CHAPTER 2

Asbestos Analysis Methods

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CONTENTS

2.1 INTRODUCTION

The value of a standard method is that it defines procedures in such a way that different laboratories working independently will achieve similar results when using the same method. There are more than 30 different "standard" methods available for the analysis of asbestos in a variety of media. The methods include those for determining the amount of asbestos in air, water, bulk building materials, surface dust, carpet, soil, and specific product materials such as vermiculite and talc. Some methods, although in draft or interim forms, have become generally recognized and used as standard methods by the analytical community. Governmental agencies, such as the Occupational Safety and Health Administration (OSHA), the National Institute of Occupational Safety and Health

(NIOSH), the U.S. Environmental Protection Agency (EPA), the California Air Resources Board (CARB), and the New York State Department of Health, have promulgated some of the methods. Consensus standards groups such as the American Society for Testing and Materials (ASTM), the International Standards Organization (ISO), and the American Water Works Association (AWWA) have published other methods. A number of methods have gained acceptance after being published in the scientific literature. Which method to use in a particular situation depends on the media to be tested and level of information required.

Because the concern with asbestos is related to its fibrous nature, microscopy is the chief analytical tool used for its analysis. Different microscopes have advantages and disadvantages with regard to cost and the ability to provide information about asbestos fibers. Polarized light microscopy (PLM) is the standard way to analyze for asbestos in bulk materials. Phase-contrast microscopy (PCM) is the instrumental technique used for many occupational air sample analyses. Transmission electron microscopy (TEM) and, in some cases, scanning electron microscopy (SEM) are used for all types of samples when small fibers are involved or specific identification of individual asbestos fibers is desired.

2.2 SAMPLE COLLECTION

The collection of samples for analysis depends on the media to be tested and the specific procedures for sample collection are usually provided in the particular analysis method. In general, air samples are collected on membrane filters, water samples in glass or plastic bottles, surface dust by microvacuum or wipe samplers, and solid materials such as building materials, soil and specific products in plastic bags or rigid plastic containers. Air samples are collected on either mixed cellulose ester (MCE) or polycarbonate (PC) filters using either 37- or 25-mm air cassettes. To be quantitative, air samples must be collected with a measured amount of air volume, and surface dust samples must be collected from measured areas of a surface.

2.3 POLARIZED LIGHT MICROSCOPY

A PLM (Figure 2.1) is a compound light microscope that contains a piece of polarizing material in the light path below the sample and another in the light path above the sample. The "PLM method" uses a stereo light microscope (Figure 2.2) to help in taking apart a bulk sample and a polarizing light microscope to identify the fibers among the binders and fillers. Work in the 1980s by McCrone[1,2] established the procedures for asbestos fiber identification by PLM. The PLM identification of asbestos fibers depends on several optical crystallographic properties: refractive indices, dispersion staining, birefringence, sign of elongation, and extinction angle.

The *refractive index* of a substance is numerically equal to the ratio of the velocity of light in a vacuum to its velocity in a substance.[1] The velocity (of light) in any given substance depends on composition; in general, the higher the atomic number of the atoms involved, the lower the velocity and the higher the index.[1] *Dispersion staining* produces its color, not by any chemical interaction but by virtue of the difference between the dispersion of refractive index for a particle and the liquid medium in which the particle is immersed.[1] *Birefringence* refers to the difference between the two refractive indices at right angles to the axis of the microscope.[2] Elongated particles are said to have a positive *sign of elongation* when they have a greater refractive index in the parallel direction than in the perpendicular direction.[1] *Extinction* refers to the behavior on rotation of the microscope stage when a crystalline substance is observed between crossed polarizing sheets. Each particle will show alternate brightness (polarization colors) and darkness (extinction). The particle shows parallel extinction when a prominent direction, for example, length of a fiber, is oriented parallel to the polarizer or analyzer vibration direction in its darkness position.[2]

