資料１

解体現場、大気濃度調査等に関する現状と課題等の情報収集に関する質問・回答

【第２回専門委員会　(株)ＥＦＡラボラトリーズへの質問】

○　TEM 一日納期について、フィルターにサンプリングした後、３時間以内で分析することができるとの説明であったが、確認する石綿の長さ及び幅の範囲により分析時間は異なる。マニュアルではどのようになっているのか。

○　ニューヨーク州 Department of Health の ELAP (Environmental Laboratory Approval Program) と言われている分析機関認定プログラムに 25 社の大気 TEM 分析機関が登録されているとあるが、各機関は電子顕微鏡を何台持っているのか。1 サンプルに対して 1 日納期が達成されるというただし書きがつくのか。

(回答)

上記質問で 25 社が TEM 分析機関とおっしゃっているのは米国ニューヨーク州の ELAP のみの登録機関数です。前回の私の回答には 2012 年 7 月 24 日現在の米国全体の NVLAP の TEM 分析機関の登録数も 79 社と紹介しております。NVLAP の 10 月 17 日改定版では現在の米国の TEM 分析機関は 80 社となっています。以下の Web サイト http://ts.nist.gov/standards/scopes/temtm.htm をご確認ください。

米国のフォレンジック・アナリティカル・ラボラトリーズの TEM アナリストの M. Floyd 氏からヒアリングした現在の米国エアモニタリングの実情を述べ、1 日の分析キャパシティを述べます。次に 10 月 15～17 日のアムステルダムの ISO アスベスト分析会議に出席していたドイツの Dr. R. König 及びフランスの Dr. M. Misseri からヒアリングしたエアモニタリング分析キャパシティを述べます。最後に AHERA の Appendix A to Subpart E of Part 763 の *II. Mandatory Transmission Electron Microscopy Method* に示された試料採取、試料準備、TEM 分析、作業手順、報告書作成、品質管理保証手順を添付します。

【現在の米国エアモニタリングの実情】Floyd 氏よりヒアリング

ほとんどの米国ラボでは AHERA の 13 試料中、5 つの内側試料のみを先ず分析するように準備します。エアモニタリングの発注者は、外側試料やブランク試料の分析にお金を使うより、不合格の場合の再クリーニング作業や再クリーニング後の新たな 13 試料の分析にお金を使おうと考えるからです。そして事実上

全てのコンサルタントは MCE フィルターに AHERA のエアモニタリング試料を採取します。MCE フィルターの方が PC フィルターより TEM 分析のグリッド観察試料の作製が早いからです。

経験を積んだ TEM 分析機関では、40 サンプルのバッチ（例えば 8 セットの内側 5 試料）の TEM グリッド観察試料を、3～4 時間で作成します。経験を積んだ TEM 分析者はきれいな試料であれば 1 時間程で 5 試料分析できます。この計算でいくと TEM 一台当たり 12 時間で 40 サンプルの分析ができることになります。8 時間交代で 24 時間運営しているラボは、24 時間で 80 サンプル分析ができることになります。さらに TEM グリッド観察試料を作成する専属のスタッフと TEM 分析だけを行う 8 時間交代 3 人の専属スタッフが並行して作業すれば、理論上 24 時間で 120 サンプルができることになります。実際は、時々おこる TEM の修理・補修、キャリブレーション作業、ワンバッチ 10-20％の品質管理保証（QA/QC）分析等が入るので、24 時間 3 交代制の場合、最大 60 から 70 サンプルの容量と考えた方が良いと思います。

しかし、ほとんどの米国の登録 TEM 分析機関は、通常の営業時間の生産性を上げるために、及び TEM の故障で修理に出す時のバックアップのために、2 台以上の TEM を所有しています。現在たくさんのサンプルをもらう事は少なくなりましたが、今でも夏の繁忙期には、1 日当たり 40～60 サンプル TEM 分析がルーチンになっています。

ラッシュ分析については、5 試料のグリッド観察試料を 2 時間で準備し、1 時間の TEM 観察で 3 時間の納期になる訳ですが、試料が汚れていて分析時間が長引く可能性や、グリッド試料作製時の問題対応等を配慮して、通常最短納期を 4 時間にしてラッシュ分析を実施するようにしています。

