

## **Appendix C**

### **Description of Sampling and Monitoring Techniques and Procedures**



Airborne Asbestos Analysis  
By  
Transmission Electron Microscopy

Code of Federal Regulations (CFR) Title 40, Part 763 Appendix A  
Asbestos Hazardous Emissions Reduction Act (AHERA)



an accredited inspector under paragraphs (a) (3), (4), (5) of this section, or an architect, project engineer or accredited inspector under paragraph (a)(7) of this section, the local education agency shall have 180 days following the date of identification of ACBM to comply with this subpart E.

APPENDIX A TO SUBPART E—INTERIM TRANSMISSION ELECTRON MICROSCOPY ANALYTICAL METHODS—MANDATORY AND NONMANDATORY—AND MANDATORY SECTION TO DETERMINE COMPLETION OF RESPONSE ACTIONS

I. Introduction

The following appendix contains three units. The first unit is the mandatory transmission electron microscopy (TEM) method which all laboratories must follow; it is the minimum requirement for analysis of air samples for asbestos by TEM. The mandatory method contains the essential elements of the TEM method. The second unit contains the complete non-mandatory method. The non-mandatory method supplements the mandatory method by including additional steps to improve the analysis. EPA recommends that the non-mandatory method be employed for analyzing air filters; however, the laboratory may choose to employ the mandatory method. The non-mandatory method contains the same minimum requirements as are outlined in the mandatory method. Hence, laboratories may choose either of the two methods for analyzing air samples by TEM.

The final unit of this Appendix A to subpart E defines the steps which must be taken to determine completion of response actions. This unit is mandatory.

II. Mandatory Transmission Electron Microscopy Method

A. Definitions of Terms

1. *Analytical sensitivity*—Airborne asbestos concentration represented by each fiber counted under the electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005 structures/cm<sup>3</sup>.

2. *Asbestiform*—A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

3. *Aspect ratio*—A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.

4. *Bundle*—A structure composed of three or more fibers in a parallel arrangement

with each fiber closer than one fiber diameter.

5. *Clean area*—A controlled environment which is maintained and monitored to assure a low probability of asbestos contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm<sup>2</sup> in an area of 0.057 mm<sup>2</sup> (nominally 10 200-mesh grid openings) and a maximum of 53 structures/mm<sup>2</sup> for any single preparation for that same area.

6. *Cluster*—A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

7. *ED*—Electron diffraction.

8. *EDXA*—Energy dispersive X-ray analysis.

9. *Fiber*—A structure greater than or equal to 0.5 μm in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.

10. *Grid*—An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.

11. *Intersection*—Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.

12. *Laboratory sample coordinator*—That person responsible for the conduct of sample handling and the certification of the testing procedures.

13. *Filter background level*—The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on a blank (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm<sup>2</sup>.

14. *Matrix*—Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

15. *NSD*—No structure detected.

16. *Operator*—A person responsible for the TEM instrumental analysis of the sample.

17. *PCM*—Phase contrast microscopy.

18. *SAED*—Selected area electron diffraction.

19. *SEM*—Scanning electron microscope.

20. *STEM*—Scanning transmission electron microscope.

21. *Structure*—a microscopic bundle, cluster, fiber, or matrix which may contain asbestos.

22. *Scm<sup>3</sup>*—Structures per cubic centimeter.

23. *Smm<sup>2</sup>*—Structures per square millimeter.

24. *TEM*—Transmission electron microscope.

B. Sampling

1. The sampling agency must have written quality control procedures and documents which verify compliance.

2. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (References 1, 2, 3, and 5 of Unit II.J.).

3. Sampling for airborne asbestos following an abatement action must use commercially available cassettes.

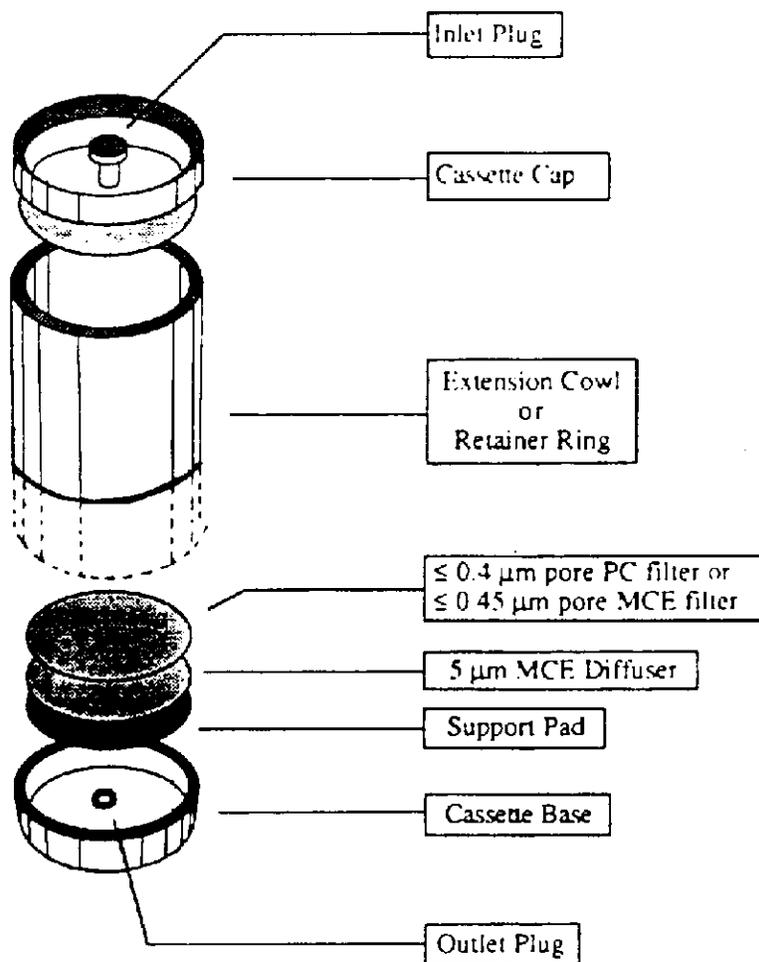
4. Prescreen the loaded cassette collection filters to assure that they do not contain

concentrations of asbestos which may interfere with the analysis of the sample. A filter blank average of less than 18 s/mm<sup>2</sup> in an area of 0.057 mm<sup>2</sup> (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm<sup>2</sup> for that same area is acceptable for this method.

5. Use sample collection filters which are either polycarbonate having a pore size less than or equal to 0.4 μm or mixed cellulose ester having a pore size less than or equal to 0.45 μm.

6. Place these filters in series with a 5.0 μm backup filter (to serve as a diffuser) and a support pad. See the following Figure 1:

FIGURE I--SAMPLING CASSETTE CONFIGURATION



7. Reloading of used cassettes is not permitted.

8. Orient the cassette downward at approximately 45 degrees from the horizontal.

9. Maintain a log of all pertinent sampling information.

10. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter (not the filter which will be used in sampling) before and after the sampling operation.

11. Record all calibration information.

12. Ensure that the mechanical vibrations from the pump will be minimized to prevent transferral of vibration to the cassette.

13. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by damping out any pump action fluctuations if necessary.

14. The final plastic barrier around the abatement area remains in place for the sampling period.

15. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust. (See suggested protocol in Unit III.B.7.d.)

16. Select an appropriate flow rate equal to or greater than 1 liter per minute (L/min) or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.

17. A minimum of 13 samples are to be collected for each testing site consisting of the following:

a. A minimum of five samples per abatement area.

b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.

c. Two field blanks are to be taken by removing the cap for not more than 30 seconds and replacing it at the time of sampling before sampling is initiated at the following places:

i. Near the entrance to each abatement area.

ii. At one of the ambient sites. (DO NOT leave the field blanks open during the sampling period.)

d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.

18. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.

19. The following Table I specifies volume ranges to be used:

TABLE 1--NUMBER OF 200 MESH EM GRID OPENINGS (0.0037 MM<sup>2</sup>) THAT NEED TO BE ANALYZED TO MAINTAIN SENSITIVITY OF 0.005 STRUCTURES/CC BASED ON VOLUME AND EFFECTIVE FILTER AREA

Effective Filter Area 385 sq mm		Effective Filter Area 855 sq mm	
Volume (liters)	# of grid openings	Volume (liters)	# of grid openings
560	24	1,250	24
600	23	1,300	23
700	19	1,400	21
800	17	1,600	19
900	15	1,800	17
1,000	14	2,000	15
1,100	12	2,200	14
1,200	11	2,400	13
1,300	10	2,500	12
1,400	10	2,800	11
1,500	9	3,000	10
1,600	8	3,200	9
1,700	8	3,400	9
1,800	8	3,600	8
1,900	7	3,800	8
2,000	7	4,000	8
2,100	6	4,200	7
2,200	6	4,400	7
2,300	6	4,600	7
2,400	6	4,800	6
2,500	5	5,000	6
2,600	5	5,200	6
2,700	5	5,400	6
2,800	5	5,600	5
2,900	5	5,800	5
3,000	5	6,000	5
3,100	4	6,200	5
3,200	4	6,400	5
3,300	4	6,600	5
3,400	4	6,800	4
3,500	4	7,000	4
3,600	4	7,200	4
3,700	4	7,400	4
3,800	4	7,600	4

Note minimum volumes required:  
 25 mm : 560 liters  
 37 mm : 1250 liters

Filter diameter of 25 mm = effective area of 385 sq mm  
 Filter diameter of 37 mm = effective area of 855 sq mm

20. Ensure that the sampler is turned up-right before interrupting the pump flow.

21. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory.

22. Ensure that the samples are stored in a secure and representative location.

23. Do not change containers if portions of these filters are taken for other purposes.

24. A summary of Sample Data Quality Objectives is shown in the following Table II:

TABLE II--SUMMARY OF SAMPLING AGENCY DATA QUALITY OBJECTIVES

This table summarizes the data quality objectives from the performance of this method in terms of precision, accuracy, completeness, representativeness, and comparability. These objectives are assured by the periodic control checks and reference checks listed here and described in the text of the method.

Unit Operation	QC Check	Frequency	Conformance Expectations
Sampling materials	Sealed blank	1 per I/O site	95%
Sample procedures	Field blanks	2 per I/O site	95%
	Pump calibration	Before and after each field series	90%
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample shipment	Review of sending report	Each sample	95% complete

#### C. Sample Shipment

Ship bulk samples to the analytical laboratory in a separate container from air samples.

#### D. Sample Receiving

1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.

2. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected.

#### E. Sample Preparation

1. All sample preparation and analysis shall be performed by a laboratory independent of the abatement contractor.

2. Wet-wipe the exterior of the cassettes to minimize contamination possibilities before taking them into the clean room facility.

3. Perform sample preparation in a well-equipped clean facility.

NOTE: The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA-filtered. The cumulative analytical blank concentration must average less than 18  $\mu\text{m}^3$  in an area of 0.057  $\text{m}^2$  (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53  $\mu\text{m}^3$  for that same area.

4. Preparation areas for air samples must not only be separated from preparation areas for bulk samples, but they must be prepared in separate rooms.

5. Direct preparation techniques are required. The object is to produce an intact film containing the particulates of the filter surface which is sufficiently clear for TEM analysis.

a. TEM Grid Opening Area measurement must be done as follows:

i. The filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique.

ii. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass and examining it under the PCM. Use a calibrated graticule to measure the average field diameters. From the data, calculate the field area for an average grid opening.

iii. Measurements can also be made on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.

b. TEM specimen preparation from polycarbonate (PC) filters. Procedures as described in Unit III.G or other equivalent methods may be used.

c. TEM specimen preparation from mixed cellulose ester (MCE) filters.

i. Filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique or the Burdette procedure (Ref. 7 of Unit II.J.)

ii. Plasma etching of the collapsed filter is required. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the conditions that should be used. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a

known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for the particulate ash and operating conditions will then be set such that a 1-2  $\mu\text{m}$  (10 percent) layer of collapsed surface will be removed.

iii. Procedures as described in Unit III, or other equivalent methods may be used to prepare samples.

#### F. TEM Method

1. An 80-120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated gradations is required. If the TEM is equipped with EDXA it must either have a STEM attachment or be capable of producing a spot less than 250 nm in diameter at crossover. The microscope shall be calibrated routinely for magnification and camera constant.

2. *Determination of Camera Constant and ED Pattern Analysis.* The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved by using a carbon-coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulate. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one-half the diameter of the rings times the interplanar spacing of the ring being measured.

3. *Magnification Calibration.* The magnification calibration must be done at the fluorescent screen. The TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica (e.g., one containing 2,160 lines/mm). Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should

be metric). A logbook must be maintained, and the dates of calibration and the values obtained must be recorded. The frequency of calibration depends on the past history of the particular microscope. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate a eucentric tilting specimen stage, all specimens and the cross grating replica must be placed at the eucentric position.

4. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory.

5. Microscope settings: 80-120 kV, grid assessment 250-1,000X, then 15,000-20,000X screen magnification for analysis.

6. Approximately one-half (0.5) of the predetermined sample area to be analyzed shall be performed on one sample grid preparation and the remaining half on a second sample grid preparation.

7. Individual grid openings with greater than 5 percent openings (holes) or covered with greater than 25 percent particulate matter or obviously having nonuniform loading must not be analyzed.

8. Reject the grid if:

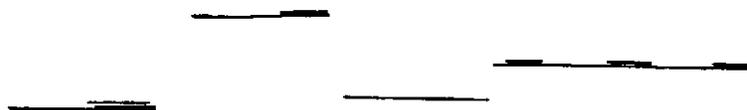
- Less than 50 percent of the grid openings covered by the replica are intact.
- The replica is doubled or folded.
- The replica is too dark because of incomplete dissolution of the filter.

9. *Recording Rules.*

a. Any continuous grouping of particles in which an asbestos fiber with an aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5  $\mu\text{m}$  is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, or 2 definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. An intersection is a non-parallel touching or crossing of fibers, with the projection having an aspect ratio of 3:1 or greater. See the following Figure 2:

FIGURE 2--COUNTING GUIDELINES USED IN DETERMINING ASBESTOS STRUCTURES

Count as 1 fiber; 1 Structure; no intersections.