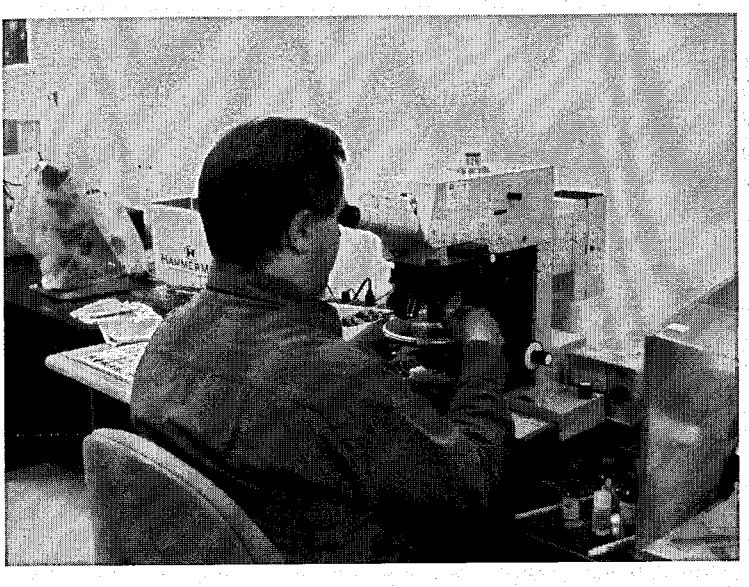

Figure 2.1 Analyst using a PLM for asbestos analysis.

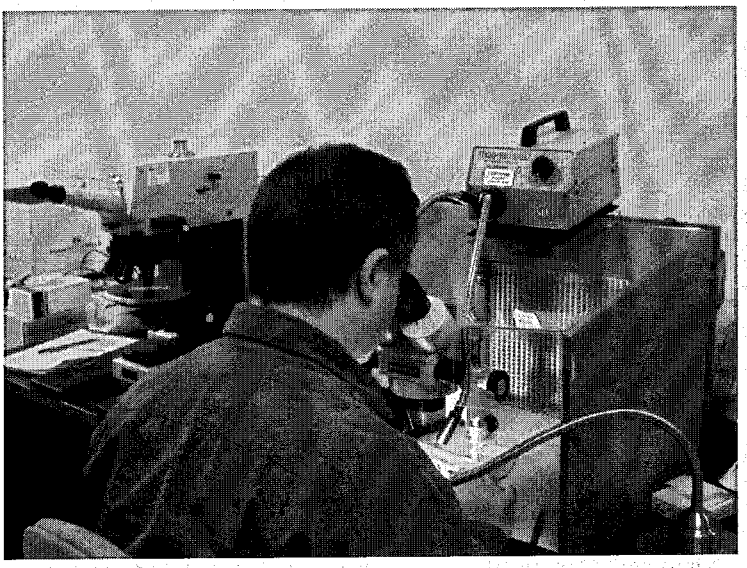

Figure 2.2 Analyst using a stereo-binocular microscope in a HEPA-filtered hood to examine a bulk sample for asbestos.

Because of the size of the wavelength of light, PLM methods of identification are limited to fibers approximately 1 μm in diameter (Figure 2.3).

2.4 BULK ASBESTOS METHODS

The U.S. EPA has defined asbestos-containing material as any material or product that contains more than 1% asbestos.[3,4] The bulk analysis procedure most often specified is the "Method