もし TEM 分析機関が目開き面積 0.013mm^2 のグリッドを使用した場合、1200～1800L の採取試料は 5 目開き、1485～1800 L の採取試料は 4 目開きの分析で分析が完了します。TEM 分析者は、最初にグリッドの状態が分析に適しているかの評価（開いていないグリッド、適切な負荷量、フィルターの溶解度チェック等）を行います。この評価に問題なければ、ランダムにグリッドを選んで、左上の角から右上の角の方向に動かして、反対側に行ったら TEM の観察視野の直径 4 分の 3 ドロップさせ、右側から左方向に動かして観察します。この方法で目開き全体が観察されるまで同じ作業を繰り返します。

アスベスト繊維らしきものが見つかった場合、AHERA の形態定義（長さ 0.5μm 以上、アスペクト比 5:1 以上）を確認します(1)。そして AHERA のストラクチャーの定義（繊維、繊維束、クラスター、またはマトリックス）で分類します。次に、SAED 分析をこれらの確認されたストラクチャーについて行います。SAED パターンは写真で保存し、測定を行います。SAED パターンがアスベスト鉱物と

合致したら、EDX 分析を行います。特にナトリウム、マグネシウム、ケイ素、カルシウム、鉄のアスベスト鉱物組成をアスベストの種類の同定に使います。アルミニウム、カリウム、またはマンガンのピークがケイ素のピークの 5%より多く存在する場合はアスベストでないと見なします。繊維の形態、SAED パターン、主要元素組成比の 3 つが、規制されているアスベストの特定に必要です。

アスベストが特定されると大きさ、SAED と EDX の分析結果が集計用紙に記録されます。分析者は次のグリッド目開きに移って、終わるまで分析を繰り返します。TEM 分析者は、もし 50 本目のアスベストストラクチャーをカウントした目開きをカウントし終わったら分析をストップします。または分析感度が 0.005 ストラクチャー/cc 以下になったら（通常 4～5 目開き）分析は完了します。

セットサンプルの全試料が分析されたら、集計データは報告書にされ、品質チェックを受けて、お客様に送られます。多くの分析機関が集計シートのコンピュータ化をしており、試料が分析されると自動的に分析結果が出てくるようになっています。

(1)亀元注釈

AHERA のクリアランス・サンプリングは、採取前に作業エリアの表面の拭取りを徹底してから行うので、試料自体はきれいなサンプルで、ほとんど繊維状粒子は確認されません。1 目開きに 1 ストラクチャーも確認されない場合が多い。

AHERA の TEM 分析の規定では fiber の定義を「長さ：5μ以上、アスペクト比：5 対 1 以上」としており、繊維径の規定はありません。グリッドのスキャン分析は倍率：15,000～20,000 倍で行うように規定されています。分析者は長さが 0.5μｍ以上の繊維状粒子をみつけると蛍光板上でアスペクト比を確認し、繊維の長さと径、アスペクト比を記録します。TEM 分析をルーチンワークとしている分析者によると繊維径が最低 0.05～0.025μｍ程度まで確認出来るとのこと。

【解体除去現場のクリアランス・サンプリングの分析キャパシティ】

König 博士（APC）：一台の TEM で、きれいなサンプルなら、一人一日で 8～12 試料程度と思います。

Misseri 博士（ユーロフィン・フランス）：米国のクリアランス・サンプリングと似ています。11 台 TEM を持っていますが、建材分析とも併用しており、エアモニタリング分析の 1 日のキャパシティは 100 試料と考えています。

添付資料

Appendix A to Subpart E of Part 763 の *II. Mandatory Transmission Electron Microscopy Method*

construction document for the building, or, to the best of his or her knowledge, no ACBM was used as a building material in the building. The local education agency shall submit a copy of the signed statement of the architect, project engineer, or accredited inspector to the EPA Regional Office and shall include the statement in the management plan for that school.

(b) The exclusion, under paragraphs (a) (1) through (4) of this section, from conducting the inspection under §763.85(a) shall apply only to homogeneous or sampling areas of a school building that were inspected and sampled before October 17, 1987. The local education agency shall conduct an inspection under §763.85(a) of all areas inspected before October 17, 1987, that were not sampled or were not assumed to be ACBM.

(c) If ACBM is subsequently found in a homogeneous or sampling area of a local education agency that had been identified as receiving an exclusion by 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 OF PART 763—INTERIM TRANSMISSION ELECTRON MICROSCOPY ANALYTICAL METHODS—MANDATORY AND NON-MANDATORY—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 items 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/cm³.

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 3:1.

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 flask which on subsequent analysis has an average of less than 10 structures/cm² in an area of 0.01 cm² (nominally 2×200-mesh grid openings) and a maximum of 50 structures/cm² 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 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 3:1 or greater.