Count as 2 fibers if space between fibers is greater than width of 1 fiber diameter or number of intersections is equal to or less than 1.



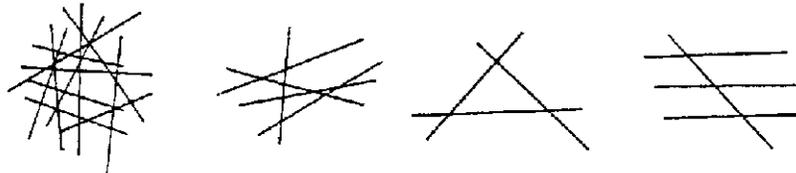
Count as 3 structures if space between fibers is greater than width of 1 fiber diameter or if the number of intersections is equal to or less than 2.



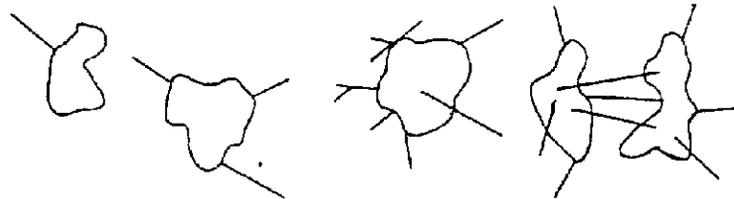
Count bundles as 1 structure; 3 or more parallel fibrils less than 1 fiber diameter separation.



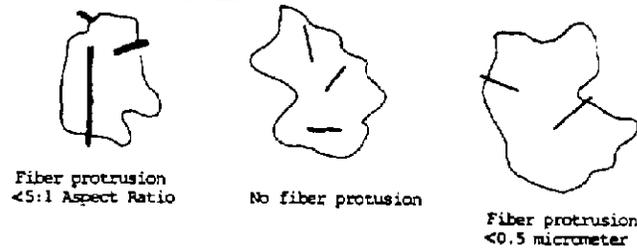
Count clusters as 1 structure; fibers having greater than or equal to 3 intersections.



Count matrix as 1 structure.



DO NOT COUNT AS STRUCTURES:



— <0.5 micrometer in length  
 — <5:1 Aspect Ratio

- i. *Fiber*. A structure having a minimum length greater than or equal to 0.5  $\mu\text{m}$  and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.
- ii. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
- iii. *Cluster*. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated

- from the group. Groupings must have more than two intersections.
- iv. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
- b. Separate categories will be maintained for fibers less than 5  $\mu\text{m}$  and for fibers equal to or greater than 5  $\mu\text{m}$  in length.
- c. Record NSD when no structures are detected in the field.
- d. Visual identification of electron diffraction (ED) patterns is required for each asbestos structure counted which would cause the

analysis to exceed the 70  $s/mm^2$  concentration. (Generally this means the first four fibers identified as asbestos must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)

e. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. In the event that examination of the pattern by a qualified individual indicates that the pattern has been misidentified visually, the client shall be contacted.

f. Energy Dispersive X-ray Analysis (EDXA) is required of all amphiboles which would cause the analysis results to exceed the 70  $s/mm^2$  concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)

g. If the number of fibers in the non-asbestos class would cause the analysis to exceed the 70  $s/mm^2$  concentration, the fact that they are not asbestos must be confirmed by EDXA or measurement of a zone axis diffraction pattern.

h. Fibers classified as chrysotile must be identified by diffraction or X-ray analysis and recorded on a count sheet. X-ray analysis alone can be used only after 70  $s/mm^2$  have been exceeded for a particular sample.

i. Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. (X-ray analysis alone can be used only after 70  $s/mm^2$  have been exceeded for a particular sample.)

j. If a diffraction pattern was recorded on film, record the micrograph number on the count sheet.

k. If an electron diffraction was attempted but no pattern was observed, record N on the count sheet.

l. If an EDXA spectrum was attempted but not observed, record N on the count sheet.

m. If an X-ray analysis spectrum is stored, record the file and disk number on the count sheet.

#### 10. Classification Rules.

a. *Fiber*. A structure having a minimum length greater than or equal to 0.5  $\mu m$  and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

b. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

c. *Cluster*. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

d. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

11. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid holder. Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request.

#### G. Sample Analytical Sequence

1. Under the present sampling requirements a minimum of 13 samples is to be collected for the clearance testing of an abatement site. These include five abatement area samples, five ambient samples, two field blanks, and one sealed blank.

2. Carry out visual inspection of work site prior to air monitoring.

3. Collect a minimum of 5 air samples inside the work site and 5 samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.

4. Remaining steps in the analytical sequence are contained in Unit IV of this Appendix.

#### H. Reporting

1. The following information must be reported to the client for each sample analyzed:

a. Concentration in structures per square millimeter and structures per cubic centimeter.

b. Analytical sensitivity used for the analysis.

c. Number of asbestos structures.

d. Area analyzed.

e. Volume of air sampled (which must be initially supplied to lab by client).

f. Copy of the count sheet must be included with the report.

g. Signature of laboratory official to indicate that the laboratory met specifications of the method.

h. Report form must contain official laboratory identification (e.g., letterhead).

i. Type of asbestos.

#### I. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards are to be performed along with the sample analysis as indicators that the materials used are adequate and the

operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might

develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:

TABLE III--SUMMARY OF LABORATORY DATA QUALITY OBJECTIVES

Unit Operation	QC Check	Frequency	Conformance Expectation
Sample receiving	Review of receiving report	Each sample	95% complete
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample preparation	Supplies and reagents	On receipt	Meet specs. or reject
	Grid opening size	20 openings/20 grids/lot of 1000 or 1 opening/sample	100%
	Special clean area monitoring	After cleaning or service	Meet specs. or re-clean
	Laboratory blank	1 per prep series or 10%	Meet specs. or reanalyze series
	Plasma etch blank	1 per 20 samples	75%
	Multiple preps (3 per sample)	Each sample	One with cover of 15 complete grid sqs.
	Sample analysis	System check	Each day
Alignment check		Each day	Each day
Magnification calibration with low and high standards		Each month or after service	95%
ED calibration by gold standard		Weekly	95%
EDS calibration by copper line		Daily	95%
Performance check	Laboratory blank (measure of cleanliness)	Prep 1 per series or 10% read 1 per 25 samples	Meet specs. or reanalyze series
	Replicate counting (measure of precision)	1 per 100 samples	1.5 x Poisson Std. Dev.
	Duplicate analysis (measure of reproducibility)	1 per 100 samples	2 x Poisson Std. Dev.
	Known samples of typical materials (working standards)	Training and for comparison with unknowns	100%
	Analysis of NBS SRM 1876 and/or RM 8410 (measure of accuracy and comparability)	1 per analyst per year	1.5 x Poisson Std. Dev.
	Data entry review (data validation and measure of completeness)	Each sample	95%
	Record and verify ID electron diffraction pattern of structure	1 per 5 samples	80% accuracy
Calculations and data reduction	Hand calculation of automated data reduction procedure or independent recalculation of hand-calculated data	1 per 100 samples	85%

1. When the samples arrive at the laboratory, check the samples and documentation for completeness and requirements before initiating the analysis.

2. Check all laboratory reagents and supplies for acceptable asbestos background levels

3. Conduct all sample preparation in a clean room environment monitored by laboratory blanks. Testing with blanks must also be done after cleaning or servicing the room.

4. Prepare multiple grids of each sample.

5. Provide laboratory blanks with each sample batch. Maintain a cumulative average of these results. If there are more than 53 fibers/mm<sup>2</sup> per 10 200-mesh grid openings, the system must be checked for possible sources of contamination.

6. Perform a system check on the transmission electron microscope daily.

7. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III under Unit II.1.

8. Ensure qualified operator performance by evaluation of replicate analysis and standard sample comparisons as set forth in Table III under Unit II.1.

9. Validate all data entries.

10. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III under Unit II.1.

11. Record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual identification based on his measured patterns.

12. Appropriate logs or records must be maintained by the analytical laboratory verifying that it is in compliance with the mandatory quality assurance procedures.

#### J. References

For additional background information on this method, the following references should be consulted.

1. "Guidance for Controlling Asbestos-Containing Materials in Buildings." EPA 560/5-85-021, June 1985.

2. "Measuring Airborne Asbestos Following an Abatement Action." USEPA, Office of Pollution Prevention and Toxics, EPA 600/4-85-049, 1985.

3. Small, John and E. Steel. Asbestos Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.

4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751. U.S. Bureau of Mines, 1977.

5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods. EPA 600/4-77-027a. USEPA, Office of Research and Development, 1977.

6. Method 2A: Direct Measurement of Gas Volume through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.

7. Burdette, G.J., Health & Safety Exec. Research & Lab. Services Div., London,

"Proposed Analytical Method for Determination of Asbestos in Air."

8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc., "Standard Operating Procedure for Determination of Airborne Asbestos Fibers by Transmission Electron Microscopy Using Polycarbonate Membrane Filters." WERL SOP 87-1, March 5, 1987.

9. NIOSH Method 7402 for Asbestos Fibers, 12-11-86 Draft.

10. Yamate, G., Agarwall, S.C., Gibbons, R.D., IIT Research Institute, "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy." Draft report. USEPA Contract 68-02-3266, July 1984.

11. "Guidance to the Preparation of Quality Assurance Project Plans." USEPA, Office of Pollution Prevention and Toxics, 1984.

#### III. Nonmandatory Transmission Electron Microscopy Method

##### A. Definitions of Terms

1. *Analytical sensitivity*—Airborne asbestos concentration represented by each fiber counted under the electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005  $\mu\text{cm}^3$ .

2. *Asbestiform*—A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

3. *Aspect ratio*—A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.

4. *Bundle*—A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

5. *Clean area*—A controlled environment which is maintained and monitored to assure a low probability of asbestos contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm<sup>2</sup> in an area of 0.057 mm<sup>2</sup> (nominally 10 200 mesh grid openings) and a maximum of 53 structures/mm<sup>2</sup> for no more than one single preparation for that same area.

6. *Cluster*—A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

7. *ED*—Electron diffraction.

8. *EDXA*—Energy dispersive X ray analysis.

9. *Fiber*—A structure greater than or equal to 0.5  $\mu\text{m}$  in length with an aspect ratio (length to width) of 3:1 or greater and having substantially parallel sides.

10. *Grid*—An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.

11. *Intersection*—Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.

12. *Laboratory sample coordinator*—That person responsible for the conduct of sample handling and the certification of the testing procedures.

13. *Filter background level*—The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on blanks (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm<sup>2</sup>.

14. *Matrix*—Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

15. *NSD*—No structure detected.

16. *Operator*—A person responsible for the TEM instrumental analysis of the sample.

17. *PCM*—Phase contrast microscopy.

18. *SAED*—Selected area electron diffraction.

19. *SEM*—Scanning electron microscope.

20. *STEM*—Scanning transmission electron microscope.

21. *Structure*—a microscopic bundle, cluster, fiber or matrix which may contain asbestos.

22. *Scm<sup>3</sup>*—Structures per cubic centimeter.

23. *Smm<sup>2</sup>*—Structures per square millimeter.

24. *TEM*—Transmission electron microscope.

#### B. Sampling

1. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (See References 1, 2, and 5 of Unit III.L.) Special precautions should be taken to avoid contamination of the sample. For example, materials that have not been prescreened for their asbestos background content should not be used; also, sample handling procedures which

do not take cross contamination possibilities into account should not be used.

2. Material and supply checks for asbestos contamination should be made on all critical supplies, reagents, and procedures before their use in a monitoring study.

3. Quality control and quality assurance steps are needed to identify problem areas and isolate the cause of the contamination (see Reference 5 of Unit III.L.). Control checks shall be permanently recorded to document the quality of the information produced. The sampling firm must have written quality control procedures and documents which verify compliance. Independent audits by a qualified consultant or firm should be performed once a year. All documentation of compliance should be retained indefinitely to provide a guarantee of quality. A summary of Sample Data Quality Objectives is shown in Table II of Unit II.B.

4. Sampling materials.

a. Sample for airborne asbestos following an abatement action using commercially available cassettes.

b. Use either a cowl or a filter-retaining middle piece. Conductive material may reduce the potential for particulates to adhere to the walls of the cowl.

c. Cassettes must be verified as "clean" prior to use in the field. If packaged filters are used for loading or preloaded cassettes are purchased from the manufacturer or a distributor, the manufacturer's name and lot number should be entered on all field data sheets provided to the laboratory, and are required to be listed on all reports from the laboratory.