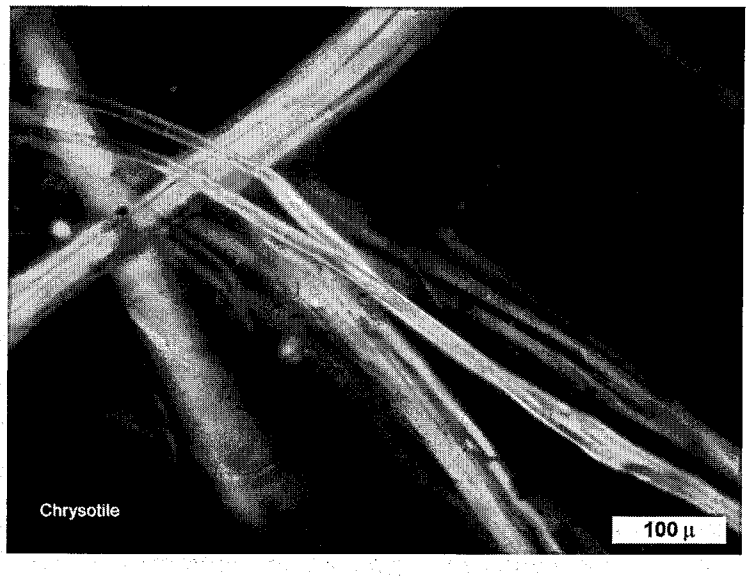

Figure 2.3 Image of asbestos as seen with a PLM.

for the Determination of Asbestos in Bulk Building Materials (EPA-600/R-93/116)" published in 1993.[5] Although it is generally accepted as an improvement over the U.S. EPA "Interim Method for the Determination of Asbestos in Bulk Insulation Samples (EPA-600/M4-82-020)" published in December 1982,[6] the 1993 method has never been formally adopted by the EPA. The NIOSH 9002 and the OSHA ID-191 methods involve similar procedures as the 1993 EPA bulk method.[7,8]

Bulk asbestos analysis performed by PLM methods involves identifying the type of asbestos present on the basis of optical properties and then estimating the relative amount of asbestos in relation to the rest of the bulk sample. The estimates are given in terms of volume percents or, in some cases, area percents. PLM analysts practice with samples of known asbestos percentages until they can visually estimate the values on a consistent basis. The PLM visually estimated asbestos percent values do not necessarily correspond to the weight percent of asbestos in a product. When all components of a bulk material have similar densities, the volume percent value is expected to be similar to the weight percent value. However, if the sample contains 12% chrysotile asbestos by weight in a binder of a denser material such as calcium carbonate (limestone), then the PLM analytical result may show 30% to 40% asbestos by volume. Similarly, if a sample contains 45% to 50% chrysotile asbestos by weight in a material that contains the same weight of a lighter component such as cellulose (paper fibers), then the PLM analytical result may show 5% to 10% asbestos by volume. In most asbestos-containing materials, the precise determination of the percent of asbestos by weight is not of great importance because once a material is shown to contain more than 1% asbestos, it is considered a regulated asbestos-containing building material. In most building products such as insulation, fireproofing, acoustical plasters, and pipe covering where asbestos was intentionally added, the amount of asbestos present is significantly more than 1%.

In some materials such as some ceiling tiles, floor tiles, caulks, paints, and joint compounds, the amount of asbestos may have been added in the low range (approximately 1%). For these materials, special procedures should be used. One special procedure is called "point counting."[9] In this procedure, the particles of the sample material are dispersed on a microscope slide and 400 nonempty points on the slide randomly selected for examination. If, on one of the points, an asbestos fiber happens to line up with the center of the microscope eyepiece crosshairs, the fiber is counted. Percentage of asbestos is calculated on the basis of the number of positive "hits" during the count. Counting three

