400X.

Commercial manufacturers and products mentioned in this method are for descriptive use only and do not constitute endorsements by USDOL-OSHA. Similar products from other sources can be substituted.

1. Introduction

This method describes the collection of airborne asbestos fibers using calibrated sampling pumps with mixed-cellulose ester (MCE) filters and analysis by phase contrast microscopy (PCM). Some terms used are unique to this method and are defined below:

Asbestos: A term for naturally occurring fibrous minerals. Asbestos includes chrysotile, crocidolite, amosite (cummingtonite-grunerite asbestos), tremolite asbestos, actinolite asbestos, anthophyllite asbestos, and any of these minerals that have been chemically treated and/or altered. The precise chemical formulation of each species will vary with the location from which it was mined. Nominal compositions are listed:

```
Chrysotile............  Mg(3)Si(2)O(5)(OH)(4)
Crocidolite...........  Na(2)Fe(3)(2)(+)Fe(2)(3)(+)Si(8)O(22)(OH)(2)
Amosite...............  (Mg,Fe)(7)Si(8)O(22)(OH)(2)
Tremolite-actinolite..  Ca(2)(Mg,Fe)(5)Si(8)O(22)(OH)(2)
Anthophyllite.........  (Mg,Fe)(7)Si(8)O(22)(OH)(2)
```

Asbestos Fiber: A fiber of asbestos which meets the criteria specified below for a fiber.

Aspect Ratio: The ratio of the length of a fiber to it's diameter (e.g. 3:1, 5:1 aspect ratios).

Cleavage Fragments: Mineral particles formed by comminution of minerals, especially those characterized by parallel sides and a moderate aspect ratio (usually less than 20:1).

Detection Limit: The number of fibers necessary to be 95% certain that the result is greater than zero.

Differential Counting: The term applied to the practice of excluding certain kinds of fibers from the fiber count because they do not appear to be asbestos.

Fiber: A particle that is 5 um or longer, with a length-to-width ratio of 3 to 1 or longer.

Field: The area within the graticule circle that is superimposed on the microscope image.

Set: The samples which are taken, submitted to the laboratory, analyzed, and for which, interim or final result reports are generated.

Tremolite, Anthophyllite, and Actinolite: The non-asbestos form of these minerals which meet the definition of a fiber. It includes any of these minerals that have been chemically treated and/or altered.

Walton-Beckett Graticule: An eyepiece graticule specifically designed for asbestos fiber counting. It consists of a circle with a projected diameter of 100 + or - 2 um (area of about 0.00785 mm(2)) with a crosshair having tic-marks at 3-um intervals in one direction and 5-um in the orthogonal direction. There are marks around the periphery of the circle to demonstrate the proper sizes and shapes of fibers. This design is reproduced in Figure 1. The disk is placed in one of the microscope eyepieces so that the design is superimposed on the field of view.

1.1. History

Early surveys to determine asbestos exposures were conducted using impinger counts of total dust with the counts expressed as million particles per cubic foot. The British Asbestos Research Council recommended filter membrane counting in 1969. In July 1969, the Bureau of Occupational Safety and Health published a filter membrane method for counting asbestos fibers in the United States. This method was refined by NIOSH and published as P & CAM 239. On May 29, 1971, OSHA specified filter membrane sampling with phase contrast counting for evaluation of asbestos exposures at work sites in the United States. The use of this technique was again required by OSHA in 1986. Phase contrast microscopy has continued

to be the method of choice for the measurement of occupational exposure to asbestos.

1.2. Principle

Air is drawn through a MCE filter to capture airborne asbestos fibers. A wedge shaped portion of the filter is removed, placed on a glass microscope slide and made transparent. A measured area (field) is viewed by PCM. All the fibers meeting defined criteria for asbestos are counted and considered a measure of the airborne asbestos concentration.

1.3. Advantages and Disadvantages

There are four main advantages of PCM over other methods:

(1) The technique is specific for fibers. Phase contrast is a fiber counting technique which excludes non-fibrous particles from the analysis.

(2) The technique is inexpensive and does not require specialized knowledge to carry out the analysis for total fiber counts.

(3) The analysis is quick and can be performed on-site for rapid determination of air concentrations of asbestos fibers.

(4) The technique has continuity with historical epidemiological studies so that estimates of expected disease can be inferred from long-term determinations of asbestos exposures.

The main disadvantage of PCM is that it does not positively identify asbestos fibers. Other fibers which are not asbestos may be included in the count unless differential counting is performed. This requires a great deal of experience to adequately differentiate asbestos from non-asbestos fibers. Positive identification of asbestos must be performed by polarized light or electron microscopy techniques. A further disadvantage of PCM is that the smallest visible fibers are about 0.2 um in diameter while the finest asbestos fibers may be as small as 0.02 um in diameter. For some exposures, substantially more fibers may be present than are actually counted.

1.4. Workplace Exposure

Asbestos is used by the construction industry in such products as shingles, floor tiles, asbestos cement, roofing felts, insulation and acoustical products. Non-construction uses include brakes, clutch facings, paper, paints, plastics, and fabrics. One of the most significant exposures in the workplace is the removal and encapsulation of asbestos in schools, public buildings, and homes. Many workers have the potential to be exposed to asbestos during these operations.

About 95% of the asbestos in commercial use in the United States is chrysotile. Crocidolite and amosite make up most of the remainder. Anthophyllite and tremolite or actinolite are likely to be encountered as contaminants in various industrial products.

1.5. Physical Properties

Asbestos fiber possesses a high tensile strength along its axis, is chemically inert, non-combustible, and heat resistant. It has a high electrical resistance and good sound absorbing properties. It can be weaved into cables, fabrics or other textiles, and also matted into asbestos papers, felts, or mats.

2. Range and Detection Limit

2.1. The ideal counting range on the filter is 100 to 1,300 fibers/mm(2). With a Walton-Beckett graticule this range is equivalent to 0.8 to 10 fibers/field. Using NIOSH counting statistics, a count of 0.8 fibers/field would give an approximate coefficient of variation (CV) of 0.13.

2.2. The detection limit for this method is 4.0 fibers per 100 fields or 5.5 fibers/mm(2). This was determined using an equation to estimate the maximum CV possible at a specific concentration (95% confidence) and a Lower Control Limit of zero. The CV value was then used to determine a corresponding concentration from historical CV vs fiber relationships. As an example:

```
Lower Control Limit (95% Confidence) = AC - 1.645(CV)(AC)

Where:

AC = Estimate of the airborne fiber concentration (fibers/cc)
     Setting the Lower Control Limit = 0 and solving for CV:
0   = AC - 1.645(CV)(AC)
CV = 0.61
```

This value was compared with CV vs. count curves. The count at which CV = 0.61 for Leidel-Busch counting statistics or for an OSHA Salt Lake Technical Center (OSHA-SLTC) CV curve (see Appendix A for further information) was 4.4 fibers or 3.9 fibers per 100 fields, respectively. Although a lower detection limit of 4 fibers per 100 fields is supported by the OSHA-SLTC data, both data sets support the 4.5 fibers per 100 fields value.

3. Method Performance -- Precision and Accuracy

Precision is dependent upon the total number of fibers counted and the uniformity of the fiber distribution on the filter. A general rule is to count at least 20 and not more than 100 fields. The count is discontinued when 100 fibers are counted, provided that 20 fields have already been counted. Counting more than 100 fibers results in only a small gain in precision. As the total count drops below 10 fibers, an accelerated loss of precision is noted.

At this time, there is no known method to determine the absolute accuracy of the asbestos analysis. Results of samples prepared through the Proficiency Analytical Testing (PAT) Program and analyzed by the OSHA-SLTC showed no significant bias when compared to PAT reference values. The PAT samples were analyzed from 1987 to 1989 (N = 36) and the concentration range was from 120 to 1,300 fibers/mm$^{(2)}$.

4. Interferences

Fibrous substances, if present, may interfere with asbestos analysis.

Some common fibers are:

```
Fiberglass
Anhydrite
Plant Fibers
Perlite Veins
Gypsum
Some Synthetic Fibers
```

Membrane Structures
Sponge Spicules
Diatoms
Microorganisms
Wollastonite

The use of electron microscopy or optical tests such as polarized light, and dispersion staining may be used to differentiate these materials from asbestos when necessary.

5. Sampling

5.1. Equipment

5.1.1. Sample assembly (The assembly is shown in Figure 3). Conductive filter holder consisting of a 25-mm diameter, 3-piece cassette having a 50-mm long electrically conductive extension cowl. Backup pad, 25-mm, cellulose. Membrane filter, mixed-cellulose ester (MCE), 25-mm, plain, white, 0.4 to 1.2-um pore size.

Notes:

1. Do not re-use cassettes.
2. Fully conductive cassettes are required to reduce fiber loss to the sides of the cassette due to electrostatic attraction.
3. Purchase filters which have been selected by the manufacturer for asbestos counting or analyze representative filters for fiber background before use. Discard the filter lot if more than 4 fibers/100 fields are found.
4. To decrease the possibility of contamination, the sampling system (filter-backup pad-cassette) for asbestos is usually preassembled by the manufacturer.
5. Other cassettes, such as the Bell-mouth, may be used within the limits of their validation.

5.1.2. Gel bands for sealing cassettes.

5.1.3. Sampling pump.

Each pump must be a battery operated, self-contained unit small enough to be placed on the monitored employee and not interfere with the work being performed. The pump must be capable of sampling at the collection rate for the required sampling time.

5.1.4. Flexible tubing, 6-mm bore.

5.1.5. Pump calibration.

Stopwatch and bubble tube/burette or electronic meter.

5.2. Sampling Procedure

5.2.1. Seal the point where the base and cowl of each cassette meet with a gel band or tape.

5.2.2. Charge the pumps completely before beginning.

5.2.3. Connect each pump to a calibration cassette with an appropriate length of 6-mm bore plastic tubing. Do not use luer connectors -- the type of cassette specified above has built-in adapters.

5.2.4. Select an appropriate flow rate for the situation being monitored. The sampling flow rate must be between 0.5 and 5.0 L/min for personal sampling and is commonly set between 1 and 2 L/min. Always choose a flow rate that will not produce overloaded filters.

5.2.5. Calibrate each sampling pump before and after sampling with a calibration cassette in-line (Note: This calibration cassette should be from the same lot of cassettes used for sampling). Use a primary standard (e.g. bubble burette) to calibrate each pump. If possible, calibrate at the sampling site.

Note: If sampling site calibration is not possible, environmental influences may affect the flow rate. The extent is dependent on the type of pump used. Consult with the pump manufacturer to determine dependence on environmental influences. If the pump is affected by temperature and pressure changes, correct the flow rate using the formula shown in the section "Sampling Pump Flow Rate Corrections" at the end of this appendix.