12. *Laboratory sample coordinator*—That person responsible for the conduct of sample

handling and the certification of the testing procedures.

13. *Fiber 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/mm².

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. *NAD*—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. *S/cm²*—Structures per cubic centimeter.

23. *S/mm²*—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.

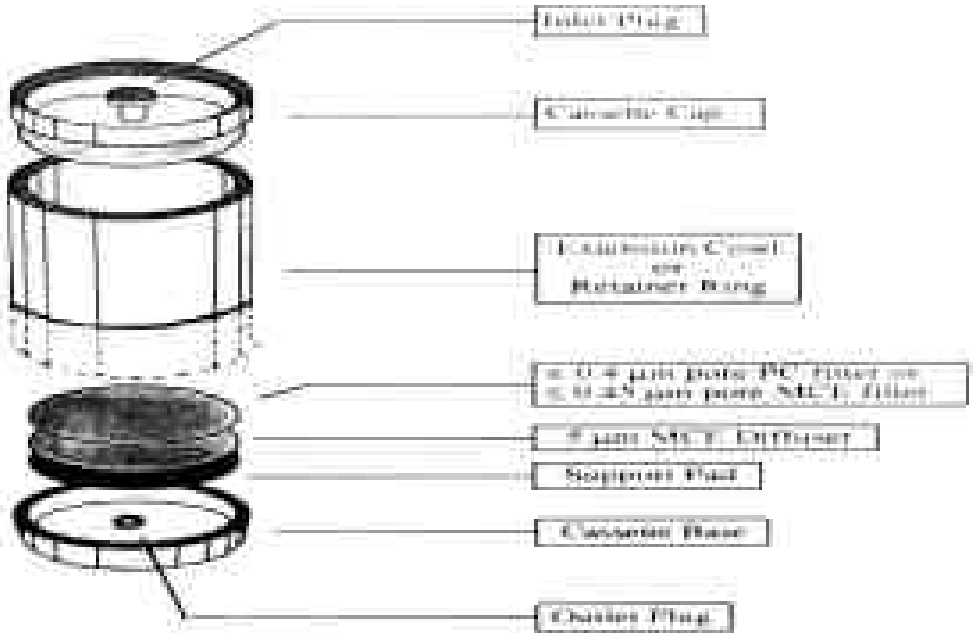
2. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (for purposes 1, 2, 3, and 5 of this §2.2.).

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

4. Prescreen the loaded cassette collection filters to ensure that they do not contain concentrations of asbestos which may interfere with the analysis of the sample. A filter blank average of less than 70 s/mm² in an area of 0.007 mm² (normally 10 200-mesh grid openings) and a single preparation with a maximum of 50 s/mm² 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:



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 representative 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 10 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:

1. Near the entrance to each abatement area.

2. 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 1 specifies volume ranges to be used:

TABLE 2--NUMBER OF 200 MESH OR OHIO OPENINGS (0.0021 MM^2) THAT NEED TO BE ANALYZED TO MAINTAIN SENSITIVITY OF 0.025 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
500	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	2,600	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

Recommended Volume Range

Recommended Volume Range

Note minimum volumes required:
25 mm : 500 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

38. Ensure that the sampler is stored upright before interrupting the pump flow.

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

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

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

42. 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 rest of the method.

Unit Operation	QC Check	Frequency	Performance Expectation
Sampling materials	Sealed blank	1 per 100 sets	95%
Sample preparation	Field blanks	2 per 100 sets	95%
	Pump calibration	Before and after each field series	95%
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample shipment	Review of sealing 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 10 s/mm² in an area of 0.007 mm² (nominally 10-100-mesh grid openings) and a single preparation with a maximum of 40 s/mm² 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 measurements 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 rod 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 200X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscope utilizing manual or automated procedures may be used provided 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 fibers, 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 particular ashing and operating conditions will then be set such that a 1–2 μm (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.

7. TEM Method

1. An 80–120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated graduations 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 of 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 sputtered 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 instrument 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, 200–1,000X, then 10,000–20,000 screen magnifications 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:*

a. Less than 80 percent of the grid openings covered by the replica are intact.

b. The replica is doubled or folded.