asbestos fibers out of 400 nonempty points, for instance, corresponds to an asbestos percent of 0.75%. A stratified point-counting method is available as a method in the Certification Manual of the New York State Department of Health Environmental Laboratory Approval Program (ELAP).[10,11] The item states: "For samples containing high amounts of asbestos, the stratified point-count technique invokes labor-saving semi-quantitative counting rules. The stratified method is based on the premise that accurate quantitation is unnecessary for materials that contain substantial amounts of asbestos. In contrast, extensive analytical effort is still required for samples that contain positive but small amounts of asbestos."[10] Although more quantitative, the point-count technique has been criticized as not being statistically valid at the 1% level.[12] For a sample in which a value of exactly 1% was determined by the 400 point-count procedure, repeated point-count analyses would be expected to fall variously within the range of 0.27% to 2.6% asbestos on the basis of Poisson statistics. To provide a more statistically valid analysis when low levels of asbestos may be present, matrix reduction is used to concentrate the asbestos fibers. When possible, combustible material is ashed away, acid-soluble material is dissolved away, and density separation is used to prepare the sample of bulk material so that low levels of asbestos fibers can be readily found. Electron microscopy can also be used to help provide quantitative values for low levels of asbestos. The EPA 1993 bulk method, the NIOSH 9002, the OSHA ID-191, and the ELAP Item 198.4 all contain some discussion of matrix reduction and use of electron microscopy.[13] A bulk microscopy method that incorporates various forms of matrix reduction for particular sample product types and use of electron microscopy is being drafted concurrently by task groups in both ASTM and ISO. A comparison of several of the bulk methods is shown in Table 2.1.

Table 2.1 Comparison of Common Methods for Measuring Asbestos in Bulk Building Materials

	EPA-600/ M4-82-020 1982	EPA-600/ R-93/116 1993	NIOSH 9002	OSHA ID-191	ASTM and ISO Bulk in Progress
Instrument	Stereo + PLM XRD	Stereo + PLM and TEM	Stereo + PLM	Stereo + PLM with mention of SEM and TEM	PLM and TEM
Sample preparation	As is and some matrix reduction	As is and some matrix reduction, gravimetric	As is and some matrix reduction	As is and organic and carbonate matrix reduction	As is and detailed matrix reduction, gravimetric
Magnification	×1–1000	×1–20,000	×10–400	–	×1–20,000
Minimum fiber diameter	Approximately >1 μm	Approximately >1 μm	Approximately >1 μm	Approximately >1 μm	Approximately >1 μm
AR	NA	Generally >10:1	NA	3:1 with mention of 100:1	Not known at this time
Measurement	Volume or areal estimation	Visual estimation	Areal estimation	Areal estimation	Volume estimation + weight measure
Identification	Refractive indices, dispersion staining, birefringence, sign of elongation, and extinction angle	Refractive indices, dispersion staining, birefringence, sign of elongation, and extinction angle	Refractive indices, dispersion staining, birefringence, sign of elongation, and extinction angle	Refractive indices, dispersion staining, birefringence, sign of elongation, and extinction angle; mention of SEM and TEM	Refractive indices, dispersion staining, birefringence, sign of elongation, and extinction angle + TEMID
Reporting	% asbestos	% asbestos and possible weight percent	% asbestos	% asbestos	Volume or areal and percent asbestos or weight percent

AR, aspect ratio.

2.5 PCM: AIR ANALYSIS

The PCM (Figure 2.4) is a compound light microscope that illuminates a specimen with a hollow cone of light. The cone of light is narrow and enters the field of view of the objective lens. Within the objective lens is a ring-shaped device that introduces a phase shift of a quarter of a wavelength of light. This illumination causes minute variations of refractive index in a transparent specimen to become visible. The phase-contrast mode pushes the ability of the light microscope to see fibers as thin as 0.25 μm in diameter, but it does so at the expense of identification. PCM is not used to identify asbestos fibers.