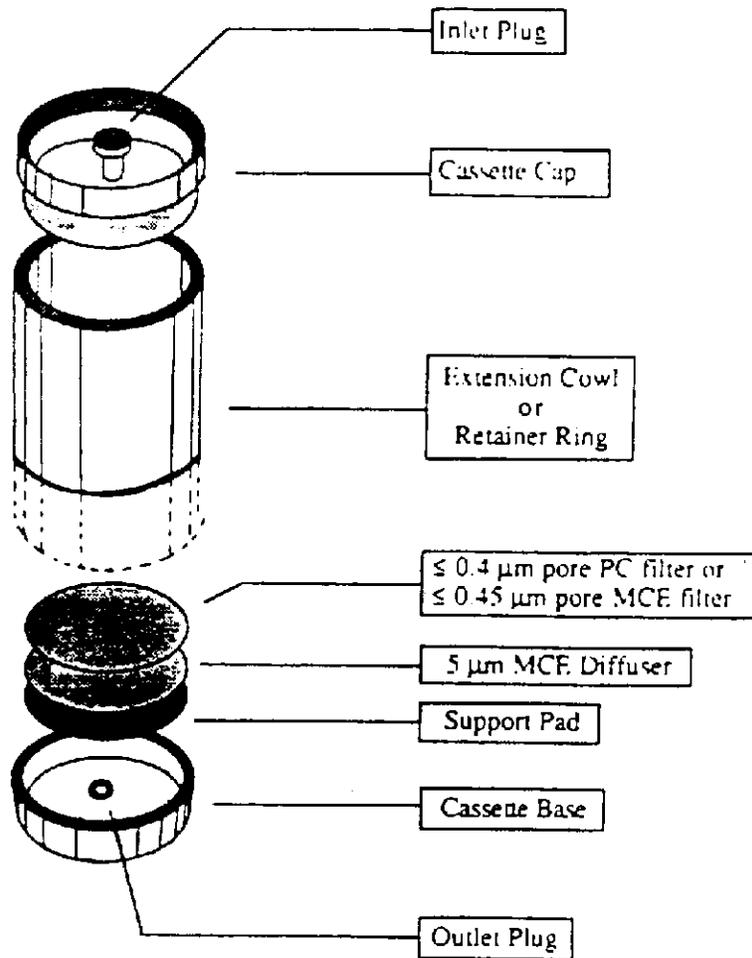
d. Assemble the cassettes in a clean facility (See definition of clean area under Unit III.A.).

e. Reloading of used cassettes is not permitted.

f. Use sample collection filters which are either polycarbonate having a pore size of less than or equal to 0.4  $\mu\text{m}$  or mixed cellulose ester having a pore size of less than or equal to 0.45  $\mu\text{m}$ .

g. Place these filters in series with a backup filter with a pore size of 5.0  $\mu\text{m}$  (to serve as a diffuser) and a support pad. See the following Figure 1:

FIGURE I--SAMPLING CASSETTE CONFIGURATION



h. When polycarbonate filters are used, position the highly reflective face such that the incoming particulate is received on this surface

i. Seal the cassettes to prevent leakage around the filter edges or between cassette part joints. A mechanical press may be useful to achieve a reproducible leak-free seal.

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Shrink fit gel-bands may be used for this purpose and are available from filter manufacturers and their authorized distributors.

j. Use wrinkle-free loaded cassettes in the sampling operation.

5. Pump setup.

a. Calibrate the sampling pump over the range of flow rates and loads anticipated for the monitoring period with this flow measuring device in series. Perform this calibration using guidance from EPA Method 2A each time the unit is sent to the field (See Reference 6 of Unit III.L.).

b. Configure the sampling system to preclude pump vibrations from being transmitted to the cassette by using a sampling stand separate from the pump station and making connections with flexible tubing.

c. Maintain continuous smooth flow conditions by damping out any pump action fluctuations if necessary.

d. Check the sampling system for leaks with the end cap still in place and the pump operating before initiating sample collection. Trace and stop the source of any flow indicated by the flowmeter under these conditions.

e. Select an appropriate flow rate equal to or greater than 1 L/min or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.

f. Orient the cassette downward at approximately 45 degrees from the horizontal.

g. Maintain a log of all pertinent sampling information, such as pump identification number, calibration data, sample location, date, sample identification number, flow rates at the beginning, middle, and end, start and stop times, and other useful information or comments. Use of a sampling log form is recommended. See the following Figure 2:



TABLE 1--NUMBER OF 200 MESH EM GRID OPENINGS (0.0057 MM<sup>2</sup>) THAT NEED TO BE ANALYZED TO MAINTAIN SENSITIVITY OF 0.005 STRUCTURES/CC BASED ON VOLUME AND EFFECTIVE FILTER AREA

	Effective Filter Area 385 sq mm		Effective Filter Area 855 sq mm		
	Volume (liters)	# of grid openings	Volume (liters)	# of grid openings	
Recommended Volume Range	560	24	1,250	24	Recommended Volume Range
	600	23	1,300	23	
	700	19	1,400	21	
	800	17	1,500	19	
	900	15	1,600	17	
	1,000	14	2,000	15	
	1,100	12	2,200	14	
	1,200	11	2,400	13	
	1,300	10	2,600	12	
	1,400	10	2,800	11	
	1,500	9	3,000	10	
	1,600	8	3,200	9	
	1,700	8	3,400	9	
	1,800	8	3,600	8	
	1,900	7	3,800	8	
	2,000	7	4,000	8	
	2,100	6	4,200	7	
	2,200	6	4,400	7	
	2,300	6	4,600	7	
	2,400	6	4,800	6	
2,500	5	5,000	6		
2,600	5	5,200	6		
2,700	5	5,400	6		
2,800	5	5,600	5		
2,900	5	5,800	5		
3,000	5	6,000	5		
3,100	4	6,200	5		
3,200	4	6,400	5		
3,300	4	6,600	5		
3,400	4	6,800	4		
3,500	4	7,000	4		
3,600	4	7,200	4		
3,700	4	7,400	4		
3,800	4	7,600	4		

Note minimum volumes required:  
25 mm : 560 liters  
37 mm : 1250 liters

Filter diameter of 25 mm = effective area of 385 sq mm  
Filter diameter of 37 mm = effective area of 855 sq mm

k. At the conclusion of sampling, turn the cassette upward before stopping the flow to minimize possible particle loss. If the sampling is resumed, restart the flow before re-orienting the cassette downward. Note the condition of the filter at the conclusion of sampling.

l. Double check to see that all information has been recorded on the data collection forms and that the cassette is securely

closed and appropriately identified using a waterproof label. Protect cassettes in individual clean resealed polyethylene bags. Bags are to be used for storing cassette caps when they are removed for sampling purposes. Caps and plugs should only be removed or replaced using clean hands or clean disposable plastic gloves.

m. Do not change containers if portions of these filters are taken for other purposes

6. Minimum sample number per site. A minimum of 13 samples are to be collected for each testing consisting of the following:

a. A minimum of five samples per abatement area.

b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.

c. Two field blanks are to be taken by removing the cap for not more than 30 sec and replacing it at the time of sampling before sampling is initiated at the following places:

i. Near the entrance to each ambient area.

ii. At one of the ambient sites.

(NOTE: Do not leave the blank open during the sampling period.)

d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.

7. Abatement area sampling.

a. Conduct final clearance sampling only after the primary containment barriers have been removed; the abatement area has been thoroughly dried; and, it has passed visual inspection tests by qualified personnel. (See Reference I of Unit III.L.)

b. Containment barriers over windows, doors, and air passageways must remain in place until the TEM clearance sampling and analysis is completed and results meet clearance test criteria. The final plastic barrier remains in place for the sampling period.

c. Select sampling sites in the abatement area on a random basis to provide unbiased and representative samples.

d. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust.

i. Equipment used in aggressive sampling such as a leaf blower and/or fan should be properly cleaned and decontaminated before use.

ii. Air filtration units shall remain on during the air monitoring period.

iii. Prior to air monitoring, floors, ceiling and walls shall be swept with the exhaust of a minimum one (1) horsepower leaf blower.

iv. Stationary fans are placed in locations which will not interfere with air monitoring equipment. Fan air is directed toward the ceiling. One fan shall be used for each 10,000 ft<sup>3</sup> of worksite.

v. Monitoring of an abatement work area with high-volume pumps and the use of circulating fans will require electrical power. Electrical outlets in the abatement area may be used if available. If no such outlets are available, the equipment must be supplied with electricity by the use of extension cords and strip plug units. All electrical power supply equipment of this type must be approved Underwriter Laboratory equipment that has not been modified. All wiring must be grounded. Ground fault interrupters should be used. Extreme care must be taken to clean up any residual water and ensure

that electrical equipment does not become wet while operational.

vi. Low volume pumps may be carefully wrapped in 6-mil polyethylene to insulate the pump from the air. High volume pumps cannot be sealed in this manner since the heat of the motor may melt the plastic. The pump exhausts should be kept free.

vii. If recleaning is necessary, removal of this equipment from the work area must be handled with care. It is not possible to completely decontaminate the pump motor and parts since these areas cannot be wetted. To minimize any problems in this area, all equipment such as fans and pumps should be carefully wet wiped prior to removal from the abatement area. Wrapping and sealing low volume pumps in 6-mil polyethylene will provide easier decontamination of this equipment. Use of clean water and disposable wipes should be available for this purpose.

e. Pump flow rate equal to or greater than 1 L/min or less than 10 L/min may be used for 25 mm cassettes. The larger cassette diameters may have comparably increased flow.

f. Sample a volume of air sufficient to ensure the minimum quantitation limits. (See Table I of Unit III B 5j.)

8. Ambient sampling.

a. Position ambient samplers at locations representative of the air entering the abatement site. If makeup air entering the abatement site is drawn from another area of the building which is outside of the abatement area, place the pumps in the building, pumps should be placed out of doors located near the building and away from any obstructions that may influence wind patterns. If construction is in progress immediately outside the enclosure, it may be necessary to select another ambient site. Samples should be representative of any air entering the work site.

b. Locate the ambient samplers at least 3 ft apart and protect them from adverse weather conditions.

c. Sample same volume of air as samples taken inside the abatement site.

#### C. Sample Shipment

1. Ship bulk samples in a separate container from air samples. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected.

2. Select a rigid shipping container and pack the cassettes upright in a noncontaminating nonfibrous medium such as a bubble pack. The use of resealable polyethylene bags may help to prevent jostling of individual cassettes.

3. Avoid using expanded polystyrene because of its static charge potential. Also avoid using particle-based packaging materials because of possible contamination.

4. Include a shipping bill and a detailed listing of samples shipped, their descriptions

and all identifying numbers or marks, sampling data, shipper's name, and contact information. For each sample set, designate which are the ambient samples, which are the abatement area samples, which are the field blanks, and which is the sealed blank if sequential analysis is to be performed.

5. Hand-carry samples to the laboratory in an upright position if possible; otherwise choose that mode of transportation least likely to jar the samples in transit.

6. Address the package to the laboratory sample coordinator by name when known and alert him or her of the package description, shipment mode, and anticipated arrival as part of the chain of custody and sample tracking procedures. This will also help the laboratory schedule timely analysis for the samples when they are received.

#### D. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards is performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined, and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the text below.

1. Prescreen the loaded cassette collection filters to assure that they do not contain concentrations of asbestos which may interfere with the analysis of the sample. A filter blank average of less than 18  $s/mm^2$  in an area of 0.057  $mm^2$  (nominally 10 200-mesh grid openings) and a maximum of 53  $s/mm^2$  for that same area for any single preparation is acceptable for this method.

2. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter—not the filter which will be used in sampling—before and after the sampling operation.

3. Record all calibration information with the data to be used on a standard sampling form.

4. Ensure that the samples are stored in a secure and representative location.

5. Ensure that mechanical calibrations from the pump will be minimized to prevent transferral of vibration to the cassette.

6. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by installing a damping chamber if necessary.

7. Open a loaded cassette momentarily at one of the indoor sampling sites when sampling is initiated. This sample will serve as an indoor field blank.

8. Open a loaded cassette momentarily at one of the outdoor sampling sites when sampling is initiated. This sample will serve as an outdoor field blank.

9. Carry a sealed blank into the field with each sample series. Do not open this cassette in the field.

10. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.

11. Ensure that the sampler is turned upright before interrupting the pump flow.

12. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory.

#### E. Sample Receiving

1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.

2. Adhere to the following procedures to ensure both the continued chain-of-custody and the accountability of all samples passing through the laboratory:

a. Note the condition of the shipping package and data written on it upon receipt.

b. Retain all bills of lading or shipping slips to document the shipper and delivery time.

c. Examine the chain-of-custody seal, if any, and the package for its integrity.

d. If there has been a break in the seal or substantive damage to the package, the sample coordinator shall immediately notify the shipper and a responsible laboratory manager before any action is taken to unpack the shipment.

e. Packages with significant damage shall be accepted only by the responsible laboratory manager after discussions with the client.

3. Unwrap the shipment in a clean, uncluttered facility. The sample coordinator or his or her designee will record the contents, including a description of each item and all identifying numbers or marks. A

Sample Receiving Form to document this information is attached for use when necessary. (See the following Figure 3.)

FIGURE 3--SAMPLE RECEIVING FORM

Date of package delivery \_\_\_\_\_ Package shipped from \_\_\_\_\_  
 Carrier \_\_\_\_\_ Shipping bill retained \_\_\_\_\_  
 \*Condition of package on receipt \_\_\_\_\_  
 \*Condition of custody seal \_\_\_\_\_  
 Number of samples received \_\_\_\_\_ Shipping manifest attached \_\_\_\_\_  
 Purchase Order No. \_\_\_\_\_ Project I.D. \_\_\_\_\_  
 Comments \_\_\_\_\_

No.	Description	Sampling Medium		Sampled Volume Liters	Receiving ID #	Assigned #
		EC	MCE			
1	_____	_____	_____	_____	_____	_____
2	_____	_____	_____	_____	_____	_____
3	_____	_____	_____	_____	_____	_____
4	_____	_____	_____	_____	_____	_____
5	_____	_____	_____	_____	_____	_____
6	_____	_____	_____	_____	_____	_____
7	_____	_____	_____	_____	_____	_____
8	_____	_____	_____	_____	_____	_____
9	_____	_____	_____	_____	_____	_____
10	_____	_____	_____	_____	_____	_____
11	_____	_____	_____	_____	_____	_____
12	_____	_____	_____	_____	_____	_____
13	_____	_____	_____	_____	_____	_____

(Use as many additional sheets as needed.)  
 Comments \_\_\_\_\_  
 Date of acceptance into sample bank \_\_\_\_\_  
 Signature of chain-of-custody recipient \_\_\_\_\_  
 Disposition of samples \_\_\_\_\_

\*Note: If the package has sustained substantial damage or the custody seal is broken, stop and contact the project manager and the shipper.

NOTE: The person breaking the chain-of-custody seal and itemizing the contents assumes responsibility for the shipment and signs documents accordingly.

4. Assign a laboratory number and schedule an analysis sequence.

5. Manage all chain-of-custody samples within the laboratory such that their integrity can be ensured and documented.

#### F. Sample Preparation

1. Personnel not affiliated with the Abatement Contractor shall be used to prepare samples and conduct TEM analysis. Wet-wipe the exterior of the cassettes to minimize contamination possibilities before taking them to the clean sample preparation facility.