5.2.6. Connect each pump to the base of each sampling cassette with flexible tubing. Remove the end cap of each cassette and take each air sample open face. Assure that each sample cassette is held open side down in the employee's breathing zone during sampling. The distance from the nose/mouth of the employee to the cassette should be about 10 cm. Secure the cassette on the collar or lapel of the employee using spring clips or other similar devices.

5.2.7. A suggested minimum air volume when sampling to determine TWA compliance is 25 L. For Excursion Limit (30 min sampling time) evaluations, a minimum air volume of 48 L is recommended.

5.2.8. The most significant problem when sampling for asbestos is overloading the filter with non-asbestos dust. Suggested maximum air sample volumes for specific environments are:

Environment	Air vol. (L)
Asbestos removal operations (visible dust).....	100
Asbestos removal operations (little dust).....	240
Office environments.....	400 to 2,400

Caution: Do not overload the filter with dust. High levels of non-fibrous dust particles may obscure fibers on the filter and lower the count or make counting impossible. If more than about 25 to 30% of the field area is obscured with dust, the result may be biased low. Smaller air volumes may be necessary when there is excessive non-asbestos dust in the air.

While sampling, observe the filter with a small flashlight. If there is a visible layer of dust on the filter, stop sampling, remove and seal the cassette, and replace with a new sampling assembly. The total dust loading should not exceed 1 mg.

5.2.9. Blank samples are used to determine if any contamination has occurred during sample handling. Prepare two blanks for the first 1 to 20 samples. For sets containing greater than 20

samples, prepare blanks as 10% of the samples. Handle blank samples in the same manner as air samples with one exception: Do not draw any air through the blank samples. Open the blank cassette in the place where the sample cassettes are mounted on the employee. Hold it open for about 30 seconds. Close and seal the cassette appropriately. Store blanks for shipment with the sample cassettes.

5.2.10. Immediately after sampling, close and seal each cassette with the base and plastic plugs. Do not touch or puncture the filter membrane as this will invalidate the analysis.

5.2.11. Attach and secure a sample seal around each sample cassette in such a way as to assure that the end cap and base plugs cannot be removed without destroying the seal. Tape the ends of the seal together since the seal is not long enough to be wrapped end-to-end. Also wrap tape around the cassette at each joint to keep the seal secure.

5.3. Sample Shipment

5.3.1. Send the samples to the laboratory with paperwork requesting asbestos analysis. List any known fibrous interferences present during sampling on the paperwork. Also, note the workplace operation(s) sampled.

5.3.2. Secure and handle the samples in such that they will not rattle during shipment nor be exposed to static electricity. Do not ship samples in expanded polystyrene peanuts, vermiculite, paper shreds, or excelsior. Tape sample cassettes to sheet bubbles and place in a container that will cushion the samples in such a manner that they will not rattle.

5.3.3. To avoid the possibility of sample contamination, always ship bulk samples in separate mailing containers.

6. Analysis

6.1. Safety Precautions

6.1.1. Acetone is extremely flammable and precautions must be taken not to ignite it. Avoid using large containers or quantities of acetone. Transfer the solvent in a ventilated laboratory hood. Do not use acetone near any open flame. For generation of acetone vapor, use a spark free heat source.

6.1.2. Any asbestos spills should be cleaned up immediately to prevent dispersal of fibers. Prudence should be exercised to avoid contamination of laboratory facilities or exposure of personnel to asbestos. Asbestos spills should be cleaned up with wet methods and/ or a High Efficiency Particulate-Air (HEPA) filtered vacuum.

Caution: Do not use a vacuum without a HEPA filter -- It will disperse fine asbestos fibers in the air.

6.2. Equipment

6.2.1. Phase contrast microscope with binocular or trinocular head.

6.2.2. Widefield or Huygenian 10X eyepieces (Note: The eyepiece containing the graticule must be a focusing eyepiece. Use a 40X phase objective with a numerical aperture of 0.65 to 0.75).

6.2.3. Kohler illumination (if possible) with green or blue filter.

6.2.4. Walton-Beckett Graticule, type G-22 with 100 plus or minus 2 um projected diameter.

6.2.5. Mechanical stage.

A rotating mechanical stage is convenient for use with polarized light.

6.2.6. Phase telescope.

6.2.7. Stage micrometer with 0.01-mm subdivisions.

6.2.8. Phase-shift test slide, mark II (Available from PTR optics Ltd., and also McCrone).

6.2.9. Precleaned glass slides, 25 mm X 75 mm. One end can be frosted for convenience in writing sample numbers, etc., or paste-on labels can be used.

6.2.10. Cover glass #1 1/2.

6.2.11. Scalpel (#10, curved blade).

6.2.12. Fine tipped forceps.

6.2.13. Aluminum block for clearing filter (see Appendix D and Figure 4).

6.2.14. Automatic adjustable pipette, 100- to 500-uL.

6.2.15. Micropipette, 5 uL.

6.3. Reagents

6.3.1. Acetone (HPLC grade).

6.3.2. Triacetin (glycerol triacetate).

6.3.3. Lacquer or nail polish.

6.4. Standard Preparation

A way to prepare standard asbestos samples of known concentration has not been developed. It is possible to prepare replicate samples of nearly equal concentration. This has been performed through the PAT program. These asbestos samples are distributed by the AIHA to participating laboratories.

Since only about one-fourth of a 25-mm sample membrane is required for an asbestos count, any PAT sample can serve as a "standard" for replicate counting.

6.5. Sample Mounting

Note: See Safety Precautions in Section 6.1. before proceeding. The objective is to produce samples with a smooth (non-grainy) background in a medium with a refractive index of approximately 1.46. The technique below collapses the filter for easier focusing and produces

permanent mounts which are useful for quality control and interlaboratory comparison.

An aluminum block or similar device is required for sample preparation.

1. Heat the aluminum block to about 70 deg. C. The hot block should not be used on any surface that can be damaged by either the heat or from exposure to acetone.

6.5.2. Ensure that the glass slides and cover glasses are free of dust and fibers.

6.5.3. Remove the top plug to prevent a vacuum when the cassette is opened. Clean the outside of the cassette if necessary. Cut the seal and/or tape on the cassette with a razor blade. Very carefully separate the base from the extension cowl, leaving the filter and backup pad in the base.

6.5.4. With a rocking motion cut a triangular wedge from the filter using the scalpel. This wedge should be one-sixth to one-fourth of the filter. Grasp the filter wedge with the forceps on the perimeter of the filter which was clamped between the cassette pieces. DO NOT TOUCH the filter with your finger. Place the filter on the glass slide sample side up. Static electricity will usually keep the filter on the slide until it is cleared.

6.5.5. Place the tip of the micropipette containing about 200 uL acetone into the aluminum block. Insert the glass slide into the receiving slot in the aluminum block. Inject the acetone into the block with slow, steady pressure on the plunger while holding the pipette firmly in place. Wait 3 to 5 seconds for the filter to clear, then remove the pipette and slide from the aluminum block.

6. Immediately (less than 30 seconds) place 2.5 to 3.5 uL of triacetin on the filter (Note: iting longer than 30 seconds will result in increased index of refraction and decreased contrast between the fibers and the preparation. This may also lead to separation of the cover slip from the slide).

6.5.7. Lower a cover slip gently onto the filter at a slight angle to reduce the possibility of forming air bubbles. If more than 30 seconds have elapsed between acetone exposure and triacetin application, glue the edges of the cover slip to the slide with lacquer or nail polish.

6.5.8. If clearing is slow, warm the slide for 15 min on a hot plate having a surface temperature of about 50 deg. C to hasten clearing. The top of the hot block can be used if the slide is not heated too long.

6.5.9. Counting may proceed immediately after clearing and mounting are completed.

6.6. Sample Analysis

Completely align the microscope according to the manufacturer's instructions. Then, align the microscope using the following general alignment routine at the beginning of every counting session and more often if necessary.

6.6.1. Alignment

Clean all optical surfaces. Even a small amount of dirt can significantly degrade the image.

(2) Rough focus the objective on a sample.

(3) Close down the field iris so that it is visible in the field of view. Focus the image of the iris with the condenser focus. Center the image of the iris in the field of view.

(4) Install the phase telescope and focus on the phase rings. Critically center the rings. Misalignment of the rings results in astigmatism which will degrade the image.

(5) Place the phase-shift test slide on the microscope stage and focus on the lines. The analyst must see line set 3 and should see at least parts of 4 and 5 but, not see line set 6 or 6. A microscope/microscopist combination which does not pass this test may not be used.

6.6.2. Counting Fibers

(1) Place the prepared sample slide on the mechanical stage of the microscope. Position the center of the wedge under the objective lens and focus upon the sample.

(2) Start counting from one end of the wedge and progress along a radial line to the other end (count in either direction from perimeter to wedge tip). Select fields randomly, without looking into the eyepieces, by slightly advancing the slide in one direction with the mechanical stage control.

(3) Continually scan over a range of focal planes (generally the upper 10 to 15 um of the filter surface) with the fine focus control during each field count. Spend at least 5 to 15 seconds per field.

(4) Most samples will contain asbestos fibers with fiber diameters less than 1 um. Look carefully for faint fiber images. The small diameter fibers will be very hard to see. However, they are an important contribution to the total count.

(5) Count only fibers equal to or longer than 5 um. Measure the length of curved fibers along the curve.

(6) Count fibers which have a length to width ratio of 3:1 or greater.

(7) Count all the fibers in at least 20 fields. Continue counting until either 100 fibers are counted or 100 fields have been viewed; whichever occurs first. Count all the fibers in the final field.

(8) Fibers lying entirely within the boundary of the Walton-Beckett graticule field shall receive a count of 1. Fibers crossing the boundary once, having one end within the circle shall receive a count of 1/2. Do not count any fiber that crosses the graticule boundary more than once. Reject and do not count any other fibers even though they may be visible outside the graticule area. If a fiber touches the circle, it is considered to cross the line.

(9) Count bundles of fibers as one fiber unless individual fibers can be clearly identified and each individual fiber is clearly not connected to another counted fiber. See Figure 1 for counting conventions.

(10) Record the number of fibers in each field in a consistent way such that filter non-uniformity can be assessed.

(11) Regularly check phase ring alignment.

(12) When an agglomerate (mass of material) covers more than 25% of the field of view,

reject the field and select another. Do not include it in the number of fields counted.

(13) Perform a "blind recount" of 1 in every 10 filter wedges (slides). Re-label the slides using a person other than the original counter.

6.7. Fiber Identification

As previously mentioned in Section 1.3., PCM does not provide positive confirmation of asbestos fibers. Alternate differential counting techniques should be used if discrimination is desirable. Differential counting may include primary discrimination based on morphology, polarized light analysis of fibers, or modification of PCM data by Scanning Electron or Transmission Electron Microscopy.