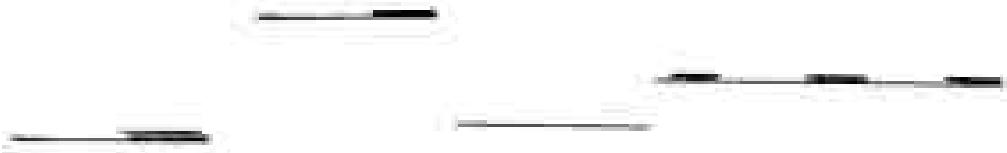
c. The replica is too dark because of incomplete dissolution of the filter.

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 μm 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 detectable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. An intersection is a nonparallel touching or crossing of fibers, with the projections having an aspect ratio of 5:1 or greater. See the following Figure 2:

FIGURE 2--COUNTING GUIDELINES USED IN DETERMINING ASBESTOS STRUCTURES

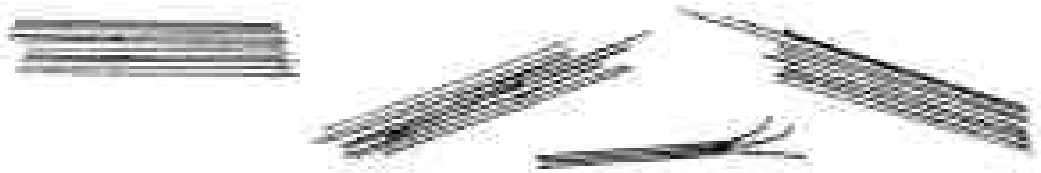
Count as 1 fiber; 1 structure; no intersections.



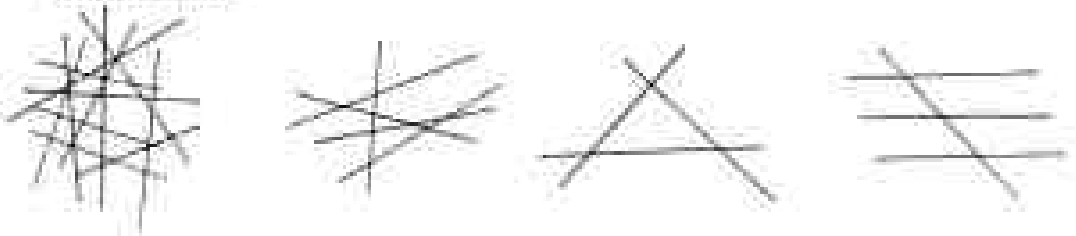
Count as 2 fibers if space between fibers is greater than width of 1 fiber diameter or number of intersections is equal to or less than 1.

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.

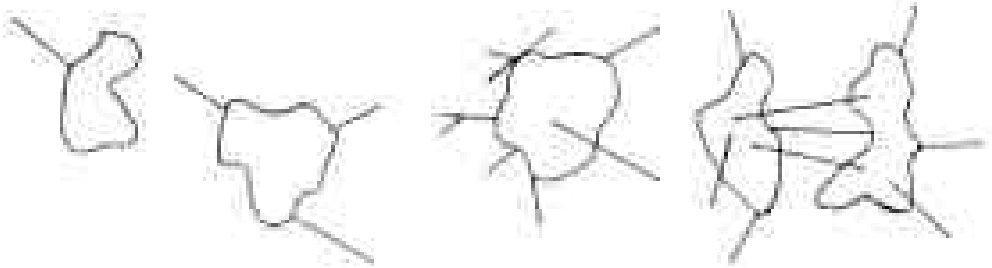
Count bundles as 1 structure; 3 or more parallel fibrils less than 1 fiber diameter separation.



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
<3:1 Aspect Ratio

1. *Fiber.* A structure having a minimum length greater than or equal to 0.5 μm and an aspect ratio (length to width) of 3:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

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

111. *Cluster.* A structure with fibers in a random arrangement such that all fibers are intertwined 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 μm and for fibers equal to or greater than 5 μm 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/mm² 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/mm² 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/mm² 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/mm² 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/mm² 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 file number on the count sheet.

10. Classification Rules.

a. *Fiber.* A structure having a minimum length greater than or equal to 0.5 μm and an aspect ratio (length to width) of 3:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dumbbelled.