2. Perform sample preparation in a well-equipped clean facility.

NOTE: The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA filtered. The cumulative analytical blank concentration must average less than 18 s/mm<sup>2</sup> in an area of 0.057 s/mm<sup>2</sup> (nominally 10 200-mesh grid openings) with no more than one single preparation to exceed 53 s/mm<sup>2</sup> for that same area.

3. Preparation areas for air samples must be separated from preparation areas for bulk samples. Personnel must not prepare air samples if they have previously been preparing bulk samples without performing appropriate personal hygiene procedures, i.e., clothing change, showering, etc.

4. *Preparation.* Direct preparation techniques are required. The objective is to produce an intact carbon film containing the particulates from the filter surface which is sufficiently clear for TEM analysis. Currently recommended direct preparation procedures for polycarbonate (PC) and mixed cellulose ester (MCE) filters are described in Unit III.F.7, and 8. Sample preparation is a subject requiring additional research. Variation on those steps which do not substantively change the procedure, which improve filter clearing or which reduce contamination problems in a laboratory are permitted.

a. Use only TEM grids that have had grid opening areas measured according to directions in Unit III.J.

b. Remove the inlet and outlet plugs prior to opening the cassette to minimize any pressure differential that may be present.

c. Examples of techniques used to prepare polycarbonate filters are described in Unit III.F.7.

d. Examples of techniques used to prepare mixed cellulose ester filters are described in Unit III.F.8.

e. Prepare multiple grids for each sample.

f. Store the three grids to be measured in appropriately labeled grid holders or polyethylene capsules.

5. Equipment.

a. Clean area.

b. Tweezers. Fine-point tweezers for handling of filters and TEM grids.

c. Scalpel Holder and Curved No. 10 Surgical Blades.

d. Microscope slides.

e. Double-coated adhesive tape.

f. Gummed page reinforcements.

g. Micro-pipet with disposal tips 10 to 100  $\mu$ L variable volume.

h. Vacuum coating unit with facilities for evaporation of carbon. Use of a liquid nitrogen cold trap above the diffusion pump will minimize the possibility of contamination of the filter surface by oil from the pumping system. The vacuum-coating unit can also be used for deposition of a thin film of gold.

i. *Carbon rod electrodes.* Spectrochemically pure carbon rods are required for use in the vacuum evaporator for carbon coating of filters.

j. *Carbon rod sharpener.* This is used to sharpen carbon rods to a neck. The use of necked carbon rods (or equivalent) allows the carbon to be applied to the filters with a minimum of heating.

k. *Low-temperature plasma asher.* This is used to etch the surface of collapsed mixed cellulose ester (MCE) filters. The asher should be supplied with oxygen, and should be modified as necessary to provide a throttle or bleed valve to control the speed of the vacuum to minimize disturbance of the filter. Some early models of ashers admit air too rapidly, which may disturb particulates on the surface of the filter during the etching step.

l. *Glass petri dishes, 10 cm in diameter, 1 cm high.* For prevention of excessive evaporation of solvent when these are in use, a good seal must be provided between the base and the lid. The seal can be improved by grinding the base and lid together with an abrasive grinding material.

m. Stainless steel mesh.

n. Lens tissue.

o. Copper 200-mesh TEM grids, 3 mm in diameter, or equivalent.

p. Gold 200-mesh TEM grids, 3 mm in diameter, or equivalent.

q. Condensation washer.

r. Carbon-coated, 200-mesh TEM grids, or equivalent.

s. Analytical balance, 0.1 mg sensitivity.

t. Filter paper, 9 cm in diameter.

u. Oven or slide warmer. Must be capable of maintaining a temperature of 65-70 °C.

v. Polyurethane foam, 5 mm thickness.

w. Gold wire for evaporation.

6. Reagents.

a. *General.* A supply of ultra-clean, fiber-free water must be available for washing of all components used in the analysis. Water

that has been distilled in glass or filtered or deionized water is satisfactory for this purpose. Reagents must be fiber-free.

b. Polycarbonate preparation method—chloroform.

c. Mixed Cellulose Ester (MCE) preparation method—acetone or the Burdette procedure (Ref. 7 of Unit III.L.).

7. TEM specimen preparation from polycarbonate filters.

a. *Specimen preparation laboratory.* It is most important to ensure that contamination of TEM specimens by extraneous asbestos fibers is minimized during preparation.

b. *Cleaning of sample cassettes.* Upon receipt at the analytical laboratory and before they are taken into the clean facility or laminar flow hood, the sample cassettes must be cleaned of any contamination adhering to the outside surfaces.

c. *Preparation of the carbon evaporator.* If the polycarbonate filter has already been carbon-coated prior to receipt, the carbon coating step will be omitted, unless the analyst believes the carbon film is too thin. If there is a need to apply more carbon, the filter will be treated in the same way as an uncoated filter. Carbon coating must be performed with a high-vacuum coating unit. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application, and must not be used. The carbon rods should be sharpened by a carbon rod sharpener to necks of about 4 mm long and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 10 to 12 cm from the surface of a microscope slide held in the rotating and tilting device.

d. *Selection of filter area for carbon coating.* Before preparation of the filters, a 75 mm x 50 mm microscope slide is washed and dried. This slide is used to support strips of filter during the carbon evaporation. Two parallel strips of double-sided adhesive tape are applied along the length of the slide. Polycarbonate filters are easily stretched during handling, and cutting of areas for further preparation must be performed with great care. The filter and the MCE backing filter are removed together from the cassette and placed on a cleaned glass microscope slide. The filter can be cut with a curved scalpel blade by rocking the blade from the point placed in contact with the filter. The process can be repeated to cut a strip approximately 3 mm wide across the diameter of the filter. The strip of polycarbonate filter is separated from the corresponding strip of backing filter and carefully placed so that it bridges the gap between the adhesive tape strips on the microscope slide. The filter strip can be held with fine-point tweezers and supported underneath by the scalpel blade during placement on the microscope slide. The analyst can place several such

strips on the same microscope slide, taking care to rinse and wet-wipe the scalpel blade and tweezers before handling a new sample. The filter strips should be identified by etching the glass slide or marking the slide using a marker insoluble in water and solvents. After the filter strip has been cut from each filter, the residual parts of the filter must be returned to the cassette and held in position by reassembly of the cassette. The cassette will then be archived for a period of 30 days or returned to the client upon request.

e. *Carbon coating of filter strips.* The glass slide holding the filter strips is placed on the rotation-tilting device, and the evaporator chamber is evacuated. The evaporation must be performed in very short bursts, separated by some seconds to allow the electrodes to cool. If evaporation is too rapid, the strips of polycarbonate filter will begin to curl, which will lead to cross-linking of the surface material and make it relatively insoluble in chloroform. An experienced analyst can judge the thickness of carbon film to be applied, and some test should be made first on unused filters. If the film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen. If the coating is too thick, the filter will tend to curl when exposed to chloroform vapor and the carbon film may not adhere to the support mesh. Too thick a carbon film will also lead to a TEM image that is lacking in contrast, and the ability to obtain ED patterns will be compromised. The carbon film should be as thin as possible and remain intact on most of the grid openings of the TEM specimen intact.

f. *Preparation of the Jaffe washer.* The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used. A washer consisting of a simple stainless steel bridge is recommended. Several pieces of lens tissue approximately 1.0 cm x 0.5 cm are placed on the stainless steel bridge, and the washer is filled with chloroform to a level where the meniscus contacts the underside of the mesh, which results in saturation of the lens tissue. See References 8 and 10 of Unit III.L.

g. *Placing of specimens into the Jaffe washer.* The TEM grids are first placed on a piece of lens tissue so that individual grids can be picked up with tweezers. Using a curved scalpel blade, the analyst excises three 3 mm square pieces of the carbon-coated polycarbonate filter from the filter strip. The three squares are selected from the center of the strip and from two points between the outer periphery of the active surface and the center. The piece of filter is placed on a TEM specimen grid with the shiny side of the TEM grid facing upwards, and the whole assembly is placed boldly onto the saturated lens tissue in the Jaffe washer. If carbon-coated grids are used, the filter should be

placed carbon-coated side down. The three excised squares of filters are placed on the same piece of lens tissue. Any number of separate pieces of lens tissue may be placed in the same Jaffe washer. The lid is then placed on the Jaffe washer, and the system is allowed to stand for several hours, preferably overnight.

h. *Condensation washing.* It has been found that many polycarbonate filters will not dissolve completely in the Jaffe washer, even after being exposed to chloroform for as long as 3 days. This problem becomes more serious if the surface of the filter was overheated during the carbon evaporation. The presence of undissolved filter medium on the TEM preparation leads to partial or complete obscuration of areas of the sample, and fibers that may be present in these areas of the specimen will be overlooked; this will lead to a low result. Undissolved filter medium also compromises the ability to obtain ED patterns. Before they are counted, TEM grids must be examined critically to determine whether they are adequately cleared of residual filter medium. It has been found that condensation washing of the grids after the initial Jaffe washer treatment, with chloroform as the solvent, clears all residual filter medium in a period of approximately 1 hour. In practice, the piece of lens tissue supporting the specimen grids is transferred to the cold finger of the condensation washer, and the washer is operated for about 1 hour. If the specimens are cleared satisfactorily by the Jaffe washer alone, the condensation washer step may be unnecessary.

8. TEM specimen preparation from MCE filters.

a. This method of preparing TEM specimens from MCE filters is similar to that specified in NIOSH Method 7402. See References 7, 8, and 9 of Unit III.L.

b. Upon receipt at the analytical laboratory, the sample cassettes must be cleaned of any contamination adhering to the outside surfaces before entering the clean sample preparation area.

c. Remove a section from any quadrant of the sample and blank filters.

d. Place the section on a clean microscope slide. Affix the filter section to the slide with a gummed paper reinforcement or other suitable means. Label the slide with a water and solvent-proof marking pen.

e. 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 minutes for the sample filter to fuse and clear.

f. Plasma etching of the collapsed filter is required.

i. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify

the conditions that should be used. This is one area of the method that requires further evaluation. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for a particular asher and operating conditions will then be set such that a 1-2  $\mu\text{m}$  (10 percent) layer of collapsed surface will be removed.

ii. Place the slide containing the collapsed filters into a low-temperature plasma asher, and etch the filter.

g. Transfer the slide to a rotating stage inside the bell jar of a vacuum evaporator. Evaporate a 1 mm x 3 mm section of graphite rod onto the cleared filter. Remove the slide to a clean, dry, covered petri dish.

h. Prepare a second petri dish as a Jaffe washer with the wicking substrate prepared from filter or lens paper placed on top of a 6 mm thick disk of clean spongy polyurethane foam. Cut a V-notch on the edge of the foam and filter paper. Use the V-notch as a reservoir for adding solvent. The wicking substrate should be thin enough to fit into the petri dish without touching the lid.

i. Place carbon-coated TEM grids face up 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 lid 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. The level of acetone should be just high enough to saturate the filter paper without creating puddles.

j. Remove about a quarter section of the carbon-coated filter samples from the glass slides using a surgical knife and tweezers. Carefully place the section of the 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 bring the acetone level up to the highest possible level without disturbing the sample preparations. Cover the petri dish. Elevate one side of the petri dish by placing a slide under it. This allows drops of condensed solvent vapors to form near the edge rather than in the center where they would drip onto the grid preparation.

#### G. TEM Method

##### 1. Instrumentation

a. Use an 80-120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated graduations. If the TEM is equipped with EDXA it must either have a STEM attachment or be capable of producing a spot less than 250 nm

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in diameter at crossover. The microscope shall be calibrated routinely (see Unit III.J.) for magnification and camera constant.

b. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory. This must be an Energy Dispersive X-ray Detector mounted on TEM column and associated hardware/software to collect, save, and read out spectral information. Calibration of Multi-Channel Analyzer shall be checked regularly for Al at 1.48 KeV and Cu at 8.04 KeV, as well as the manufacturer's procedures.

1. Standard replica grating may be used to determine magnification (e.g., 2100 lines/mm).

ii. Gold standard may be used to determine camera constant.

c. Use a specimen holder with single tilt and/or double tilt capabilities.

2. Procedure.

a. Start a new Count Sheet for each sample to be analyzed. Record on count sheet: analyst's initials and date; lab sample number; client sample number microscope identification; magnification for analysis; number of predetermined grid openings to be analyzed; and grid identification. See the following Figure 4:



1. Individual grid openings with greater than 5 percent openings (holes) or covered with greater than 25 percent particulate matter or obviously having nonuniform loading shall not be analyzed.

ii. Examine the grid at low magnification (<1000X) to determine its suitability for detailed study at higher magnifications.

iii. Reject the grid if:

(1) Less than 50 percent of the grid openings covered by the replica are intact.

(2) It is doubled or folded.

(3) It is too dark because of incomplete dissolution of the filter.

iv. If the grid is rejected, load the next sample grid.

v. If the grid is acceptable, continue on to Step 6 if mapping is to be used; otherwise proceed to Step 7.

f. Grid Map (Optional).

i. Set the TEM to the low magnification mode.

ii. Use flat edge or finder grids for mapping.

iii. Index the grid openings (fields) to be counted by marking the acceptable fields for one-half (0.5) of the area needed for analysis on each of the two grids to be analyzed. These may be marked just before examining each grid opening (field), if desired.

iv. Draw in any details which will allow the grid to be properly oriented if it is re-

loaded into the microscope and a particular field is to be reliably identified.

g. Scan the grid.

i. Select a field to start the examination.

ii. Choose the appropriate magnification (15,000 to 20,000X screen magnification).

iii. Scan the grid as follows.

(1) At the selected magnification, make a series of parallel traverses across the field. On reaching the end of one traverse, move the image one window and reverse the traverse.

NOTE: A slight overlap should be used so as not to miss any part of the grid opening (field).