A great deal of experience is required to routinely and correctly perform differential counting. It is discouraged unless it is legally necessary. Then, only if a fiber is obviously not asbestos should it be excluded from the count. Further discussion of this technique can be found in reference 8.10.

If there is a question whether a fiber is asbestos or not, follow the rule:

"WHEN IN DOUBT, COUNT."

6.8. Analytical Recommendations -- Quality Control System

6.8.1. All individuals performing asbestos analysis must have taken the NIOSH course for sampling and evaluating airborne asbestos or an equivalent course.

6.8.2. Each laboratory engaged in asbestos counting shall set up a slide trading arrangement with at least two other laboratories in order to compare performance and eliminate inbreeding of error. The slide exchange occurs at least semiannually. The round robin results shall be posted where all analysts can view individual analyst's results.

6.8.3. Each laboratory engaged in asbestos counting shall participate in the Proficiency Analytical Testing Program, the Asbestos Analyst Registry or equivalent.

6.8.4. Each analyst shall select and count prepared slides from a "slide bank". These are quality assurance counts. The slide bank shall be prepared using uniformly distributed samples taken from the workload. Fiber densities should cover the entire range routinely analyzed by the laboratory. These slides are counted blind by all counters to establish an original standard deviation. This historical distribution is compared with the quality assurance counts. A counter must have 95% of all quality control samples counted within three standard deviations of the historical mean. This count is then integrated into a new historical mean and standard deviation for the slide.

The analyses done by the counters to establish the slide bank may be used for an interim quality control program if the data are treated in a proper statistical fashion.

7. CALCULATIONS

7.1. Calculate the estimated airborne asbestos fiber concentration on the filter sample using the following formula:

Where:

AC = Airborne fiber concentration

(For Equation A, Click Here)

[illegible] = Total number of fibers greater than 5 um counted
[illegible] = Total number of fields counted on the filter
BFB = Total number of fibers greater than 5 um counted in the blank
BFL = Total number of fields counted on the blank
ECA = Effective collecting area of filter (385 mm(2) nominal for a 25-mm filter.)
FR = Pump flow rate (L/min)
MFA = Microscope count field area (mm(2)). This is 0.00785 mm(2) for a Walton-Beckett Graticule.
T = Sample collection time (min)
1,000 = Conversion of L to cc

Note: The collection area of a filter is seldom equal to 385 mm(2). It is appropriate for laboratories to routinely monitor the exact diameter using an inside micrometer. The collection area is calculated according to the formula:

Area = pi(d/2)(2)

7.2. Short-cut Calculation

Since a given analyst always has the same interpupillary distance, the number of fields per filter for a particular analyst will remain constant for a given size filter. The field size for that analyst is constant (i.e. the analyst is using an assigned microscope and is not changing the [illegible]cle).

For example, if the exposed area of the filter is always 385 mm(2) and the size of the field is always 0.00785 mm(2), the number of fields per filter will always be 49,000. In addition it is necessary to convert liters of air to cc. These three constants can then be combined such that ECA/(1,000 X MFA) = 49. The previous equation simplifies to:

(For Equation B, Click Here)

7.3. Recount Calculations

As mentioned in step 13 of Section 6.6.2., a "blind recount" of 10% of the slides is performed. In all cases, differences will be observed between the first and second counts of the same filter wedge. Most of these differences will be due to chance alone, that is, due to the random variability (precision) of the count method. Statistical recount criteria enables one to decide whether observed differences can be explained due to chance alone or are probably due to systematic differences between analysts, microscopes, or other biasing factors.

The following recount criterion is for a pair of counts that estimate AC in fibers/cc. The criterion is given at the type-I error level. That is, there is 5% maximum risk that we will reject a pair of counts for the reason that one might be biased, when the large observed difference is really due to chance.

[illegible]ject a pair of counts if:

(For Equation C, Click Here)

Where:

AC1 = lower estimated airborne fiber concentration
AC2 = higher estimated airborne fiber concentration
Cavg = average of the two concentration estimates
CV(FB) = CV for the average of the two concentration estimates

If a pair of counts are rejected by this criterion then, recount the rest of the filters in the submitted set. Apply the test and reject any other pairs failing the test. Rejection shall include a memo to the industrial hygienist stating that the sample failed a statistical test for homogeneity and the true air concentration may be significantly different than the reported value.

7.4. Reporting Results

Report results to the industrial hygienist as fibers/cc. Use two significant figures. If multiple analyses are performed on a sample, an average of the results is to be reported unless any of the results can be rejected for cause.

8. References

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8.3. Bayer, S.G., Zumwalde, R.D., Brown, T.A., Equipment and Procedure for Mounting Millipore Filters and Counting Asbestos Fibers by Phase Contrast Microscopy, Bureau of Occupational Health, U.S. Dept. of Health, Education and Welfare, Cincinnati, OH, 1969.

8.4. NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1 (DHEW/ NIOSH Pub. No. 77-157-A). National Institute for Occupational Safety and Health, Cincinnati, OH, 1977. pp. 239-1-239-21.

8.5. Asbestos, Code of Federal Regulations 29 CFR 1910.1001. 1971.

8.6. Occupational Exposure to Asbestos, Tremolite, Anthophyllite, and Actinolite. Final Rule, Federal Register 51:119 (20 June 1986). pp. 22612-22790.

8.7. Asbestos, Tremolite, Anthophyllite, and Actinolite, Code of Federal Regulations 1910.1001. 1988. pp 711-752.

8.8. Criteria for a Recommended Standard -- Occupational Exposure to Asbestos (DHEW/NIOSH Pub. No. HSM 72-10267), National Institute for Occupational Safety and Health NIOSH, Cincinnati,OH, 1972. pp. III-1-III-24.

Leidel, N.A., Bayer,S.G., Zumwalde, R.D.,Busch, K.A., USPHS/NIOSH Membrane Filter Method for Evaluating Airborne Asbestos Fibers (DHEW/NIOSH Pub. No. 79-127). National Institute for Occupational Safety and Health, Cincinnati, OH, 1979.

8.10. Dixon, W.C., Applications of Optical Microscopy in Analysis of Asbestos and Quartz, Analytical Techniques in Occupational Health Chemistry, edited by D.D. Dollberg and A.W. Verstuyft. Wash. D.C.: American Chemical Society, (ACS Symposium Series 120) 1980. pp. 13-41.

Quality Control

The OSHA asbestos regulations require each laboratory to establish a quality control program. The following is presented as an example of how the OSHA-SLTC constructed its internal CV curve as part of meeting this requirement. Data is from 395 samples collected during OSHA compliance inspections and analyzed from October 1980 through April 1986.

Each sample was counted by 2 to 5 different counters independently of one another. The standard deviation and the CV statistic was calculated for each sample. This data was then plotted on a graph of CV vs. fibers/mm(2). A least squares regression was performed using the following equation:

```
CV = antilog1(10)[A(log(10)(x))(2)+B(log(10)(x))+C]

where:

 x = the number of fibers/mm(2)

Application of least squares gave:

 A = 0.182205
 B = -0.973343
 C = 0.327499

Using these values, the equation becomes:

 CV = antilog(10)[0.182205(log(10)(x))(2)-0.973343(log(10)(x))+0.327499]
```

Sampling Pump Flow Rate Corrections

This correction is used if a difference greater than 5% in ambient temperature and/or pressure is noted between calibration and sampling sites and the pump does not compensate for the differences.

(For Equation D, Click Here)

```
Where:

Q(act) = actual flow rate
Q(cal) = calibrated flow rate (if a rotameter was used, the rotameter
         value)
P(cal) = uncorrected air pressure at calibration
P(act) = uncorrected air pressure at sampling site
T(act) = temperature at sampling site (K)
T(cal) = temperature at calibration (K)
```

Walton-Beckett Graticule

When ordering the Graticule for asbestos counting, specify the exact disc diameter needed to fit the ocular of the microscope and the diameter (mm) of the circular counting area.

Instructions for measuring the dimensions necessary are listed:

(1) Insert any available graticule into the focusing eyepiece and focus so that the graticule lines are sharp and clear.

(2) Align the microscope.

(3) Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.

(4) Measure the magnified grid length, PL (um), using the stage micrometer.

(5) Remove the graticule from the microscope and measure its actual grid length, AL (mm). This can be accomplished by using a mechanical stage fitted with verniers, or a jeweler's loupe with a direct reading scale.

(6) Let D = 100 um. Calculate the circle diameter, d(c)(mm), for the Walton-Beckett graticule and specify the diameter when making a purchase:

$$d(c) = \frac{AL \times D}{PL}$$

Example: If PL = 108 um, AL = 2.93 mm and D = 100 um, then,

$$d(c) = \frac{2.93 \times 100}{108} = 2.71\text{mm}$$

(7) Each eyepiece-objective-reticle combination on the microscope must be calibrated. Should any of the three be changed (by zoom adjustment, disassembly, replacement, etc.), the combination must be recalibrated. Calibration may change if interpupillary distance is changed. Measure the field diameter, D (acceptable range: 100 plus or minus 2 um) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine the field area (mm(2)).

```
Field Area = pi(D/2)(2)
If D = 100 um = 0.1 mm, then
Field Area = pi(0.1 mm/2)(2) = 0.00785 mm(2)
```

The Graticule is available from: Graticules Ltd., Morley Road, Tonbridge TN9 IRN, Kent, England (Telephone 011-44-732-359061). Also available from PTR Optics Ltd., 145 Newton Street, Waltham, MA 02154 [telephone (617) 891-6000] or McCrone Accessories and Components, 2506 S. Michigan Ave., Chicago, IL 60616 [phone (312)-842-7100]. The graticule is custom made for each microscope.

Counts for the Fibers in the Figure

Structure No.	Count	Explanation
1 to 6.....	1	Single fibers all contained within the circle.

7.....	1/2	Fiber crosses circle once.
8.....	0	Fiber too short.
9.....	2	Two crossing fibers.
10.....	0	Fiber outside graticule.
11.....	0	Fiber crosses graticule twice.
[illegible].....	1/2	Although split, fiber only crosses once.

(For Figure 1 of Walton-Beckett Graticule, Click Here)

[57 FR 24330, June 8, 1992; 59 FR 40964, Aug. 10, 1994; 60 FR 33972, June 29, 1995]

Next Standard (1910.1001 App C)

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Occupational Safety & Health Administration
200 Constitution Avenue, NW
Washington, DC 20210

ASBESTOS and OTHER FIBERS by PCM 7400

Various MW: Various CAS: Various RTECS: Various

METHOD: 7400, Issue 2 EVALUATION: FULL Issue 1: Rev. 3 on 15 May 1989
Issue 2: 15 August 1994

OSHA : 0.1 asbestos fiber (> 5 µm long)/cc; 1 f/cc/30 min excursion; carcinogen
MSHA: 2 asbestos fibers/cc
NIOSH: 0.1 f/cc (fibers > 5 µm long)/400 L; carcinogen
ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, fibers/cc; carcinogen

PROPERTIES: solid, fibrous, crystalline, anisotropic

SYNONYMS [CAS #]: actinolite [77536-66-4] or ferroactinolite [15669-07-5]; amosite [12172-73-5]; anthophyllite [77536-67-5]; chrysotile [12001-29-5]; serpentine [18786-24-8]; crocidolite [12001-28-4]; tremolite [77536-68-6]; amphibole asbestos [1332-21-4]; refractory ceramic fibers [142844-00-6]; fibrous glass.