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 interrupted 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 returned 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 qualitative 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 contaminant 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 samples 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 Requirement
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/200 grids/lot of 1000 or 1 opening/sample	100%
	Special clean area monitoring	After cleaning or service	Meet specs or retest
	Laboratory blank	1 per preparation or 10%	Meet specs or flag/re-prep
	Pharmacist blank	1 per 20 samples	75%
	Multiple preps (3 per sample)	Each sample	Use with error of 15% coefficient grid opening
Sample analysis	System check	Each day	Each day
	Alignment check	Each day	Each day
	Magnification calibration with low and high standards	Each month or after service	95%
	EDS calibration by grid standard	Weekly	95%
	EDS calibration by copper line	Daily	95%
Performance check	Laboratory blank (measure of cleanliness)	Prep 1 per service or 10%/ Anal 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	99%
	Repeat and verify ID electron diffraction pattern of structure	1 per 5 samples	90% accuracy
Calculations and data reduction	Hand calculation of automated data reduction procedure or independent recalculation of hand-calculated data	1 per 100 samples	95%

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/cm² per 10,000 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.I.

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

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.I.

11. Record an electron diffraction pattern of one asbestos structure (unit) 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 certified 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." USEPA 560/5-85-024, June 1985.

2. "Measuring Airborne Asbestos Following an Abatement Action." USEPA, Office of Pollution Prevention and Toxics, EPA 560/5-85-018, 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. Burdett, 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., ITE Assoc., "Standard Operating Procedure for Determination of Airborne Asbestos Fibres by Transmission Electron Microscopy Using Polycarbonate Membrane Filters." WEHL SOP #2-1, March 1, 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, 1988.

III. Nonmandatory Transmission Electron Microscopy Method

A. Definitions of Terms

1. *Analytical sensitivity*—Airborne asbestos concentrations 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 s/cm³.

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 3:1.

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 10 structures/cm² in an area of 0.007 cm² cumulatively (5,200 mesh grid openings) and a maximum of 20 structures/cm² 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 intertwined 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 3:1 or greater and having substantially parallel sides.

【第３回専門委員会
　一般社団法人日本環境測定分析協会、社団法人日本作業環境測定協会への質問】
○　分析の発注者の比率は施主、受注を受けた工事業者、石綿除去業者でどのようになっているのかできたら調べてほしい。

（回答）

アスベスト受注依頼先に関するアンケート調査結果

一般社団法人 日本環境測定分析協会
社団法人日本作業環境測定協会　精度管理センター

1　調査対象

1）日本環境測定分析協会（日環協）
[illegible] 212 社（事業所）[illegible]

2）日本作業環境測定協会（日測協）
[illegible]

調査対象 計　230 社（事業所）

2　回答数

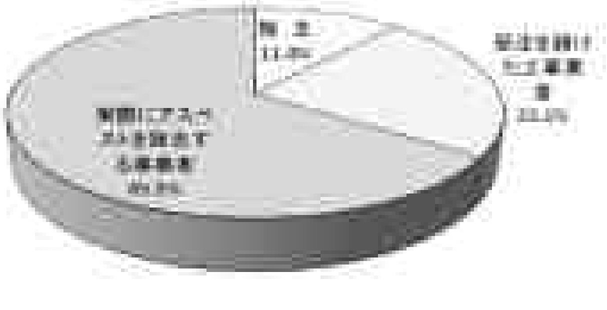
	日環協		日測協		合計	
	回答数	回答率(%)	回答数	回答率(%)	回答数	回答率(%)
全回答数	117	55.2	13	76.5	130	56.5
有効回答数	87	41.0	10	58.8	97	42.2

3　アンケート結果

石綿含有建材等除去工事におけるアスベスト濃度測定の受注依頼先

受注先	日環協		日測協		合計	
	件数	割合(%)	件数	割合(%)	件数	割合(%)
① 施主	489	13.8	96	6.0	585	11.4
② 発注を請けた工事業者	968	27.4	221	13.8	1189	23.1
③ 実際にアスベストを除去する業者	2081	58.8	1283	80.2	3364	65.5

石綿含有建材等除去工事におけるアスベスト濃度測定の受注依頼先（合算）



【第４回専門委員会　村山氏への質問】

○　オランダの報告例に関し、TEM による濃度測定の際の対象とする石綿のサイズ（特に幅）について

（回答）

オランダの報告書では、RIVM（The National Institute for Public Health and the Environment）がまとめた以下の報告の中で、TNO（Netherlands Organisation for Applied Scientific Research）が行った環境中のアスベスト濃度の測定結果を引用しています。その中で、測定対象として長さ５μｍ以上で、長さと繊維径の比が少なくとも３：１であることが示されていますが、それ以外の情報は報告書には記載されていません。他の箇所にも、お問い合わせいただいた点に関する情報の記載はありませんでした。