(2) Make parallel traverses until the entire grid opening (field) has been scanned.

h. Identify each structure for appearance and size.

i. Appearance and size: Any continuous grouping of particles in which an asbestos fiber within aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5  $\mu\text{m}$  is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, or 2 definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. See the following Figure 5:

FIGURE 5--COUNTING GUIDELINES USED IN DETERMINING ASBESTOS STRUCTURES

Count as 1 fiber; 1 Structure; no intersections.



Count as 2 fibers if space between fibers is greater than width of 1 fiber diameter or number of intersections is equal to or less than 1.



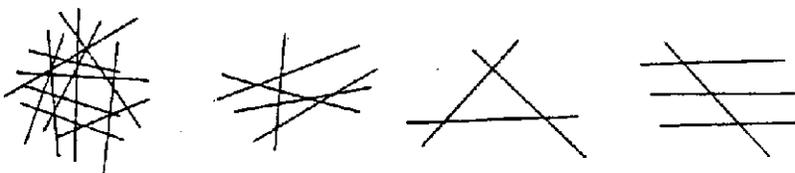
Count as 3 structures if space between fibers is greater than width of 1 fiber diameter or if the number of intersections is equal to or less than 2.



Count bundles as 1 structure; 3 or more parallel fibrils less than 1 fiber diameter separation.



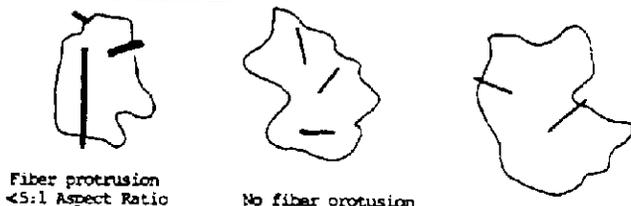
Count clusters as 1 structure; fibers having greater than or equal to 3 intersections.



Count matrix as 1 structure.



DO NOT COUNT AS STRUCTURES:



Fiber protrusion  
<5:1 Aspect Ratio

No fiber protrusion

Fiber protrusion  
<0.5 micrometer

— <0.5 micrometer in length  
— <5:1 Aspect Ratio

An intersection is a non-parallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater. Combinations such as a matrix and cluster, matrix and bundle, or bundle and cluster are categorized by the dominant fiber quality—cluster, bundle, and matrix, respectively. Separate categories will be maintained for fibers less than 5  $\mu\text{m}$  and for fibers greater than or equal to 5  $\mu\text{m}$  in length. Not required, but useful, may be to record the fiber length in 1  $\mu\text{m}$  intervals. (Identify each structure morphologically and analyze it as it enters the "window".)

(1) *Fiber*. A structure having a minimum length greater than 0.5  $\mu\text{m}$  and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed, no intersections.

(2) *Bundle*. A structure composed of 3 or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

(3) *Cluster*. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings must have more than 2 intersections.

(4) *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

(5) *NSD*. Record NSD when no structures are detected in the field.

(6) *Intersection*. Non-parallel touching or crossing of fibers, with the projection having an aspect ratio 5:1 or greater.

ii. Structure Measurement.

(1) Recognize the structure that is to be sized.

(2) Memorize its location in the "window" relative to the sides, inscribed square and to other particulates in the field so this exact location can be found again when scanning is resumed.

(3) Measure the structure using the scale on the screen.

(4) Record the length category and structure type classification on the count sheet after the field number and fiber number.

(5) Return the fiber to its original location in the window and scan the rest of the field for other fibers; if the direction of travel is not remembered, return to the right side of the field and begin the traverse again.

i. Visual identification of Electron Diffraction (ED) patterns is required for each asbestos structure counted which would cause the analysis to exceed the 70 s/mm<sup>2</sup> concentration. (Generally this means the first four fibers identified as asbestos must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)

i. Center the structure, focus, and obtain an ED pattern. (See Microscope Instruction Manual for more detailed instructions.)

ii. From a visual examination of the ED pattern, obtained with a short camera length, classify the observed structure as belonging to one of the following classifications: chrysotile, amphibole, or nonasbestos.

(1) Chrysotile: The chrysotile asbestos pattern has characteristic streaks on the layer lines other than the central line and some streaking also on the central line. There will be spots of normal sharpness on the central layer line and on alternate lines (2nd, 4th, etc.). The repeat distance between layer lines is 0.53 nm and the center doublet is at 0.73 nm. The pattern should display (002), (110), (130) diffraction maxima; distances and geometry should match a chrysotile pattern and be measured semiquantitatively.

(2) Amphibole Group [includes grunerite (amosite), crocidolite, anthophyllite, tremolite, and actinolite]: Amphibole asbestos fiber patterns show layer lines formed by very closely spaced dots, and the repeat distance between layer lines is also about 0.53 nm. Streaking in layer lines is occasionally present due to crystal structure defects.

(3) Nonasbestos: Incomplete or unobtainable ED patterns, a nonasbestos EDXA, or a nonasbestos morphology

iii. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual identification based on his measured patterns. In the event that examination of the pattern by the qualified individual indicates that the pattern had been misidentified visually, the client shall be contacted. If the pattern is a suspected chrysotile, take a photograph of the diffraction pattern at 0 degrees tilt. If the structure is suspected to be amphibole, the sample may have to be tilted to obtain a simple geometric array of spots.

j. Energy Dispersive X-Ray Analysis (EDXA).

i. Required of all amphiboles which would cause the analysis results to exceed the 70 s/mm<sup>2</sup> concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)

ii. Can be used alone to confirm chrysotile after the 70 s/mm<sup>2</sup> concentration has been exceeded.

iii. Can be used alone to confirm all non-asbestos.

iv. Compare spectrum profiles with profiles obtained from asbestos standards. The closest match identifies and categorizes the structure.

v. If the EDXA is used for confirmation, record the properly labeled spectrum on a computer disk, or if a hard copy, file with analysis data.

vi. If the number of fibers in the non-asbestos class would cause the analysis to exceed the 70 s/mm<sup>2</sup> concentration, their identities must be confirmed by EDXA or measurement of a zone axis diffraction pattern to establish that the particles are non-asbestos.

k. Stopping Rules.

i. If more than 50 asbestiform structures are counted in a particular grid opening, the analysis may be terminated.

ii. After having counted 50 asbestiform structures in a minimum of 4 grid openings, the analysis may be terminated. The grid opening in which the 50th fiber was counted must be completed.

iii. For blank samples, the analysis is always continued until 10 grid openings have been analyzed.

iv. In all other samples the analysis shall be continued until an analytical sensitivity of 0.005 s/cm<sup>2</sup> is reached.

l. Recording Rules. The count sheet should contain the following information:

i. Field (grid opening): List field number.

ii. Record "NSD" if no structures are detected.

iii. Structure information.

(1) If fibers, bundles, clusters, and/or matrices are found, list them in consecutive numerical order, starting over with each field.

(2) Length. Record length category of asbestos fibers examined. Indicate if less than 5  $\mu\text{m}$  or greater than or equal to 5  $\mu\text{m}$ .

(3) Structure Type. Positive identification of asbestos fibers is required by the method. At least one diffraction pattern of each fiber type from every five samples must be recorded and compared with a standard diffraction pattern. For each asbestos fiber reported, both a morphological descriptor and an identification descriptor shall be specified on the count sheet.

(4) Fibers classified as chrysotile must be identified by diffraction and/or X-ray analysis and recorded on the count sheet. X-ray analysis alone can be used as sole identification only after 70s/mm<sup>2</sup> have been exceeded for a particular sample.

(5) Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. (X-ray analysis alone can be used as sole identification only after 70s/mm<sup>2</sup> have been exceeded for a particular sample.)

(6) If a diffraction pattern was recorded on film, the micrograph number must be indicated on the count sheet.

(7) If an electron diffraction was attempted and an appropriate spectra is not observed, N should be recorded on the count sheet.

(8) If an X-ray analysis is attempted but not observed, N should be recorded on the count sheet.

(9) If an X-ray analysis spectrum is stored, the file and disk number must be recorded on the count sheet.

m. Classification Rules.

i. *Fiber*. A structure having a minimum length greater than or equal to 0.5  $\mu\text{m}$  and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

ii. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

iii. *Cluster*. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated

from the group. Groupings must have more than two intersections.

iv. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

v. *NSD*. Record NSD when no structures are detected in the field.

n. After all necessary analyses of a particle structure have been completed, return the goniometer stage to 0 degrees, and return the structure to its original location by recall of the original location.

o. Continue scanning until all the structures are identified, classified and sized in the field.

p. Select additional fields (grid openings) at low magnification; scan at a chosen magnification (15,000 to 20,000X screen magnification); and analyze until the stopping rule becomes applicable.

q. Carefully record all data as they are being collected, and check for accuracy.

r. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid hold. Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request.

H. Sample Analytical Sequence

1. Carry out visual inspection of work site prior to air monitoring.

2. Collect a minimum of five air samples inside the work site and five samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.

3. Analyze the abatement area samples according to this protocol. The analysis must meet the 0.005 s/cm<sup>3</sup> analytical sensitivity.

4. Remaining steps in the analytical sequence are contained in Unit IV. of this Appendix.

I. Reporting

The following information must be reported to the client. See the following Table I:



9. Signature of laboratory official to indicate that the laboratory met specifications of the AHERA method.

10. Report form must contain official laboratory identification (e.g., letterhead).

11. Type of asbestos.

#### J. Calibration Methodology

**NOTE:** Appropriate implementation of the method requires a person knowledgeable in electron diffraction and mineral identification by ED and EDXA. Those inexperienced laboratories wishing to develop capabilities may acquire necessary knowledge through analysis of appropriate standards and by following detailed methods as described in References 8 and 10 of Unit III.L.

1. *Equipment Calibration.* In this method, calibration is required for the air-sampling equipment and the transmission electron microscope (TEM).

a. *TEM Magnification.* The magnification at the fluorescent screen of the TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica. A logbook must be maintained, and the dates of calibration depend on the past history of the particular microscope; no frequency is specified. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate an eucentric tilting specimen stage, all specimens and the cross grating replica must be placed at the eucentric position.

b. *Determination of the TEM magnification on the fluorescent screen*

i. Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

ii. Insert a diffraction grating replica (for example a grating containing 2,160 lines/mm) 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 the goniometer stage tilt is 0 degrees.

iii. Adjust microscope magnification to 10,000X or 20,000X. Measure the distance (mm) between two widely separated lines on the grating replica. Note the number of spaces between the lines. Take care to measure between the same relative positions on the lines (e.g., between left edges of lines).

**NOTE:** The more spaces included in the measurement, the more accurate the final

calculation. On most microscopes, however, the magnification is substantially constant only within the central 8-10 cm diameter region of the fluorescent screen.

iv. Calculate the true magnification (M) on the fluorescent screen:

$$M = XC/Y$$

where:

X=total distance (mm) between the designated grating lines;

C=calibration constant of the grating replica (lines/mm);

Y=number of grating replica spaces counted along X.

c. *Calibration of the EDXA System.* Initially, the EDXA system must be calibrated by using two reference elements to calibrate the energy scale of the instrument. When this has been completed in accordance with the manufacturer's instructions, calibration in terms of the different types of asbestos can proceed. The EDXA detectors vary in both solid angle of detection and in window thickness. Therefore, at a particular accelerating voltage in use on the TEM, the count rate obtained from specific dimensions of fiber will vary both in absolute X-ray count rate and in the relative X-ray peak heights for different elements. Only a few minerals are relevant for asbestos abatement work, and in this procedure the calibration is specified in terms of a "fingerprint" technique. The EDXA spectra must be recorded from individual fibers of the relevant minerals, and identifications are made on the basis of semiquantitative comparisons with these reference spectra.

d. *Calibration of Grid Openings.*

i. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass slide and examining it under the PCM. Use a calibrated graticule to measure the average field diameter and use this number to calculate the field area for an average grid opening. Grids are to be randomly selected from batches up to 1,000.

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

ii. The mean grid opening area must be measured for the type of specimen grids in use. This can be accomplished on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.

e. *Determination of Camera Constant and ED Pattern Analysis.*

i. The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved by using a carbon-coated grid on which a thin film of gold has been

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sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film.

ii. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulates. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one-half the diameter,  $D$ , of the rings times the interplanar spacing,  $d$ , of the ring being measured.

### K. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of sensitive sam-

pling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards is performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:

TABLE III--SUMMARY OF LABORATORY  
DATA QUALITY OBJECTIVES

Unit Operation	QC Check	Frequency	Conformance Expected
Sample receiving	Review of receiving report	Each sample	95% complete
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample preparation	Supplies and reagents	On receipt	Meet specs. or reject
	Grid opening size	20 openings/20 grids/lot of 1000 or 1 opening/sample	100%
	Special clean area monitoring	After cleaning or service	Meet specs or re-clean
	Laboratory blank	1 per prep series or 10%	Meet specs. or reanalyze series
	Plasma each blank	1 per 20 samples	75%
	Multiple preps (3 per sample)	Each sample	One with cover of 15 complete grid sqs.
	Sample analysis	System check	Each day
	Alignment check	Each day	Each day
	Magnification calibration with low and high standards	Each month or after service	95%
	ED calibration by gold standard	Weekly	95%
	EDS calibration by copper line	Daily	95%
Performance check	Laboratory blank (measure of cleanliness)	Prep 1 per series or 10% read 1 per 25 samples	Meet specs or reanalyze series
	Replicate counting (measure of precision)	1 per 100 samples	1.5 x Poisson Std. Dev.
	Duplicate analysis (measure of reproducibility)	1 per 100 samples	2 x Poisson Std. Dev.
	Known samples of typical materials (working standards)	Training and for comparison with unknowns	100%
	Analysis of NBS SRM 1876 and/or RM 8410 (measure of accuracy and comparability)	1 per analysis per year	1.5 x Poisson Std. Dev.
	Data entry review (data validation and measure of completeness)	Each sample	95%
	Record and verify ID electron diffraction pattern of structure	1 per 5 samples	80% accuracy
Calculations and data reduction	Hand calculation of automated data reduction procedure or independent recalculation of hand-calculated data	1 per 100 samples	85%

1. When the samples arrive at the laboratory, check the samples and documentation for completeness and requirements before initiating the analysis.