SAMPLING

SAMPLER: FILTER (0.45- to 1.2-µm cellulose ester membrane, 25-mm; conductive cowl on cassette)

FLOW RATE*: 0.5 to 16 L/min

VOL-MIN*: -MAX*: 400 L @ 0.1 fiber/cc (step 4, sampling) *Adjust to give 100 to 1300 fiber/mm²

SHIPMENT: routine (pack to reduce shock)

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 80 to 100 fibers counted

BIAS: See EVALUATION OF METHOD

OVERALL PRECISION ($\hat{S}_{rT}$): 0.115 to 0.13 [1]

ACCURACY: See EVALUATION OF METHOD

MEASUREMENT

TECHNIQUE: LIGHT MICROSCOPY, PHASE CONTRAST

ANALYTE: fibers (manual count)

SAMPLE PREPARATION: acetone - collapse/triacetin - immersion

COUNTING RULES: described in previous version of this method as "A" rules [1,3]

EQUIPMENT:
1. positive phase-contrast microscope
2. Walton-Beckett graticule (100-µm field of view) Type G-22
3. phase-shift test slide (HSE/NPL)

CALIBRATION: HSE/NPL test slide

RANGE: 100 to 1300 fibers/mm² filter area

ESTIMATED LOD: 7 fibers/mm² filter area

PRECISION ($\bar{S}_r$): 0.10 to 0.12 [1]; see EVALUATION OF METHOD

APPLICABILITY: The quantitative working range is 0.04 to 0.5 fiber/cc for a 1000-L air sample. The LOD depends on sample volume and quantity of interfering dust, and is <0.01 fiber/cc for atmospheres free of interferences. The method gives an index of airborne fibers. It is primarily used for estimating asbestos concentrations, though PCM does not differentiate between asbestos and other fibers. Use this method in conjunction with electron microscopy (e.g., Method 7402) for assistance in identification of fibers. Fibers < ca. 0.25 µm diameter will not be detected by this method [4]. This method may be used for other materials such as fibrous glass by using alternate counting rules (see Appendix C).

INTERFERENCES: If the method is used to detect a specific type of fiber, any other airborne fiber may interfere since all particles meeting the counting criteria are counted. Chain-like particles may appear fibrous. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

OTHER METHODS: This revision replaces Method 7400, Revision #3 (date 5/15/89).

REAGENTS:

1. Acetone,* reagent grade.
2. Triacetin (glycerol triacetate), reagent grade.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically conductive extension cowl and cellulose ester filter, 0.45- to 1.2-µm pore size, and backup pad.

   NOTE 1: Analyze representative filters for fiber background before use to check for clarity and background. Discard the filter lot if mean is ≥5 fibers per 100 graticule fields. These are defined as laboratory blanks. Manufacturer-provided quality assurance checks on filter blanks are normally adequate as long as field blanks are analyzed as described below.

   NOTE 2: The electrically conductive extension cowl reduces electrostatic effects. Ground the cowl when possible during sampling.

   NOTE 3: Use 0.8-µm pore size filters for personal sampling. The 0.45-µm filters are recommended for sampling when performing TEM analysis on the same samples. However, their higher pressure drop precludes their use with personal sampling pumps.

   NOTE 4: Other cassettes have been proposed that exhibit improved uniformity of fiber deposit on the filter surface, e.g., bellmouthed sampler (Envirometrics, Charleston, SC). These may be used if shown to give measured concentrations equivalent to sampler indicated above for the application.
2. Personal sampling pump, battery or line-powered vacuum, of sufficient capacity to meet flow-rate requirements (see step 4 for flow rate), with flexible connecting tubing.
3. Wire, multi-stranded, 22-gauge; 1", hose clamp to attach wire to cassette.
4. Tape, shrink- or adhesive-.
5. Slides, glass, frosted-end, pre-cleaned, 25 x 75-mm.
6. Cover slips, 22- x 22-mm, No. 1-1/2, unless otherwise specified by microscope manufacturer.
7. Lacquer or nail polish.
8. Knife, #10 surgical steel, curved blade.
9. Tweezers.

EQUIPMENT:

10. Acetone flash vaporization system for clearing filters on glass slides (see ref. [5] for specifications or see manufacturer's instructions for equivalent devices).
11. Micropipets or syringes, 5-µL and 100- to 500-µL.
12. Microscope, positive phase (dark) contrast, with green or blue filter, adjustable field iris, 8 to 10X eyepiece, and 40 to 45X phase objective (total magnification ca. 400X); numerical aperture = 0.65 to 0.75.
13. Graticule, Walton-Beckett type with 100-µm diameter circular field (area = 0.00785 mm2) at the specimen plane (Type G-22). Available from Optometrics USA, P.O. Box 699, Ayer, MA 01432 [phone (508)-772-1700], and McCrone Accessories and Components, 850 Pasquinelli Drive, Westmont, IL 60559 [phone (312) 887-7100].
    NOTE: The graticule is custom-made for each microscope. (see APPENDIX A for the custom-ordering procedure).
14. HSE/NPL phase contrast test slide, Mark II. Available from Optometrics USA (address above).
15. Telescope, ocular phase-ring centering.
16. Stage micrometer (0.01-mm divisions).

SPECIAL PRECAUTIONS: Acetone is extremely flammable. Take precautions not to ignite it. Heating of acetone in volumes greater than 1 mL must be done in a ventilated laboratory fume hood using a flameless, spark- free heat source.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. To reduce contamination and to hold the cassette tightly together, seal the crease between the cassette base and the cowl with a shrink band or light colored adhesive tape. For personal sampling, fasten the (uncapped) open-face cassette to the worker's lapel. The open face should be oriented downward.
   NOTE: The cowl should be electrically grounded during area sampling, especially under conditions of low relative humidity. Use a hose clamp to secure one end of the wire (Equipment, Item 3) to the monitor's cowl. Connect the other end to an earth ground (i.e., cold water pipe).
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Handle field blanks in a manner representative of actual handling of associated samples in the set. Open field blank cassettes at the same time as other cassettes just prior to sampling. Store top covers and cassettes in a clean area (e.g., a closed bag or box) with the top covers from the sampling cassettes during the sampling period.
4. Sample at 0.5 L/min or greater [6]. Adjust sampling flow rate, Q (L/min), and time, t (min), to produce a fiber density, E, of 100 to 1300 fibers/mm^2 ($3.85 \cdot 10^4$ to $5 \cdot 10^5$ fibers per 25-mm filter with effective collection area A_c= 385 mm^2) for optimum accuracy. These variables are related to the action level (one-half the current standard), L (fibers/cc), of the fibrous aerosol being sampled by:

$$t = \frac{A_c \cdot E}{Q \cdot L \cdot 10^3}, \text{ min.}$$

NOTE 1: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. The collection efficiency does not appear to be a function of flow rate in the range of 0.5 to 16 L/min for asbestos fibers [7]. Relatively large diameter fibers (>3 µm) may exhibit significant aspiration loss and inlet deposition. A sampling rate of 1 to 4 L/min for 8 h is appropriate in atmospheres containing ca. 0.1 fiber/cc in the absence of significant amounts of non-asbestos dust. Dusty atmospheres require smaller sample volumes (≤400 L) to obtain countable samples. In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If ≥ 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration.

NOTE 2: OSHA regulations specify a minimum sampling volume of 48 L for an excursion measurement, and a maximum sampling rate of 2.5 L/min [3].

5. At the end of sampling, replace top cover and end plugs.
6. Ship samples with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.
   NOTE: Do not use untreated polystyrene foam in shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

NOTE 1: The object is to produce samples with a smooth (non-grainy) background in a medium with refractive index ≤1.46. This method collapses the filter for easier focusing and produces permanent (1 - 10 years) mounts which are useful for quality control and interlaboratory comparison. The aluminum "hot block" or similar flash vaporization techniques may be used outside the laboratory [2]. Other mounting techniques meeting the above criteria may also be used (e.g., the laboratory fume hood procedure for generating acetone vapor as described in Method 7400 - revision of 5/15/85, or the non-permanent field mounting technique used in P&CAM 239 [3,7,8,9]). Unless the effective filtration area is known, determine the area and record the information referenced against the sample ID number [1,9,10,11].

NOTE 2: Excessive water in the acetone may slow the clearing of the filter, causing material to be washed off the surface of the filter. Also, filters that have been exposed to high humidities prior to clearing may have a grainy background.

7. Ensure that the glass slides and cover slips are free of dust and fibers.
8. Adjust the rheostat to heat the "hot block" to ca. 70 °C [2].
   NOTE: If the "hot block" is not used in a fume hood, it must rest on a ceramic plate and be isolated from any surface susceptible to heat damage.
9. Mount a wedge cut from the sample filter on a clean glass slide.
   a. Cut wedges of ca. 25% of the filter area with a curved-blade surgical steel knife using a rocking motion to prevent tearing. Place wedge, dust side up, on slide.
      NOTE: Static electricity will usually keep the wedge on the slide.

b. Insert slide with wedge into the receiving slot at base of "hot block". Immediately place tip of a micropipet containing ca. 250 µL acetone (use the minimum volume needed to consistently clear the filter sections) into the inlet port of the PTFE cap on top of the "hot block" and inject the acetone into the vaporization chamber with a slow, steady pressure on the plunger button while holding pipet firmly in place. After waiting 3 to 5 sec for the filter to clear, remove pipet and slide from their ports.

CAUTION: Although the volume of acetone used is small, use safety precautions. Work in a well-ventilated area (e.g., laboratory fume hood). Take care not to ignite the acetone. Continuous use of this device in an unventilated space may produce explosive acetone vapor concentrations.

c. Using the 5-µL micropipet, immediately place 3.0 to 3.5 µL triacetin on the wedge. Gently lower a clean cover slip onto the wedge at a slight angle to reduce bubble formation. Avoid excess pressure and movement of the cover glass.

NOTE: If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours. If excessive triacetin remains at the edge of the filter under the cover slip, fiber migration may occur.

d. Mark the outline of the filter segment with a glass marking pen to aid in microscopic evaluation.

e. Glue the edges of the cover slip to the slide using lacquer or nail polish [12]. Counting may proceed immediately after clearing and mounting are completed.

NOTE: If clearing is slow, warm the slide on a hotplate (surface temperature 50 °C) for up to 15 min to hasten clearing. Heat carefully to prevent gas bubble formation.