2. Check all laboratory reagents and supplies for acceptable asbestos background levels.

3. Conduct all sample preparation in a clean room environment monitored by laboratory blanks and special testing after cleaning or servicing the room.

4. Prepare multiple grids of each sample.

5. Provide laboratory blanks with each sample batch. Maintain a cumulative average of these results. If this average is greater than 53  $\mu\text{m}^2$  per 10 200-mesh grid openings, check the system for possible sources of contamination.

6. Check for recovery of asbestos from cellulose ester filters submitted to plasma asher.

7. Check for asbestos carryover in the plasma asher by including a blank alongside the positive control sample.

8. Perform a systems check on the transmission electron microscope daily.

9. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III of Unit III.K.

10. Ensure qualified operator performance by evaluation of replicate counting, duplicate analysis, and standard sample comparisons as set forth in Table III of Unit III.K.

11. Validate all data entries.

12. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III.

13. Record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions.

The outline of quality control procedures presented above is viewed as the minimum required to assure that quality data is produced for clearance testing of an asbestos abated area. Additional information may be gained by other control tests. Specifics on those control procedures and options available for environmental testing can be obtained by consulting References 6, 7, and 11 of Unit III.L.

#### L. References

For additional background information on this method the following references should be consulted.

1. "Guidelines for Controlling Asbestos-Containing Materials in Buildings." EPA 560/5-85-024, June 1985.

2. "Measuring Airborne Asbestos Following an Abatement Action." USEP/Office of Pollution Prevention and Toxics, EPA 600/4-85-049, 1985.

3. Small, John and E. Steel. Asbestos Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.

4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751, U.S. Bureau of Mines, 1977.

5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods, EPA 600/4-77-027a, USEPA, Office of Research and Development, 1977.

6. Method 2A: Direct Measurement of Gas Volume Through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.

7. Burdette, G.J. Health & Safety Exec., Research & Lab. Services Div., London, "Proposed Analytical Method for Determination of Asbestos in Air."

8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc. "Standard Operating Procedure for Determination of Airborne Asbestos Fibers by Transmission Elec-

tron Microscopy Using Polycarbonate Membrane Filters." WERL SOP 87-1, March 5, 1987.

9. NIOSH. Method 7402 for Asbestos Fibers, December 11, 1986 Draft.

10. Yamate, C., S.C. Agarwall, R.D. Gibbons, IIT Research Institute. "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy." Draft report, USEPA Contract 68-02-3266, July 1984.

11. Guidance to the Preparation of Quality Assurance Project Plans. USEPA, Office of Pollution Prevention and Toxics, 1984.

#### IV. Mandatory Interpretation of Transmission Electron Microscopy Results to Determine Completion of Response Actions

##### A. Introduction

A response action is determined to be completed by TEM when the abatement area has been cleaned and the airborne asbestos concentration inside the abatement area is no higher than concentrations at locations outside the abatement area. "Outside" means outside the abatement area, but not necessarily outside the building. EPA reasons that an asbestos removal contractor cannot be expected to clean an abatement area to an airborne asbestos concentration that is lower than the concentration of air entering the abatement area from outdoors or from other parts of the building. After the abatement area has passed a thorough visual inspection, and before the outer containment barrier is removed, a minimum of five air samples inside the abatement area and a minimum of five air samples outside the abatement area must be collected. Hence, the response action is determined to be completed when the average airborne asbestos concentration measured inside the abatement area is not statistically different from the average airborne asbestos concentration measured outside the abatement area.

The inside and outside concentrations are compared by the Z-test, a statistical test that takes into account the variability in the measurement process. A minimum of five samples inside the abatement area and five samples outside the abatement area are required to control the false negative error rate, i.e., the probability of declaring the removal complete when, in fact, the air concentration inside the abatement area is significantly higher than outside the abatement area. Additional quality control is provided by requiring three blanks (filters through which no air has been drawn) to be analyzed to check for unusually high filter contamination that would distort the test results.

When volumes greater than or equal to 1,199 L for a 25 mm filter and 2,799 L for a 37 mm filter have been collected and the average number of asbestos structures on samples inside the abatement area is no greater than 70 /mm<sup>2</sup> of filter, the response action

may be considered complete without comparing the inside samples to the outside samples. EPA is permitting this initial screening test to save analysis costs in situations where the airborne asbestos concentration is sufficiently low so that it cannot be distinguished from the filter contamination/background level (fibers deposited on the filter that are unrelated to the air being sampled). The screening test cannot be used when volumes of less than 1,199 L for 25 mm filter or 2,799 L for a 37 mm filter are collected because the ability to distinguish levels significantly different from filter background is reduced at low volumes.

The initial screening test is expressed in structures per square millimeter of filter because filter background levels come from sources other than the air being sampled and cannot be meaningfully expressed as a concentration per cubic centimeter of air. The value of 70 s/mm<sup>2</sup> is based on the experience of the panel of microscopists who consider one structure in 10 grid openings (each grid opening with an area of 0.0057 mm<sup>2</sup>) to be comparable with contamination/background levels of blank filters. The decision is based, in part, on Poisson statistics which indicate that four structures must be counted on a filter before the fiber count is statistically distinguishable from the count for one structure. As more information on the performance of the method is collected, this criterion may be modified. Since different combinations of the number and size of grid openings are permitted under the TEM protocol, the criterion is expressed in structures per square millimeter of filter to be consistent across all combinations. Four structures per 10 grid openings corresponds to approximately 70 s/mm<sup>2</sup>.

#### B. Sample Collection and Analysis

1. A minimum of 13 samples is required: five samples collected inside the abatement area, five samples collected outside the abatement area, two field blanks, and one sealed blank.

2. Sampling and TEM analysis must be done according to either the mandatory or nonmandatory protocols in Appendix A. At least 0.057 mm<sup>2</sup> of filter must be examined on blank filters.

#### C. Interpretation of Results

1. The response action shall be considered complete if either:

a. Each sample collected inside the abatement area consists of at least 1,199 L of air for a 25 mm filter, or 2,799 L of air for a 37 mm filter, and the arithmetic mean of their asbestos structure concentrations per square millimeter of filter is less than or equal to 70 s/mm<sup>2</sup>; or

b. The three blank samples have an arithmetic mean of the asbestos structure con-

centration on the blank filters that is less than or equal to 70 s/mm<sup>2</sup> and the average airborne asbestos concentration measured inside the abatement area is not statistically higher than the average airborne asbestos concentration measured outside the abatement area as determined by the Z-test. The Z-test is carried out by calculating

$$Z = \frac{\bar{Y}_i - \bar{Y}_o}{0.8(1/n_i + 1/n_o)^{1/2}}$$

where  $\bar{Y}_i$  is the average of the natural logarithms of the inside samples and  $\bar{Y}_o$  is the average of the natural logarithms of the outside samples,  $n_i$  is the number of inside samples and  $n_o$  is the number of outside samples. The response action is considered complete if Z is less than or equal to 1.65.

NOTE: When no fibers are counted, the calculated detection limit for that analysis is inserted for the concentration.

2. If the abatement site does not satisfy either (1) or (2) of this Section C, the site must be recleaned and a new set of samples collected.

#### D. Sequence for Analyzing Samples

It is possible to determine completion of the response action without analyzing all samples. Also, at any point in the process, a decision may be made to terminate the analysis of existing samples, reclean the abatement site, and collect a new set of samples. The following sequence is outlined to minimize the number of analyses needed to reach a decision.

1. Analyze the inside samples.

2. If at least 1,199 L of air for a 25 mm filter or 2,799 L of air for a 37 mm filter is collected for each inside sample and the arithmetic mean concentration of structures per square millimeter of filter is less than or equal to 70 s/mm<sup>2</sup>, the response action is complete and no further analysis is needed.

3. If less than 1,199 L of air for a 25 mm filter or 2,799 L of air for a 37 mm filter is collected for any of the inside samples, or the arithmetic mean concentration of structures per square millimeter of filter is greater than 70 s/mm<sup>2</sup>, analyze the three blanks.

4. If the arithmetic mean concentration of structures per square millimeter on the blank filters is greater than 70 s/mm<sup>2</sup>, terminate the analysis, identify and correct the source of blank contamination, and collect a new set of samples.

5. If the arithmetic mean concentration of structures per square millimeter on the blank filters is less than or equal to 70 s/mm<sup>2</sup>, analyze the outside samples and perform the Z-test.

6. If the Z-statistic is less than or equal to 1.65, the response action is complete. If the Z-statistic is greater than 1.65, reclean the abatement site and collect a new set of samples.

[52 FR 41857, Oct. 30, 1987]

**APPENDIX B TO SUBPART E—WORK PRACTICES AND ENGINEERING CONTROLS FOR SMALL-SCALE, SHORT-DURATION OPERATIONS MAINTENANCE, AND REPAIR (O&M) ACTIVITIES INVOLVING ACM**

This appendix is not mandatory. In that LEAs may choose to comply with all the requirements of 40 CFR 763.121. Section 763.91(b) extends the protection provided by EPA in its 40 CFR 763.121 for worker protection during asbestos abatement projects to employees of local education agencies who perform small-scale, short-duration operations, maintenance and repair (O&M) activities involving asbestos-containing materials and are not covered by the OSHA asbestos construction standard at 29 CFR 1926.58 or an asbestos worker protection standard adopted by a State as part of a State plan approved by OSHA under section 18 of the Occupational Safety and Health Act. Employers wishing to be exempt from the requirements of §763.121 (e)(6) and (f)(2)(i) may instead comply with the provisions of this appendix when performing small-scale, short-duration O&M activities.

**Definition of Small-Scale, Short-Duration Activities**

For the purposes of this appendix, small-scale, short-duration maintenance activities are tasks such as, but not limited to:

1. Removal of asbestos-containing insulation on pipes.
2. Removal of small quantities of asbestos-containing insulation on beams or above ceilings.
3. Replacement of an asbestos-containing gasket on a valve.
4. Installation or removal of a small section of drywall.
5. Installation of electrical conduits through or proximate to asbestos-containing materials.

Small-scale, short-duration maintenance activities can be further defined, for the purposes of this subpart, by the following considerations:

1. Removal of small quantities of asbestos-containing materials (ACM) only if required in the performance of another maintenance activity not intended as asbestos abatement.
2. Removal of asbestos-containing thermal system insulation not to exceed amounts greater than those which can be contained in a single glove bag.

3. Minor repairs to damaged thermal system insulation which do not require removal.

4. Repairs to a piece of asbestos-containing wallboard.

5. Repairs, involving encapsulation, enclosure or removal, to small amounts of friable asbestos-containing material only if required in the performance of emergency or routine maintenance activity and not intended solely as asbestos abatement. Such work may not exceed amounts greater than those which can be contained in a single prefabricated minienclosure. Such an enclosure shall conform spatially and geometrically to the localized work area, in order to perform its intended containment function.

OSHA concluded that the use of certain engineering and work practice controls is capable of reducing employee exposures to asbestos to levels below the final standard's action level (0.1 f/cm<sup>3</sup>). (See 51 FR 22714, June 20, 1986.) Several controls and work practices, used either singly or in combination, can be employed effectively to reduce asbestos exposures during small maintenance and renovation operations. These include:

- I. Wet methods.
2. Removal methods.
  - i. Use of glove bags.
  - ii. Removal of entire asbestos insulated pipes or structures.
  - iii. Use of minienclosures.
3. Enclosure of asbestos materials.
4. Maintenance programs.

This appendix describes these controls and work practices in detail.

**Preparation of the Area Before Renovation or Maintenance Activities**

The first step in preparing to perform a small-scale, short-duration asbestos renovation or maintenance task, regardless of the abatement method that will be used, is the removal from the work area of all objects that are movable to protect them from asbestos contamination. Objects that cannot be removed must be covered completely with 6-mil-thick polyethylene plastic sheeting before the task begins. If objects have already been contaminated, they should be thoroughly cleaned with a High Efficiency Particulate Air (HEPA) filtered vacuum or be wet-wiped before they are removed from the work area or completely encased in the plastic.

*Wet methods.* Whenever feasible, and regardless of the abatement method to be used (e.g., removal, enclosure, use of glove bags), wet methods must be used during small-scale, short-duration maintenance and renovation activities that involve disturbing asbestos-containing materials. Handling asbestos materials wet is one of the most reliable methods of ensuring that asbestos fibers do not become airborne, and this practice should therefore be used whenever feasible.



California Environmental Protection Agency



Method 435

Determination of Asbestos  
Content of Serpentine Aggregate

Adopted: June 6, 1991



## Method 435

### Determination of Asbestos Content of Serpentine Aggregate

#### 1 PRINCIPLE AND APPLICABILITY

##### 1.1 Principle.

Asbestos fibers may be released from serpentine rock formations and are determined by microscopic techniques. The results are very sensitive to sampling procedures. The analytical results are reported in percent asbestos fibers which is the percent number of asbestos fibers contained in 400 randomly chosen particles of a bulk sample. Since the homogeneity of the material is unknown, the uncertainty in the sampling cannot be defined. The uncertainty of the analytical technique is two percent if twenty asbestos fibers are counted in a sample of 400 particles. The derivation of this uncertainty value is explained in Section 7.4.

##### 1.2 Applicability.

This method is applicable to determining asbestos content of serpentine aggregate in storage piles, on conveyor belts, and on surfaces such as roads, shoulders, and parking lots.

#### 2 DEFINITIONS

##### 2.1 Bulk Sample

A sample of bulk material.

##### 2.2 Grab Sample

A sample taken from a volume of material.

##### 2.3 Composite Sample

A mixture or blend of material from more than one grab sample.

##### 2.4 Serpentine

Serpentinite, serpentine rock or serpentine material.

## 2.5 Executive Officer

The term Executive Officer as used in this method shall mean the Executive Officer of the Air Resources Board (ARB) or Air Pollution Control Officer/Executive Officer of a local air pollution control district/air quality management district.

## 3 APPLICABLE SOURCES

This method can be used to obtain bulk material samples from three types of sources:

1. Serpentine aggregate storage piles,
2. Serpentine aggregate conveyor belts
3. Serpentine aggregate covered surfaces.

## 4 SAMPLING APPARATUS

### 4.1 Serpentine Aggregate Storage Piles.

Tube insertion often provides the simplest method of aggregate material investigation and sampling. Insertion tubes shall be adequate to provide a relatively rapid continuous penetration force.

- 4.1.1 Thin-walled tubes should be manufactured as shown in Figure 1. The tube should have an outside diameter between 2 to 5 inches and be made of metal or plastic having adequate strength for penetration into aggregate piles. These tubes shall be clean and free of surface irregularities including projecting weld seams. Further information on these tubes can be found in Table 1 and ASTM D 1587-83, which is incorporated herein by reference.
- 4.1.2 The insertion tube can be made out of commercially available two inch PVC Schedule 40 pipe. Further information on the tube can be found in Table 2.
- 4.1.3 A round point shovel may be used.
- 4.2 Serpentine Aggregate Conveyor Belts.
  - 4.2.1 Sampling of aggregate off a conveyor belt requires a hand trowel, a small brush, and a dust pan.
  - 4.2.2 Two templates as shown in Figure 2 are needed to isolate material on the conveyor belt.
  - 4.2.3 An automated belt sampler may be used.

#### 4.3 Serpentine Aggregate Covered Surfaces.

A shovel, a hand or machine-operated auger or other suitable equipment can be used to collect samples of aggregate materials on covered surfaces.

##### 4.3.1 Hand-Operated Augers.

4.3.1.1 Helical Augers-Small lightweight augers such as spiral-type augers and ship-type augers may be used. A description of these augers can be found in ASTM D1452-80, which is incorporated herein by reference.

4.3.1.2 Orchard barrel and open spiral-type tubular augers may be used to collect samples. These augers range in size from 1.5 through 8 inches, and have the common characteristic of appearing essentially tubular when viewed from the digging end. Further description of these auger types can be found in ASTM D1452-80.

4.3.1.3 Clam Shell or Iwan-Type post-hole augers may be used to collect samples from surfaces generally 2 through 8 inches in diameter and have a common mean of blocking the escape of soil from the auger. Further description of these augers can be found in ASTM D1452-80.

##### 4.3.2 Machine-Operated Augers

Machine-Operated Augers such as helical augers and stinger augers may be used. These augers are normally operated by heavy-duty, high-torque machines, designed for heavy construction work. Further description of these augers can be found in ASTM D1452-80.

4.3.3 A round point shovel can also be used to obtain a sample of aggregate covered surface material.

## 5 SAMPLING

The sampling procedure has been developed to provide an unbiased collection of bulk samples. A sampling plan, including a description of how the grab samples will be randomly collected and the number of samples to be collected, shall be developed. Prior to conducting any sampling the sampling plan shall be submitted to the Executive Officer for approval, if the sampling is conducted for determining compliance with a rule or regulation. The amount of composite 200 mesh material, as described below, shall be sufficient to provide sample to the source or Executive Officer, if requested, and a sample to be archived for future use.

A single test as described below shall cover:

- a) 1000 tons of aggregate for piles and conveyor belts, or
- b) one acre aggregate covered surface, or
- c) one mile of aggregate covered road, or

- d) two acres or two miles of dual aggregate covered shoulders.

Exposure to airborne asbestos fibers is a health hazard. Asbestos has been listed by the Governor as causing cancer and identified by the Air Resources Board as a toxic air contaminant. Serpentine aggregate may contain asbestos. Bulk samples collected can contain friable asbestos fibers and may release fibers during sampling, handling or crushing steps. Adequate safety precautions should be followed to minimize the inhalation of asbestos fibers. Crushing should be carried out in a ventilated hood with continuous airflow (negative pressure) exhausting through an HEPA filter. Handling of samples without these precautions may result in the inhalation of airborne asbestos fibers.

#### 5.1 Serpentine Aggregate Storage Piles.

Serpentine aggregate storage piles typically have a conical or a triangular prism shape. The aggregate is introduced at the top of the pile and is allowed to flow over the side. This action, called sloughing, causes a size segregation to occur with the finer material deposited towards the top of the pile.

The locations where grab samples will be taken are randomly chosen over the surface of the pile. The method of randomly choosing the sampling locations is left up to sampling personnel but must follow the procedures specified in the sampling personnel plan. For 1000 tons of product, a grab sample shall be taken at a minimum of three randomly chosen sampling locations. A minimum of three grab samples shall be taken even if the product pile contains less than 1000 tons of material. The slough is raked or shoveled away from the sampling location. A sampling apparatus is inserted one foot into the pile and the material is removed and is placed in an appropriate sized sampling container. Some of the possible sampling apparatus is discussed in Section 4.1. Each of the grab samples shall be placed in the same sample container. This composited sample shall be crushed to produce a material with a nominal size of less than three-eighths of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80, which is incorporated herein by reference, shall be used to reduce the size of the crushed grab sample to a one pint aliquot. The one pint aliquot shall be further crushed using a Braun mill or equivalent to produce a material of which the majority shall be less than 200 Tyler mesh. An aliquot of the 200 mesh material shall be put into a labeled sealed container. The label shall contain all the information described in Section 6 (except item 4).

#### 5.2 Serpentine Aggregate Conveyor Belts.

Serpentine aggregate is transported from the rock crushing plant to a product stacking belt and finally to a storage pile or to a waiting truck for delivery to a buyer.

The grab samples shall be taken from the product stacking belt or if this is not possible then at the first transfer point before the stockpile. The grab samples shall be collected by stopping the belt a minimum of three times or using an automated sampler. The method of randomly choosing the sampling locations and intervals is left up to sampling

personnel but must follow the procedure specified in the sampling plan. For 1000 tons of product, a grab sample is taken at a minimum of three randomly selected intervals. A minimum of three samples shall be taken even if the generated product is less than 1000 tons. Each time the belt is stopped to take a grab sample, templates, as shown in Figure 2, are placed a minimum of six inches apart to isolate the material on the belt. The material within the templates is removed with a small shovel or with a brush and a dust pan for the finer material and is placed in an appropriate sized sampling container. This composited sample shall be crushed to produce a material with a nominal size of less than three-eighths of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80, which is incorporated herein by reference, shall be used to reduce the size of the crushed grab sample to a one pint aliquot. The one pint aliquot shall be further crushed using a Bruan mill or equivalent to produce a material which the majority of which shall be less than 200 Tyler mesh. An aliquot of the 200 mesh material shall be put into a labeled sealed container. The label must contain all the information listed in Section 6 (except item 4).

### 5.3 Serpentine Aggregate Covered Surfaces.

#### 5.3.1 Serpentine Aggregate Covered Roads

A serpentine aggregate-covered road shall be characterized by taking grab samples from a minimum of three randomly chosen locations per mile of road. The method of randomly choosing the sampling locations is left up to sampling personnel but must follow the procedures specified in the sampling plan. A minimum of three samples shall be taken even if the road is less than one mile long. Section 4.3 describes some of the possible sampling apparatus used to collect the grab samples. Grab samples shall not contain underlying soils. Each of the grab samples shall be placed in the same sample container. This composited sample shall be crushed to produce a material with a nominal size of less than three-eighths of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80, which is incorporated herein by reference, shall be used to reduce the size of the crushed grab sample to a one pint aliquot. The one pint aliquot shall be further crushed using a Bruan mill or equivalent to produce a material which the majority of which shall be less than 200 Tyler mesh. An aliquot of the 200 mesh material shall be put into a labeled sealed container. The label must contain all the information listed in Section 6 (except item 4).

#### 5.3.2 Serpentine Aggregate Covered Areas

A serpentine aggregate-covered play yard or parking lot shall be characterized by taking grab samples from a minimum of three randomly chosen locations per acre. The method of randomly choosing the sampling locations is left up to sampling personnel but must follow the procedures specified in the sampling plan. A minimum of three samples shall be taken even if the road is less than one mile long. Section 4.3 describes some of the possible sampling apparatus used to collect the grab samples.

Grab samples shall not contain underlying soils. Each of the grab samples shall be placed in the same sample container. This composited sample shall be crushed to produce a material with a nominal size of less than three-eighths of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80, which is incorporated herein by reference, shall be used to reduce the size of the crushed grab sample to a one pint aliquot. The one pint aliquot shall be further crushed using a Bruan mill or equivalent to produce a material which the majority of which shall be less than 200 Tyler mesh. An aliquot of the 200 mesh material shall be put into a labeled sealed container. The label must contain all the information listed in Section 6 (except item 4).

### 5.3.3 Serpentine Aggregate Covered Road Shoulders

The sampling procedure specified in Section 5.3.1 or 5.3.2 shall be used for road shoulders covered with serpentine aggregate. The only difference is that a minimum of three grab samples shall be taken over a length of two miles of shoulder or over an area of two acres of shoulder surface. The word shoulder is meant to imply shoulders on both sides of the road. For serpentine aggregated covered shoulders, the sampling plan specified in Section 5 shall indicate whether the samples are collected on a two mile or two acre basis.

## 6 SAMPLING LOG

A sample log must be kept showing:

- 1) A unique sample number.
- 2) Facility name.
- 3) Facility address or location where sample is taken.
- 4) A rough sketch, video tape, or photograph of the specific sampling locations.
- 5) Date and time of sampling.
- 6) Name of person performing sampling.

## 7 ANALYTICAL PROCEDURES

### 7.1 Principle and Applicability.

Samples of serpentine aggregate taken for asbestos identification are first examined for homogeneity and preliminary fiber identification at low magnification. Positive identification of suspect fibers is made by analysis of subsamples with the polarized light microscope.

The principles of optical mineralogy are well established.<sup>2,3</sup> A light microscope equipped with two polarizing filters coupled with dispersion staining is used to observe specific optical characteristics of a sample. The use of plane polarized light allows the determination of refractive indices along specific crystallographic axes. Morphology and

color are also observed. A retardation plate is placed in the polarized light path for determination of the sign of elongation using orthoscopic illumination. Orientation of the two filters such that their vibration planes are perpendicular (cross polars) allows observation of the birefringence and extinction characteristics of anisotropic particles.

Quantitative analysis involves the use of point counting. Point counting is a standard technique in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting<sup>3</sup> and the interpretation of point count data<sup>4</sup> is available.

This method is applicable to all bulk samples of serpentine aggregate submitted for identification and quantification of asbestos components.

## 7.2 Range.

The analytical method may be used for analysis of samples containing from 0 to 100 percent asbestos. The upper detection limit is 100 percent. The lower detection limit is 0.25 percent.

## 7.3 Interferences.

Fibrous organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the asbestos content. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in the identification.

## 7.4 Analytical Uncertainty.

The uncertainty method is two percent if twenty asbestos fibers are counted in a sample of 400 particles. The uncertainty of the analytical method may be assessed by a 95% confidence interval for the true percentage of asbestos fibers in the rock. The number of asbestos fibers in the sample is assumed to have a binomial distribution. If twenty asbestos fibers are found in a sample of 400 particles, a one-sided confidence interval for the true percentage has an upper bound of seven percent or an analytical uncertainty of two percent.<sup>11</sup> The confidence interval used here is an "exact" interval computed directly from the binomial distribution.

## 7.5 Apparatus.

### 7.5.1 Microscope. A low-power binocular microscope, preferable stereoscopic, is used to examine the bulk sample as received.

- Microscope: binocular, 10-45X
- Light Source: incandescent, fluorescent, halogen or fiber optic
- Forceps, Dissecting Needles, and Probes

- \* Glassine Paper, Clean Glass Plate, or Petri dish
- \* Compound Microscope requirements: A polarized light microscope complete with polarizer, analyzer, port for wave retardation plate, 360° graduated rotating stage, substage condenser, lamp, and lamp iris
- \* Polarized Light Microscope: described above
- \* Objective Lenses: 10X
- \* Dispersion Staining Objective Lens: 10X
- \* Ocular Lens: 10X
- \* Eyepiece Reticule: 25 point or 100 point Chalkley Point Array or cross-hair
- \* Compensator Plate: 550 millimicron retardation
- \* First Order Red I Compensator: 530 nanometers

#### 7.6 Reagents.

Refractive Index Liquids: 1.490 - 1.570, 1.590 - 1.720 in increments of 0.002 or 0.004.

Refractive Index Liquids for Dispersion Staining: High-dispersion series, 1.550, 1.605, 1.630 (optical).

UICC Asbestos Reference Sample Set: Available from UICC MRC Pneumoconiosis Unit, Lisndough Hospital Penarth, Glamorgan CF6 1xw, UK and commercial distributors.

Tremolite-asbestos: Available from J. T. Baker.

Actinolite-asbestos: Available from J. T. Baker.

Chrysotile, Amosite, and Crocidolite is available from the National Institute of Standards and Technology.

Anthrophyllite, Tremolite, Actinolite will be available from the National Institute of Standards and Technology during the first quarter of 1990.

## 8 PROCEDURES

Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are usually friable and may release fibers during handling or matrix reduction steps. All samples and slide preparations should be carried out in a ventilated hood or glove box with continuous airflow (negative pressure) exhausting through an HEPA filter. Handling of samples without these precautions may result in exposure of the analyst and contamination of samples by airborne fibers.

### 8.1 Sample Preparation.

An aliquot of bulk material is removed from the one pint sample container. The aliquot is spread out on a glass slide. A drop of staining solution with appropriate refractive index is added to the aliquot. A cover slide is placed on top of the sample slide.

The first preparation should use the refractive index solution for Chrysotile. If during the identification phase other asbestiforms are suspected to be present in the sample, due to their morphology, then additional analyses shall be performed with the appropriate solutions. Report the percentages of each asbestiform and combine percentages to determine total asbestos concentrations.