CALIBRATION AND QUALITY CONTROL:

10. Microscope adjustments. Follow the manufacturers instructions. At least once daily use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric. With each microscope, keep a logbook in which to record the dates of microscope cleanings and major servicing.

a. Each time a sample is examined, do the following:

(1) Adjust the light source for even illumination across the field of view at the condenser iris. Use Kohler illumination, if available. With some microscopes, the illumination may have to be set up with bright field optics rather than phase contract optics.

(2) Focus on the particulate material to be examined.

(3) Make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.

b. Check the phase-shift detection limit of the microscope periodically for each analyst/microscope combination:

(1) Center the HSE/NPL phase-contrast test slide under the phase objective.

(2) Bring the blocks of grooved lines into focus in the graticule area.

NOTE: The slide contains seven blocks of grooves (ca. 20 grooves per block) in descending order of visibility. For asbestos counting the microscope optics must completely resolve the grooved lines in block 3 although they may appear somewhat faint, and the grooved lines in blocks 6 and 7 must be invisible when centered in the graticule area. Blocks 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope which fails to meet these requirements has resolution either too low or too high for fiber counting.

(3) If image quality deteriorates, clean the microscope optics. If the problem persists, consult the microscope manufacturer.

11. Document the laboratory's precision for each counter for replicate fiber counts.

a. Maintain as part of the laboratory quality assurance program a set of reference slides to be used on a daily basis [13]. These slides should consist of filter preparations including a range of loadings and background dust levels from a variety of sources including both field and reference samples (e.g., PAT, AAR, commercial samples). The Quality Assurance Officer

should maintain custody of the reference slides and should supply each counter with a minimum of one reference slide per workday. Change the labels on the reference slides periodically so that the counter does not become familiar with the samples.

b. From blind repeat counts on reference slides, estimate the laboratory intra- and intercounter precision. Obtain separate values of relative standard deviation (S_r) for each sample matrix analyzed in each of the following ranges: 5 to 20 fibers in 100 graticule fields, >20 to 50 fibers in 100 graticule fields, and >50 to 100 fibers in 100 graticule fields. Maintain control charts for each of these data files.

NOTE: Certain sample matrices (e.g., asbestos cement) have been shown to give poor precision [9]

12. Prepare and count field blanks along with the field samples. Report counts on each field blank.

NOTE 1: The identity of blank filters should be unknown to the counter until all counts have been completed.

NOTE 2: If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.

13. Perform blind recounts by the same counter on 10% of filters counted (slides relabeled by a person other than the counter). Use the following test to determine whether a pair of counts by the same counter on the same filter should be rejected because of possible bias: Discard the sample if the absolute value of the difference between the square roots of the two counts (in fiber/mm^2) exceeds 2.77 (X)S_r', where X = average of the square roots of the two fiber counts (in fiber/mm^2) and $S_r' = \frac{S_r}{2}$, where S_r is the intracounter relative standard deviation for the appropriate count range (in fibers) determined in step 11. For more complete discussions see reference [13].

NOTE 1: Since fiber counting is the measurement of randomly placed fibers which may be described by a Poisson distribution, a square root transformation of the fiber count data will result in approximately normally distributed data [13].

NOTE 2: If a pair of counts is rejected by this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts. It is not necessary to use this statistic on blank counts.

14. The analyst is a critical part of this analytical procedure. Care must be taken to provide a non-stressful and comfortable environment for fiber counting. An ergonomically designed chair should be used, with the microscope eyepiece situated at a comfortable height for viewing. External lighting should be set at a level similar to the illumination level in the microscope to reduce eye fatigue. In addition, counters should take 10-to-20 minute breaks from the microscope every one or two hours to limit fatigue [14]. During these breaks, both eye and upper back/neck exercises should be performed to relieve strain.

15. All laboratories engaged in asbestos counting should participate in a proficiency testing program such as the AIHA-NIOSH Proficiency Analytical Testing (PAT) Program for asbestos and routinely exchange field samples with other laboratories to compare performance of counters.

MEASUREMENT:

16. Center the slide on the stage of the calibrated microscope under the objective lens. Focus the microscope on the plane of the filter.

17. Adjust the microscope (Step 10).

NOTE: Calibration with the HSE/NPL test slide determines the minimum detectable fiber diameter (ca. 0.25 µm) [4].

18. Counting rules: (same as P&CAM 239 rules [1,10,11]: see examples in APPENDIX B).

a. Count any fiber longer than 5 µm which lies entirely within the graticule area.

(1) Count only fibers longer than 5 µm. Measure length of curved fibers along the curve.

(2) Count only fibers with a length-to-width ratio equal to or greater than 3:1.

b. For fibers which cross the boundary of the graticule field:

(1) Count as 1/2 fiber any fiber with only one end lying within the graticule area, provided that the fiber meets the criteria of rule a above.

(2) Do not count any fiber which crosses the graticule boundary more than once.
(3) Reject and do not count all other fibers.

c. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.

d. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 graticule fields regardless of count.

19. Start counting from the tip of the filter wedge and progress along a radial line to the outer edge. Shift up or down on the filter, and continue in the reverse direction. Select graticule fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. Ensure that, as a minimum, each analysis covers one radial line from the filter center to the outer edge of the filter. When an agglomerate or bubble covers ca. 1/6 or more of the graticule field, reject the graticule field and select another. Do not report rejected graticule fields in the total number counted.

NOTE 1: When counting a graticule field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count. A minimum counting time of 15 seconds per field is appropriate for accurate counting.

NOTE 2: This method does not allow for differentiation of fibers based on morphology. Although some experienced counters are capable of selectively counting only fibers which appear to be asbestiform, there is presently no accepted method for ensuring uniformity of judgment between laboratories. It is, therefore, incumbent upon all laboratories using this method to report total fiber counts. If serious contamination from non-asbestos fibers occurs in samples, other techniques such as transmission electron microscopy must be used to identify the asbestos fiber fraction present in the sample (see NIOSH Method 7402). In some cases (i.e., for fibers with diameters >1 µm), polarized light microscopy (as in NIOSH Method 7403) may be used to identify and eliminate interfering non-crystalline fibers [15].

NOTE 3: Do not count at edges where filter was cut. Move in at least 1 mm from the edge.

NOTE 4: Under certain conditions, electrostatic charge may affect the sampling of fibers. These electrostatic effects are most likely to occur when the relative humidity is low (below 20%), and when sampling is performed near the source of aerosol. The result is that deposition of fibers on the filter is reduced, especially near the edge of the filter. If such a pattern is noted during fiber counting, choose fields as close to the center of the filter as possible [5].

NOTE 5: Counts are to be recorded on a data sheet that provides, as a minimum, spaces on which to record the counts for each field, filter identification number, analyst's name, date, total fibers counted, total fields counted, average count, fiber density, and commentary. Average count is calculated by dividing the total fiber count by the number of fields observed. Fiber density (fibers/mm^2) is defined as the average count (fibers/field) divided by the field (graticule) area (mm^2/field).

CALCULATIONS AND REPORTING OF RESULTS

20. Calculate and report fiber density on the filter, E (fibers/mm^2), by dividing the average fiber count per graticule field, F/n_f, minus the mean field blank count per graticule field, B/n_b, by the graticule field area, A_f (approx. 0.00785 mm^2):

$$E = \frac{\left(\frac{F}{n_f} - \frac{B}{n_b}\right)}{A_f}, \text{ fibers/mm}^2.$$

NOTE: Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with >50% of filter area covered with particulate should be reported as "uncountable" or "probably biased." Other fiber counts outside the 100-1300 fiber/mm^2 range should be reported as having "greater than optimal variability" and as being "probably biased."

21. Calculate and report the concentration, C (fibers/cc), of fibers in the air volume sampled, V (L), using the effective collection area of the filter, A_c (approx. 385 mm^2 for a 25-mm filter):

$$C = \frac{(E)(A_c)}{V \cdot 10^3}.$$

NOTE: Periodically check and adjust the value of A_c, if necessary.

22. Report intralaboratory and interlaboratory relative standard deviations (from Step 11) with each set of results.

NOTE: Precision depends on the total number of fibers counted [1,16]. Relative standard deviation is documented in references [1,15-17] for fiber counts up to 100 fibers in 100 graticule fields. Comparability of interlaboratory results is discussed below. As a first approximation, use 213% above and 49% below the count as the upper and lower confidence limits for fiber counts greater than 20 (Fig. 1).

EVALUATION OF METHOD:

A. This method is a revision of P&CAM 239 [10]. A summary of the revisions is as follows:

1. Sampling:
   The change from a 37-mm to a 25-mm filter improves sensitivity for similar air volumes. The change in flow rates allows for 2-m^3 full-shift samples to be taken, providing that the filter is not overloaded with non-fibrous particulates. The collection efficiency of the sampler is not a function of flow rate in the range 0.5 to 16 L/min [10].
2. Sample Preparation Technique:
   The acetone vapor-triacetin preparation technique is a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of P&CAM 239 [2,4,10]. The aluminum "hot block" technique minimizes the amount of acetone needed to prepare each sample.
3. Measurement:
   a. The Walton-Beckett graticule standardizes the area observed [14,18,19].
   b. The HSE/NPL test slide standardizes microscope optics for sensitivity to fiber diameter [4,14].
   c. Because of past inaccuracies associated with low fiber counts, the minimum recommended loading has been increased to 100 fibers/mm^2 filter area (a total of 78.5 fibers counted in 100 fields, each with field area = .00785 mm^2.) Lower levels generally result in an overestimate of the fiber count when compared to results in the recommended analytical range [20]. The recommended loadings should yield intracounter S_r in the range of 0.10 to 0.17 [21,22,23].

B. Interlaboratory comparability:
An international collaborative study involved 16 laboratories using prepared slides from the asbestos cement, milling, mining, textile, and friction material industries [9]. The relative standard deviations (S_r) varied with sample type and laboratory. The ranges were:

	Intralaboratory S_r	Interlaboratory S_r	Overall S_r
AIA (NIOSH A Rules)*	0.12 to 0.40	0.27 to 0.85	0.46
Modified CRS (NIOSH B Rules)**	0.11 to 0.29	0.20 to 0.35	0.25

* Under AIA rules, only fibers having a diameter less than 3 µm are counted and fibers attached to particles larger than 3 µm are not counted. NIOSH A Rules are otherwise similar to the AIA rules.
** See Appendix C.

A NIOSH study conducted using field samples of asbestos gave intralaboratory S_r in the range 0.17 to 0.25 and an interlaboratory S_r of 0.45 [21]. This agrees well with other recent studies [9,14,16].