### 8.2 Fiber Identification.

Positive identification of asbestos requires the determination of the following optical properties:

- Morphology (3 to 1 minimum aspect ratio)
- Color and pleochroism
- Refractive indices
- Birefringence
- Extinction characteristics
- Sign of elongation

Table 3 lists the above properties for commercial asbestos fibers. Natural variations in the conditions under which deposits of asbestiform minerals are formed will occasionally produce exceptions to the published values and differences from the UICC standards. The sign of elongation is determined by use of the compensator plate and crossed polars. Refractive indices may be determined by the Becke line test. Becke line test or dispersion staining shall be used to identify asbestos fibers. Central stop dispersion staining colors are presented in Table 4. Available high-dispersion (HD) liquids should be used.

### 8.3 Quantification of Asbestos Content.

Asbestos quantification is performed by a point-counting procedure. An ocular reticle (point array) or cross-hair is used to visually superimpose points on the microscope field of view. The point counting rules are as follows:

1. Record the number of points positioned directly above each particle or fiber.
2. Record only one point if two points are positioned over same particle or fiber.
3. Record the number of points positioned on the edge of a particle or fiber.
4. If an asbestos fiber and a matrix particle overlap so that a point is superimposed on their visual intersection, a point is scored for both categories.
5. If a test point lies over an ambiguous structure, no particle or fiber is recorded. Examples of "ambiguous" structures are:
  - a) fibers whose dispersion colors are difficult to see
  - b) structures too small to categorize.
6. A fiber mat or bundle is counted as one fiber.

For the purpose of the method, "asbestos fibers" are defined as mineral fibers having an aspect ratio greater than 3:1 and being positively identified as one of the minerals in Table 3.

A total of 400 points superimposed on either asbestos fibers or nonasbestos matrix material must be counted over at least eight different preparations of representative subsamples. Take eight forceps samples and mount each separately with the appropriate refractive index liquid. The preparation should not be heavily loaded. The sample should be uniformly dispersed to avoid overlapping particles and allow 25 - 50 percent empty area within the fields of view. Count 50 nonempty points on each preparation, using either

a reticle with 100 points (Chalkley Point Array) and counting 25 points in at least two randomly selected fields.

or

a reticle with 25 points (Chalkley Point Array) and counting at least two randomly selected fields.

or

a reticle with a standard cross-hair and counting at least 50 randomly selected fields.

For samples with mixtures of isotropic and anisotropic materials present, viewing the sample with slightly uncrossed polars or the addition of the compensator plate to the polarized light path will allow simultaneous discrimination of both particle types. Quantitation should be performed at 100X. Confirmation of the quantitation result by a second analyst on 10 percent of the analyzed samples should be used as standard quality control procedure. All optical properties in Section 8.2 shall be determined to positively identify asbestos.

#### EXCEPTION I

If the sample is suspected of containing no asbestos a visual technique can be used to report that the sample does not contain asbestos. The rules are as follows:

1. Prepare three slides as described in Section 8.3.
2. View 10 fields per preparation. Identify all fibers.
3. If all fibers are nonasbestos, report no asbestos were found and that visual technique was used.
4. If one fiber is determined to be asbestos, discontinue the visual method and perform the point counting technique as described above.

#### EXCEPTION II

If the sample is suspected to have an asbestos content in excess of ten percent, a visual technique can be used to report that the sample contains greater than ten percent asbestos. The standard operating procedure of the visual technique allowed in the National Institute of Standards and Technology's National Voluntary Laboratory Accreditation Program, Bulk Asbestos Handbook, National Institute of Standards and Technology publication number NISTTR 88-3879 dated October 1988, which is incorporated herein by reference, shall be followed.

## 9 CALCULATIONS

The percent asbestos is calculated as follows:

$$\% \text{ asbestos} = \left( \frac{a}{n} \right) 100\%$$

Where:

- a = number of asbestos counts
- n = number of nonempty points counted (400)
- If a = 0, report "No asbestos detected."
- If a > 0, report the calculated value to the nearest 0.25%

If "no asbestos detected" is reported by the point counting technique, the analyst may report the observation of asbestos fibers in the non-counted portions of the sample.

## 10 ALTERNATIVE METHODS

### 10.1 Alternative Sampling Methods.

Alternative sampling methods may be used as long as they are substantially equivalent to the sampling methods discussed in Section 5 and approved by the Executive Officer of the Air Resources Board. The ARB Executive Officer may require the submittal of test data or other information to demonstrate equivalency.

### 10.2 Analytical Methods.

An alternative analytical method may be used as long as it produces results substantially equivalent to the results produced by the point counting method and approved by the Executive Officer of the Air Resources Board. The ARB Executive Officer may require the submittal of test data or other information to demonstrate equivalency.

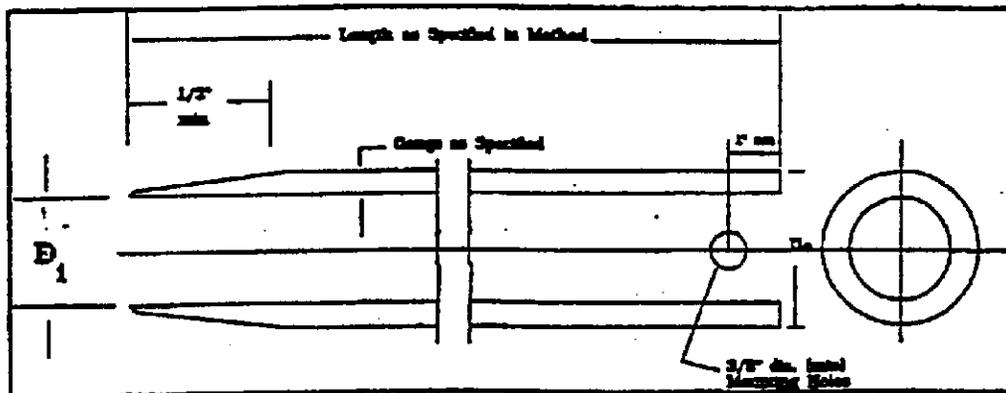
## 11 REFERENCES

- 11.1 G. S. Koch, Jr., R. F. Link. Statistical Analysis of Geological Data. New York. Dover Publications, Inc. December 1985.
- 11.2 Paul F. Kerr. Optical Mineralogy, 4th ed. New York. McGraw-Hill. 1977.
- 11.3 E.M. Chamot and C. W. Mason. Handbook of Chemical Microscopy, Volume One, 3rd ed. New York. John Wiley & Sons. 1958.

- 11.4 F. Chayes. Petrographic Model Analysis: An Elementary statistical Appraisal. New York. John Wiley & Sons. 1958.
- 11.5 E. P. Brantly, Jr., K. W. Gold, I. E. Myers, and D. E. Lentzen. Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method. U. S. Environmental Protection Agency. October 1981.
- 11.6 U. S. Environmental Protection Agency. Asbestos-Containing Materials in School Buildings: A Guidance Document, Parts 1 and 2 EPA/OTS No. C00090m Nareg. 1979.
- 11.7 D. Lucas, T. Harwell, and A. V. Rao. Asbestos Containing Materials in School Buildings: Guidance for Asbestos Analytical Programs, EPA 580/13-80-017a. U. S. Environmental Protection Agency. December 1980.
- 11.8 D. H. Taylor and J. S. Bloom. Hexametaphosphate Pretreatment of Insulation Samples for Identification of Fibrous Constituents. Microscope, 28. 1980.
- 11.9 W. J. Campbell, R. L. Blake, L. L. Brown, E. E. Cather, and J. J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties: Mineralogical Definitions and Identification-Characterization. U. S. Bureau of Mines Information Circular 8751. 1977.
- 11.10 Walter C. McCrone. Asbestos Particle Atlas. Ann Arbor. Ann Arbor Science Publishers. June 1980.
- 11.11 John Moore. Biostatistician. Personnel Communication. February 8, 1990.

Figure 1

Thin Wall Tube for Sampling



Note 1 Minimum of two mounting holes on opposite sides for 2 to 3 inch diameter sampler.

Note 2 Minimum of four mounting holes spaced at  $90^\circ$  for samplers 4 inch diameter and larger.

Note 3 Tube held with hardened screws.

Note 4 Two inch outside-diameter tubes are specified with an 18-gauge wall thickness to comply with area ratio criteria accepted for "undisturbed samples." Users are advised that such tubing is difficult to locate and can be extremely expensive in small quantities. Sixteen-gauge tubes are generally readily available.

Table 1  
Suitable Thin Walled Steel Sample Tube<sup>A</sup>

---

OUTSIDE DIAMETER:			
	2	3	5
inches			
millimeters	50.8	76.2	127
WALL THICKNESS:			
Ewg	18	16	11
inches	0.049	0.065	0.120
millimeters	1.24	1.65	3.05
TUBE LENGTH:			
inches	36	36	54
meters	0.91	0.91	1.45
CLEARANCE RATIO, %	1	1	1

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<sup>A</sup> The three diameters recommended in Table 1 are indicated for purposes of standardization, and are not intended to indicate that sampling tubes of intermediate or larger diameters are not acceptable. Lengths of tubes shown are illustrative. Proper lengths to be determined as suited to field conditions.

**Table 2**  
**Dimensional Tolerances for Thin Walled Tubes**

Nominal Tube Diameters from Table 1 <sup>A</sup> Tolerances, inches			
Size Outside Diameter	2	3	4
Outside Diameter	+0.007 -0.000	+0.010 -0.000	+0.015 -0.000
Inside Diameter	+0.000 -0.007	+0.000 -0.010	+0.000 -0.015
Wall Thickness	+0.007	+0.010	+0.015
Ovality	0.015	0.020	0.030
Straightness	0.030/ft	0.030/ft	0.030/ft

<sup>A</sup> Intermediate or larger diameters should be proportional. Tolerances shown are essentially standard commercial manufacturing tolerances for seamless steel mechanical tubing. Specify only two of the first three tolerances; O. D. and I. D. or O. D. and Wall, or I. D. and Wall.

Figure 2

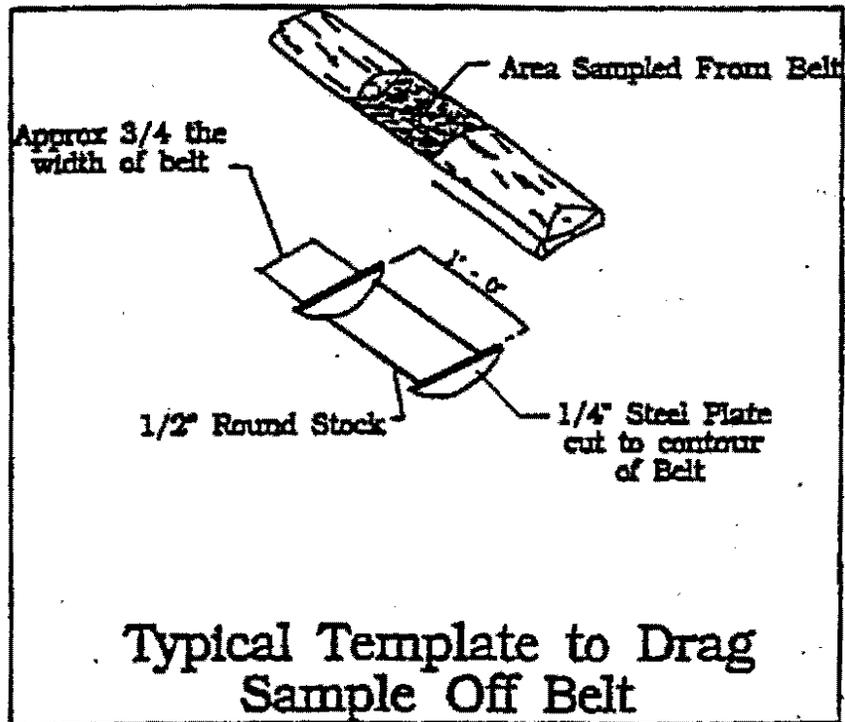


Table 3

## Optical Properties of Asbestos Fibers

Mineral	Morphology <sup>a</sup> , color	Refractive Indices <sup>b</sup>		Birefringence	Extinction	Sign of Elongation
		alpha	gamma			
Chrysotile (asbestiform serpentine)	Wavy fibers. Fiber bundles have splayed ends and "kinks." Aspect ratio typically >10:1. Colorless <sup>c</sup> , nonpleochloric.	1.493 - 1.560	1.517 - 1.562 <sup>f</sup> (normally 1.556)	0.002 - 0.014	to fiber length	+ (length slow)
Amosite (asbestiform grunerite)	Straight, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so. Opaque inclusions may be present.	1.635 - 1.696	1.655 - 1.729 <sup>f</sup> (normally 1.696 - 1.710)	0.020 - 0.33	to fiber length	+ (length slow)
Crocidolite (asbestiform riebeckite)	Straight, rigid fibers. Thick fibers and bundles common, blue to purple-blue in color. Pleochroic. Birefringence is generally masked by blue color.	1.654 - 1.701	1.668 - 1.717 <sup>e</sup> (normally close to 1.700)	0.014 - 0.016	to fiber length	- (length fast)
Anthophyllite-asbestos	Straight fibers and fiber bundles showing splayed ends. Colorless to light brown. pleochroic absent.	1.596 - 1.652	1.615 - 1.676 <sup>f</sup>	0.019 - 0.024	to fiber length	+ (length slow)
Tremolite-actinolite-asbestos	Straight and curved fibers <sup>d</sup> and fiber bundles. Large bundles show splayed ends. Tremolite is colorless and actinolite is green. Weakly to moderately pleochroic.	1.599 - 1.668	1.622 - 1.688 <sup>f</sup>	0.023 - 0.020	to fiber length	+ (length slow)

<sup>a</sup> From Reference 6; colors cited are seen by observation with plane polarized light.

<sup>b</sup> From Reference 7 and 9.

<sup>c</sup> Fibers subjected to heating may be brownish.

<sup>d</sup> Fibers defined as having aspect ratio >3:1.

<sup>e</sup> ⊥ to fiber length.

<sup>f</sup> || to fiber length.