At this time, there is no independent means for assessing the overall accuracy of this method. One measure of reliability is to estimate how well the count for a single sample agrees with the mean count from a large number of laboratories. The following discussion indicates how this estimation can be carried out based on measurements of the interlaboratory variability, as well as showing how the results of this method relate to the theoretically attainable counting precision and to measured intra- and interlaboratory S_r. (NOTE: The following discussion does not include bias estimates and should not be taken to indicated that lightly loaded samples are as accurate as properly loaded ones).

Theoretically, the process of counting randomly (Poisson) distributed fibers on a filter surface will give an S_r that depends on the number, N, of fibers counted:

$$S_r = 1/(N)^{1/2} \qquad (1)$$

Thus S_r is 0.1 for 100 fibers and 0.32 for 10 fibers counted. The actual S_r found in a number of studies is greater than these theoretical numbers [17,19,20,21].

An additional component of variability comes primarily from subjective interlaboratory differences. In a study of ten counters in a continuing sample exchange program, Ogden [15] found this subjective component of intralaboratory S_r to be approximately 0.2 and estimated the overall S_r by the term:

$$\frac{[N + (0.2 \cdot N)^2]^{1/2}}{N} \qquad (2)$$

Ogden found that the 90% confidence interval of the individual intralaboratory counts in relation to the means were +2 S_r and - 1.5 S_r. In this program, one sample out of ten was a quality control sample. For laboratories not engaged in an intensive quality assurance program, the subjective component of variability can be higher.

In a study of field sample results in 46 laboratories, the Asbestos Information Association also found that the variability had both a constant component and one that depended on the fiber count [14]. These results gave a subjective interlaboratory component of S_r (on the same basis as Ogden's) for field samples of ca. 0.45. A similar value was obtained for 12 laboratories analyzing a set of 24 field samples [21]. This value falls slightly above the range of S_r (0.25 to 0.42 for 1984-85) found for 80 reference laboratories in the NIOSH PAT program for laboratory-generated samples [17].

A number of factors influence S_r for a given laboratory, such as that laboratory's actual counting performance and the type of samples being analyzed. In the absence of other information, such as from an interlaboratory quality assurance program using field samples, the value for the subjective component of variability is chosen as 0.45. It is hoped that the laboratories will carry out the recommended interlaboratory quality assurance programs to improve their performance and thus reduce the S_r.

The above relative standard deviations apply when the population mean has been determined. It is more useful, however, for laboratories to estimate the 90% confidence interval on the mean count from a single sample fiber count (Figure 1). These curves assume similar shapes of the count distribution for interlaboratory and intralaboratory results [16].

For example, if a sample yields a count of 24 fibers, Figure 1 indicates that the mean interlaboratory count will fall within the range of 227% above and 52% below that value 90% of the time. We can apply these percentages directly to the air concentrations as well. If, for instance, this sample (24 fibers counted) represented a 500-L volume, then the measured concentration is 0.02 fibers/mL (assuming 100 fields counted, 25-mm filter, 0.00785 mm^2 counting field area). If this same sample were counted by a group of laboratories, there is a 90% probability that the mean would fall between 0.01 and 0.08 fiber/mL. These limits should be reported in any comparison of results between laboratories.

Note that the S_r of 0.45 used to derive Figure 1 is used as an estimate for a random group of laboratories. If several laboratories belonging to a quality assurance group can show that their interlaboratory S_r is smaller, then it is more correct to use that smaller S_r. However, the estimated S_r of 0.45 is to be used in the absence of such information. Note also that it has been found that S_r can be higher for certain types of samples, such as asbestos cement [9].

Quite often the estimated airborne concentration from an asbestos analysis is used to compare to a regulatory standard. For instance, if one is trying to show compliance with an 0.5 fiber/mL standard using a single sample on which 100 fibers have been counted, then Figure 1 indicates that the 0.5 fiber/mL standard must be 213% higher than the measured air concentration. This indicates that if one measures a fiber concentration of 0.16 fiber/mL (100 fibers counted), then the mean fiber count by a group of laboratories (of which the compliance laboratory might be one) has a 95% chance of being less than 0.5 fibers/mL; i.e., 0.16 + 2.13 x 0.16 = 0.5.

It can be seen from Figure 1 that the Poisson component of the variability is not very important unless the number of fibers counted is small. Therefore, a further approximation is to simply use +213% and −49% as the upper and lower confidence values of the mean for a 100-fiber count.

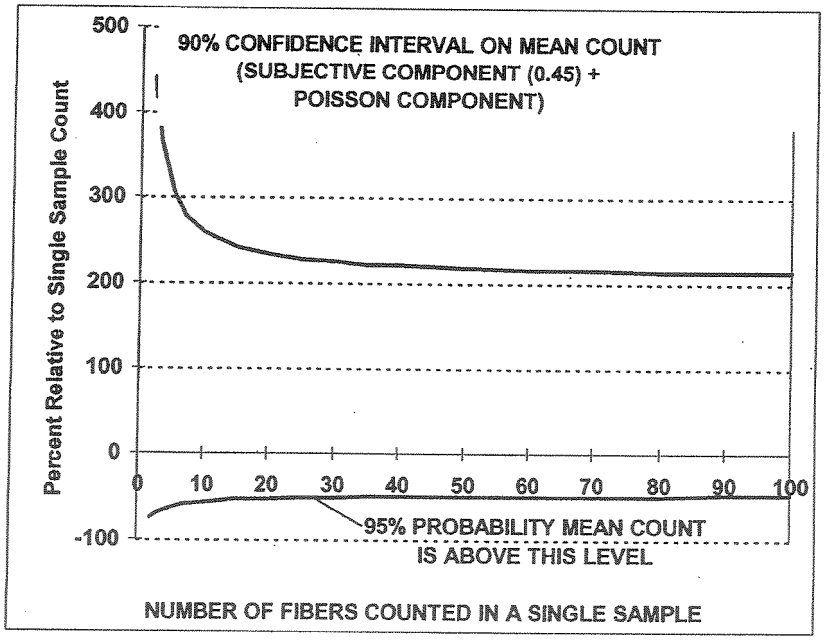

Figure 1. Interlaboratory Precision of Fiber Counts

The curves in Figures 1 are defined by the following equations:

$$UCL = \frac{2X + 2.25 + [(2.25 + 2X)^2 - 4(1 - 2.25 S_r^2) X^2]^{1/2}}{2(1 - 2.25 S_r^2)} \quad (3)$$

$$LCL = \frac{2X + 4 - [(4 + 2X)^2 - 4(1 - 4 S_r^2) X^2]^{1/2}}{2(1 - 4 S_r^2)} \quad (4)$$

where S_r = subjective interlaboratory relative standard deviation, which is close to the total interlaboratory S_r when approximately 100 fibers are counted.
X = total fibers counted on sample
LCL = lower 95% confidence limit.
UCL = upper 95% confidence limit.
Note that the range between these two limits represents 90% of the total range.

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APPENDIX A: CALIBRATION OF THE WALTON-BECKETT GRATICULE:

Before ordering the Walton-Beckett graticule, the following calibration must be done to obtain a counting area (D) 100 µm in diameter at the image plane. The diameter, d_c (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.

1. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.
2. Set the appropriate interpupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.
3. Install the 40 to 45X phase objective.
4. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.
5. Measure the magnified grid length of the graticule, L_o (µm), using the stage micrometer.
6. Remove the graticule from the microscope and measure its actual grid length, L_a (mm). This can best be accomplished by using a stage fitted with verniers.
7. Calculate the circle diameter, d_c (mm), for the Walton-Beckett graticule:

$$d_c = \frac{L_a}{L_o} \times D. \qquad (5)$$

Example: If L_o = 112 µm, L_a = 4.5 mm and D = 100 µm, then d_c = 4.02 mm.

8. Check the field diameter, D (acceptable range 100 µm ± 2 µm) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (acceptable range 0.00754 mm^2 to 0.00817 mm^2).

APPENDIX B: COMPARISON OF COUNTING RULES:

Figure 2 shows a Walton-Beckett graticule as seen through the microscope. The rules will be discussed as they apply to the labeled objects in the figure.

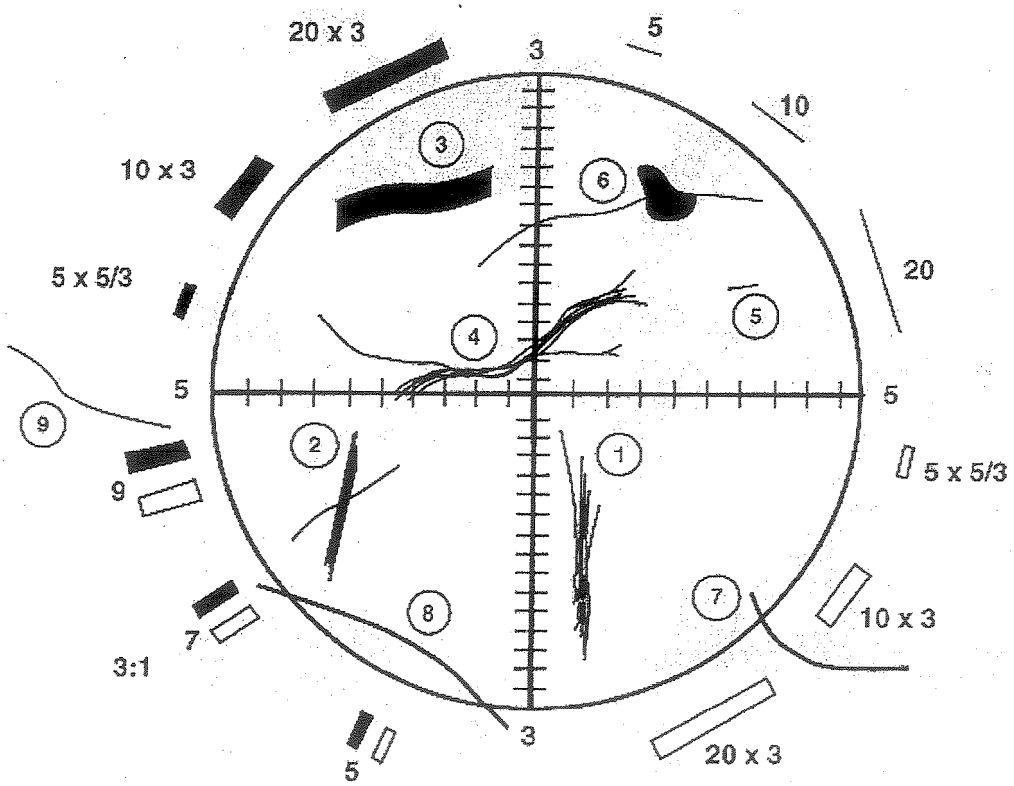

Figure 2. Walton-Beckett graticule with fibers.

These rules are sometimes referred to as the "A" rules.

FIBER COUNT

Object	Count	DISCUSSION
1	1 fiber	Optically observable asbestos fibers are actually bundles of fine fibrils. If the fibrils seem to be from the same bundle the object is counted as a single fiber. Note, however, that all objects meeting length and aspect ratio criteria are counted whether or not they appear to be asbestos.
2	2 fiber	If fibers meeting the length and aspect ratio criteria (length >5 µm and length-to-width ratio >3 to 1) overlap, but do not seem to be part of the same bundle, they are counted as separate fibers.
3	1 fiber	Although the object has a relatively large diameter (>3 µm), it is counted as fiber under the rules. There is no upper limit on the fiber diameter in the counting rules. Note that fiber width is measured at the widest compact section of the object.
4	1 fiber	Although long fine fibrils may extend from the body of a fiber, these fibrils are considered part of the fiber if they seem to have originally been part of the bundle.
5	Do not count	If the object is ≤5 µm long, it is not counted.
6	1 fiber	A fiber partially obscured by a particle is counted as one fiber. If the fiber ends emanating from a particle do not seem to be from the same fiber and each end meets the length and aspect ratio criteria, they are counted as separate fibers.
7	1/2 fiber	A fiber which crosses into the graticule area one time is counted as 1/2 fiber.
8	Do not count	Ignore fibers that cross the graticulate boundary more than once. count
9	Do not count	Ignore fibers that lie outside the graticule boundary.

APPENDIX C. ALTERNATE COUNTING RULES FOR NON-ASBESTOS FIBERS

Other counting rules may be more appropriate for measurement of specific non-asbestos fiber types, such as fibrous glass. These include the "B" rules given below (from NIOSH Method 7400, Revision #2, dated 8/15/87), the World Health Organization reference method for man-made mineral fiber [24], and the NIOSH fibrous glass criteria document method [25]. The upper diameter limit in these methods prevents measurements of non-thoracic fibers. It is important to note that the aspect ratio limits included in these methods vary. NIOSH recommends the use of the 3:1 aspect ratio in counting fibers.

It is emphasized that hybridization of different sets of counting rules is not permitted. Report specifically which set of counting rules are used with the analytical results.

"B" COUNTING RULES:

1. Count only ends of fibers. Each fiber must be longer than 5 µm and less than 3 µm diameter.
2. Count only ends of fibers with a length-to-width ratio equal to or greater than 5:1.
3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets rules 1 and 2 above. Add split ends to the count as appropriate if the split fiber segment also meets the criteria of rules 1 and 2 above.
4. Count visibly free ends which meet rules 1 and 2 above when the fiber appears to be attached to another particle, regardless of the size of the other particle. Count the end of a fiber obscured by another particle if the particle covering the fiber end is less than 3 µm in diameter.
5. Count free ends of fibers emanating from large clumps and bundles up to a maximum of 10 ends (5 fibers), provided that each segment meets rules 1 and 2 above.
6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 graticule fields. Stop at 100 graticule fields, regardless of count.
7. Divide total end count by 2 to yield fiber count.

APPENDIX D. EQUIVALENT LIMITS OF DETECTION AND QUANTITATION

	fiber density on filter*		fiber concentration in air, f/cc	
	fibers per 100 fields	fibers/mm^2	400-L air sample	1000-L air sample
	200	255	0.25	0.10
	100	127	0.125	0.05
LOQ	80	102	0.10	0.04
	50	64	0.0625	0.025
	25	32	0.03	0.0125
	20	25	0.025	0.010
	10	12.7	0.0125	0.005
	8	10.2	0.010	0.004
LOD	5.5	7	0.00675	0.0027

* Assumes 385 mm^2 effective filter collection area, and field area = 0.00785 mm^2, for relatively "clean" (little particulate aside from fibers) filters.

ASBESTOS by TEM

7402

FORMULA: Various MW: Various CAS: Various RTECS: Various

METHOD: 7402 **EVALUATION: PARTIAL** **Issue 1: 15 May 1989**
Issue 2: 15 August 1994

OSHA : 0.1 asbestos fibers (>5 µm long)/cc; 1 f/cc/30 min excursion; carcinogen
MSHA: 2 asbestos fibers/cc
NIOSH: 0.1 f/cc (fibers > 5 µm long)/400 L; carcinogen
ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, fibers/cc; carcinogen

PROPERTIES: solid, fibrous, crystalline, anistropic

SYNONYMS [CAS#]: actinolite [77536-66-4] or ferroactinolite [15669-07-5]; amosite [12172-73-5]; anthophyllite [77536-67-5]; chrysotile [12001-29-5]; serpentine [18786-24-8]; crocidolite [12001-28-4]; tremolite [77536-68-6]; amphibole asbestos [1332-21-4].

SAMPLING

SAMPLER: FILTER (0.45- to 1.2-µm cellulose ester membrane, 25-mm diameter; conductive cassette)

FLOW RATE: 0.5 to 16 L/min

VOL-MIN*: 400 L @ 0.1 fiber/cc
-MAX*: (step 4, sampling)
*Adjust for 100 to 1300 fibers/mm^2

SHIPMENT: routine (pack to reduce shock)

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 80 to 100 fibers counted

BIAS: not determined

OVERALL PRECISION ($\hat{S}_{rT}$): see EVALUATION OF METHOD

ACCURACY: not determined

MEASUREMENT

TECHNIQUE: MICROSCOPY, TRANSMISSION ELECTRON (TEM)

ANALYTE: asbestos fibers

SAMPLE PREPARATION: modified Jaffe wick

EQUIPMENT: transmission electron microscope; energy dispersive X-ray system (EDX) analyzer

CALIBRATION: qualitative electron diffraction; calibration of TEM magnification and EDX system

RANGE: 100 to 1300 fibers/mm^2 filter area [1]

ESTIMATED LOD: 1 confirmed asbestos fiber above 95% of expected mean blank value

PRECISION ($\hat{S}_r$): 0.28 when 65% of fibers are asbestos; 0.20 when adjusted fiber count is applied to PCM count [2].

APPLICABILITY: The quantitative working range is 0.04 to 0.5 fiber/cc for a 1000-L air sample. The LOD depends on sample volume and quantity of interfering dust, and is <0.01 fiber/cc for atmospheres free of interferences. This method is use d to determine asbestos fibers in the optically visible range and is intended to complement the results obtained by phase con trast microscopy (Method 7400).

INTERFERENCES: Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification. Some non-asbestos amphibole m inerals may give electron diffraction patterns similar to asbestos amphiboles.

OTHER METHODS: This method is designed for use with Method 7400 (phase contrast microscopy).

REAGENTS:

1. Acetone. (See SPECIAL PRECAUTIONS.)

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically-conductive extension cowl, cellulose ester membrane filter, 0.45- to 1.2-µm pore size, and backup pad.
   NOTE 1: Analyze representative filters for fiber background before use. Discard the filter lot if mean count is >5 fibers/100 fields. These are defined as laboratory blanks.
   NOTE 2: Use an electrically-conductive extension cowl to reduce electrostatic effects on fiber sampling and during sample shipment. Ground the cowl when possible during sampling.
   NOTE 3: 0.8-µm pore size filters are recommended for personal sampling. 0.45-µm filters are recommended for sampling when performing TEM analysis on the samples because the particles deposit closer to the filter surface. However, the higher pressure drop through these filters normally preclude their use with personal sampling pumps.
2. Personal sampling pump, 0.5 to 16 L/min, with flexible connecting tubing.
3. Microscope, transmission electron, operated at ca. 100 kV, with electron diffraction and energy-dispersive X-ray capabilities, and having a fluorescent screen with inscribed or overlaid calibrated scale (Step 15).
   NOTE: The scale is most efficient if it consists of a series of lines inscribed on the screen or partial circles every 2 cm distant from the center.
4. Diffraction grating replica with known number of lines/mm.
5. Slides, glass, pre-cleaned, 25- x 75-mm.
6. Knife, surgical steel, curved-blade.
7. Tweezers.
8. Grids, 200-mesh TEM copper, (optional: carbon-coated).
9. Petri dishes, 15-mm depth. The top and bottom of the petri dish must fit snugly together. To assure a tight fit, grind the top and bottom pieces together with an abrasive such as carborundum to produce a ground-glass contact surface.
10. Foam, clean polyurethane, spongy, 12-mm thick.
11. Filters, Whatman No. 1 qualitative paper or equivalent, or lens paper.
12. Vacuum evaporator.
13. Cork borer, (about 8-mm).
14. Pen, waterproof, marking.
15. Reinforcement, page, gummed.
16. Asbestos standard bulk materials for reference; e.g. SRM #1866, available from the National Institute of Standards and Technology.
17. Carbon rods, sharpened to 1 mm x 8 mm.
18. Microscope, light, phase contrast (PCM), with Walton-Beckett graticule (see method 7400).
19. Grounding wire, 22-gauge, multi-strand.
20. Tape, shrink- or adhesive-.

SPECIAL PRECAUTIONS: Acetone is extremely flammable (flash point = 0 °F). Take precautions not to ignite it. Heating of acetone must be done in a fume hood using a flameless, spark-free heat source. Asbestos is a confirmed human carcinogen. Handle only in a well-ventilated fume hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. For personal sampling, fasten sampler to worker's lapel near worker's mouth. Remove the top cover from cowl extension ("open-face") and orient sampler face down. Wrap joint between extender and monitor body with tape to help hold the cassette together and provide a marking surface to identify the cassette. Where possible, especially at low %RH, attach sampler to electrical ground to reduce electrostatic effects during sampling.
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Remove top covers from the field blank cassettes and store top covers and cassettes in a clean area (e.g., closed bag or box) during sampling. Replace top covers when sampling is completed.
4. Sample at 0.5 to 16 L/min [3]. Adjust sampling rate, Q (L/min), and time, t (min), to produce fiber density, E, of 100 to 1300 fibers/mm ^{2} [$3.85 \cdot 10^4$ to $5 \cdot 10^5$ fibers per 25-mm filter with effective collection area (A_c= 385 mm^2)] for optimum accuracy. Do not exceed ca. 0.5 mg total dust loading on the filter. These variables are related to the action level (one-half the current standard), L (fibers/cc), of the fibrous aerosol being sampled by:

$$t = \frac{A_c \cdot E}{Q \cdot L \cdot 10^3}, \text{ min.}$$

NOTE: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for 8 h (700 to 2800 L) is appropriate in atmospheres containing ca. 0.1 fiber/cc in the absence of significant amounts of non-asbestos dust. Dusty atmospheres require smaller sample volumes (≤400 L) to obtain countable samples. In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust [3].

5. At the end of sampling, replace top cover and small end caps.
6. Ship samples upright with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.
   NOTE: Do not use untreated polystyrene foam in the shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

7. Remove circular sections from any of three quadrants of each sample and blank filter using a cork borer [4]. The use of three grid preparations reduces the effect of local variations in dust deposit on the filter.
8. Affix the circular filter sections to a clean glass slide with a gummed page reinforcement. Label the slide with a waterproof marking pen.
   NOTE: Up to eight filter sections may be attached to the same slide.
9. Place the slide in a petri dish which contains several paper filters soaked with 2 to 3 mL acetone. Cover the dish. Wait 2 to 4 min for the sample filter(s) to fuse and clear.
   NOTE: The "hot block" clearing technique [5] of Method 7400 or the DMF clearing technique [6] may be used instead of steps 8 and 9.
10. Transfer the slide to a rotating stage inside the bell jar of a vacuum evaporator. Evaporate a 1- by 5-mm section of a graphite rod onto the cleared filter(s). Remove the slide to a clean, dry, covered petri dish [4].
11. Prepare a second petri dish as a Jaffe wick washer with the wicking substrate prepared from filter or lens paper placed on top of a 12-mm thick disk of clean, spongy polyurethane foam [7].

Cut a V-notch on the edge of the foam and filter paper. Use the V-notch as a reservoir for adding solvent.

NOTE: The wicking substrate should be thin enough to fit into the petri dish without touching the lid.

12. Place the TEM grid on the filter or lens paper. Label the grids by marking with a pencil on the filter paper or by putting registration marks on the petri dish halves and marking with a waterproof marker on the dish lid. In a fume hood, fill the dish with acetone until the wicking substrate is saturated.

    NOTE: The level of acetone should be just high enough to saturate the filter paper without creating puddles.

13. Remove about a quarter section of the carbon-coated filter from the glass slide using a surgical knife and tweezers. Carefully place the excised filter, carbon side down, on the appropriately-labeled grid in the acetone-saturated petri dish. When all filter sections have been transferred, slowly add more solvent to the wedge-shaped trough to raise the acetone level as high as possible without disturbing the sample preparations. Cover the petri dish. Elevate one side of the petri dish by placing a slide under it (allowing drops of condensed acetone to form near the edge rather than in the center where they would drip onto the grid preparation).

CALIBRATION AND QUALITY CONTROL:

14. Determine the TEM magnification on the fluorescent screen:
    a. Define a field of view on the fluorescent screen either by markings or physical boundaries.
       NOTE: The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric) [7].
    b. Insert a diffraction grating replica into the specimen holder and place into the microscope. Orient the replica so that the grating lines fall perpendicular to the scale on the TEM fluorescent screen. Ensure that goniometer stage tilt is zero.
    c. Adjust microscope magnification to 10,000X. Measure the distance (mm) between the same relative positions (e.g., between left edges) of two widely-separated lines on the grating replica. Count the number of spaces between the lines.
       NOTE: On most microscopes the magnification is substantially constant only within the central 8- to 10-cm diameter region of the fluorescent screen.
    d. Calculate the true magnification (M) on the fluorescent screen:

$$m = \frac{X \cdot G}{Y}$$

       where: X = total distance (mm) between the two grating lines;
       G = calibration constant of the grating replica (lines/mm);
       Y = number of grating replica spaces counted
    e. After calibration, note the apparent sizes of 0.25 and 5.0 µm on the fluorescent screen. (These dimensions are the boundary limits for counting asbestos fibers by phase contrast microscopy.)

15. Measure 20 grid openings at random on a 200-mesh copper grid by placing a grid on a glass slide and examining it under the PCM. Use the Walton-Beckett graticule to measure the grid opening dimensions. Calculate an average graticule field dimension from the data and use this number to calculate the graticule field area for an average grid opening.

    NOTE: A grid opening is considered as one graticule field.

16. Obtain reference selected area electron diffraction (SAED) or microdiffraction patterns from standard asbestos materials prepared for TEM analysis.

    NOTE: This is a visual reference technique. No quantitative SAED analysis is required [7]. Microdiffraction may produce clearer patterns on very small fibers or fibers partially obscured by other material.

    a. Set the specimen holder at zero tilt.

b. Center a fiber, focus, and center the smallest field-limiting aperture on the fiber. Obtain a diffraction pattern. Photograph each distinctive pattern and keep the photo for comparison to unknowns.

NOTE: Not all fibers will present diffraction patterns. The objective lens current may need adjustment to give optimum pattern visibility. There are many more amphiboles which give diffraction patterns similar to the analytes named on p. 7402-1. Some, but not all, of these can be eliminated by chemical separations. Also, some non-amphiboles (e.g., pyroxenes, some talc fibers) may interfere.

17. Acquire energy-dispersive X-ray (EDX) spectra on approximately 5 fibers having diameters between 0.25 and 0.5 µm of each asbestos variety obtained from standard reference materials [7].

NOTE: The sample may require tilting to obtain adequate signal. Use same tilt angle for all spectra.

a. Prepare TEM grids of all asbestos varieties.
b. Use acquisition times (at least 100 sec) sufficient to show a silicon peak at least 75% of the monitor screen height at a vertical scale of ≥500 counts per channel.
c. Estimate the elemental peak heights visually as follows:
   (1) Normalize all peaks to silicon (assigned an arbitrary value of 10).
   (2) Visually interpret all other peaks present and assign values relative to the silicon peak.
   (3) Determine an elemental profile for the fiber using the elements Na, Mg, Si, Ca, and Fe. Example: 0-4-10-3-<1 [7].

   NOTE: In fibers other than asbestos, determination of Al, K, Ti, S, P, and F may also be required for fiber characterization.

   (4) Determine a typical range of profiles for each asbestos variety and record the profiles for comparison to unknowns.

MEASUREMENT:

18. Perform a diffraction pattern inspection on all sample fibers counted under the TEM, using the procedures given in step 17. Assign the diffraction pattern to one of the following structures:
   a. chrysotile;
   b. amphibole;
   c. ambiguous;
   d. none.

   NOTE: There are some crystalline substances which exhibit diffraction patterns similar to those of asbestos fibers. Many of these, (brucite, halloysite, etc.) can be eliminated from consideration by chemistry. There are, however, several minerals (e.g., pyroxenes, massive amphiboles, and talc fibers) which are chemically similar to asbestos and can be considered interferences. The presence of these substances may warrant the use of more powerful diffraction pattern analysis before positive identification can be made. If interferences are suspected, morphology can play an important role in making positive identification.

19. Obtain EDX spectra in either the TEM or STEM modes from fibers on field samples using the procedure of step 18. Using the diffraction pattern and EDX spectrum, classify the fiber:
   a. For a chrysotile structure, obtain EDX spectra on the first five fibers and one out of ten thereafter. Label the range profiles from 0-5-10-0-0 to 0-10-10-0-0 as "chrysotile."
   b. For an amphibole structure, obtain EDX spectra on the first 10 fibers and one out of ten thereafter. Label profiles ca. 0-2-10-0-7 as "possible amosite"; profiles ca. 1-1-10-0-6 as "possible crocidolite"; profiles ca. 0-4-10-3-<1 as "possible tremolite"; and profiles ca. 0-3-10-0-1 as "possible anthophyllite."

      NOTE: The range of profiles for the amphiboles will vary up to ± 1 unit for each of the elements present according to the relative detector efficiency of the spectrometer.

   c. For an ambiguous structure, obtain EDX spectra on all fibers. Label profiles similar to the chrysotile profile as "possible chrysotile." Label profiles similar to the various amphiboles as "possible amphiboles." Label all others as "unknown" or "non-asbestos."

20. Counting and Sizing:
    a. Insert the sample grid into the specimen grid holder and scan the grid at zero tilt at low magnification (ca. 300 to 500X). Ensure that the carbon film is intact and unbroken over ca. 75% of the grid openings.
    b. In order to determine how the grids should be sampled, estimate the number of fibers per grid opening during a low-magnification scan (500 to 1000X). This will allow the analyst to cover most of the area of the grids during the fiber count and analysis. Use the following rules when picking grid openings to count [7,8]:
        (1) Light loading (<5 fibers per grid opening): count total of 40 grid openings.
        (2) Moderate loading (5 to 25 fibers per grid opening): count minimum of 40 grid openings or 100 fibers.
        (3) Heavy loading (>25 fibers per opening): count a minimum of 100 fibers and at least 6 grid openings.

        Note that these grid openings should be selected approximately equally among the three grid preparations and as randomly as possible from each grid.
    c. Count only grid openings that have the carbon film intact. At 500 to 1000X magnification, begin counting at one end of the grid and systematically traverse the grid by rows, reversing direction at row ends. Select the number of fields per traverse based on the loading indicated in the initial scan. Count at least 2 field blanks per sample set to document possible contamination of the samples. Count fibers using the following rules:
        (1) Count all particles with diameter greater than 0.25 µm that meet the definition of a fiber (aspect ratio ≥3:1, longer than 5 µm). Use the guideline of counting all fibers that would have been counted under phase contrast light microscopy (Method 7400). Use higher magnification (10000X) to determine fiber dimensions and countability under the acceptance criteria. Analyze a minimum of 10% of the fibers, and at least 3 asbestos fibers, by EDX and SAED to confirm the presence of asbestos. Fibers of similar morphology under high magnification can be identified as asbestos without SAED. Particles which are of questionable morphology should be analyzed by SAED and EDX to aid in identification.
        (2) Count fibers which are partially obscured by the grid as half fibers.
            NOTE: If a fiber is partially obscured by the grid bar at the edge of the field of view, count it as a half fiber only if more than 2.5 µm of fiber is visible.
        (3) Size each fiber as it is counted and record the diameter and length:
            (a) Move the fiber to the center of the screen. Read the length of the fiber directly from the scale on the screen.
                NOTE 1: Data can be recorded directly off the screen in µm and later converted to µm by computer.
                NOTE 2: For fibers which extend beyond the field of view, the fiber must be moved and superimposed upon the scale until its entire length has been measured.
            (b) When a fiber has been sized, return to the lower magnification and continue the traverse of the grid area to the next fiber.
    d. Record the following fiber counts:
        (1) f_s, f_b = number of asbestos fibers in the grid openings analyzed on the sample filter and corresponding field blank, respectively.
        (2) F_s, F_b = number of fibers, regardless of identification, in the grid openings analyzed on the sample filter and corresponding field blank, respectively.

CALCULATIONS:

21. Calculate and report the fraction of optically visible asbestos fibers on the filter, $(f_s - f_b)/(F_s - F_b)$. Apply this fraction to fiber counts obtained by PCM on the same filter or on other filters for which the TEM sample is representative. The final result is an asbestos fiber count. The type of asbestos present should also be reported.
22. As an integral part of the report, give the model and manufacturer of the TEM as well as the model and manufacturer of the EDX system.

EVALUATION OF METHOD:

The TEM method, using the direct count of asbestos fibers, has been shown to have a precision of 0.275 (s_r) in an evaluation of mixed amosite and wollastonite fibers. The estimate of the asbestos fraction, however, had a precision of 0.11 (s_r). When this fraction was applied to the PCM count, the overall precision of the combined analysis was 0.20 [2].

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