The most commonly used PCM method, NIOSH 7400, requires a positive PCM (dark) with green or blue filter, an adjustable field iris, ×8 to 10 eyepieces, and a ×40 to 45 phase objective (total magnification is approximately ×400).[14] Most PCM analysts use binocular PCMs. Within one of the eyepieces there is a Walton–Beckett-type graticule, which forms an analysis area of approximately 0.00785 mm^2 at the specimen plane. The other U.S. Government–promulgated PCM method, OSHA ID-160, has similar requirements.[15] Under the PCM methods, fibers are counted when they are greater than 5 μm in length and have an aspect ratio (AR) (length to width) of at least 3:1. The NIOSH 7400 method "A" counting rules used for counting asbestos fibers have no upper limit on the diameter of the fiber counted. A fiber that appears to be partially obscured by a particle is counted as one fiber. If the fiber ends emanating from a particle do not seem to be from the same fiber and each end meets the length and AR criteria, they are counted as separate fibers. Results of the PCM methods are given in terms of fibers per cubic centimeter of air. According to the current regulations (as of June 2009) promulgated by OSHA, an employer shall ensure that no employee is exposed to an airborne concentration of asbestos in excess of 0.1 fiber per cubic centimeter of air as an 8-h time-weighted average as determined by the OSHA ID-160 method or by an equivalent method (e.g., NIOSH 7400).[16] OSHA has also established a short-term excursion limit. Under this rule, an employer shall ensure that no employee is exposed to an airborne concentration of asbestos in excess of 1.0 fiber per cubic centimeter of air (1 f/cc) as averaged over a sampling period of 30 min as determined by the OSHA ID-160 method or by an equivalent method.[17] The only method considered to be an equivalent method by OSHA at this time is the NIOSH 7400 method

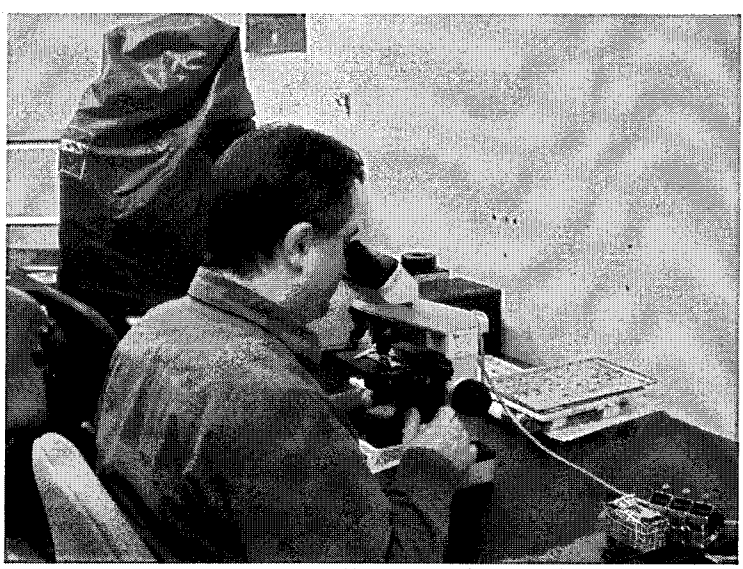

Figure 2.4 Analyst using a PCM for asbestos analysis.

(with NIOSH 7402 in some cases). To be considered by OSHA to be equivalent, a method must pass the equivalency test found in 29CFR1910 (General Industry Asbestos Standard).

2.6 TRANSMISSION ELECTRON MICROSCOPY

The TEM (Figure 2.5) uses electromagnetic coils as lenses to form magnified images with an electron beam in the same way that a light microscope uses glass lenses and a light beam to form images. Electrons can be accelerated with high potential energies, which produce a beam with a very small wavelength and thus allow much higher magnifications than can be achieved with the wavelengths of light. The commonly used TEM methods call for a TEM that can operate at an accelerating potential of 80,000 (80 kV) to 120,000 V. If operating properly at 80 to 120 kV, a TEM is easily capable of obtaining a direct screen magnification of approximately ×100,000 with a resolution better than 10 nm. This allows the smallest asbestos fibers, which are approximately 20 nm (0.02 μm) in diameter, to be examined. In addition to the analysis of fiber morphology by TEM (Figure 2.6), selected area electron diffraction (SAED) and x-ray energy dispersive spectroscopy (EDS) can be used to gain information about a particle's crystal structure and elemental composition. TEM with SAED and EDS is referred to as analytical electron microscopy. Examples of a chrysotile SAED pattern and EDS spectra from reference asbestos minerals are shown in Figures 2.7 and 2.8.

The NIOSH 7402 method is the complementary TEM method for the PCM 7400 method.[18] With 7402, fibers greater than 5 μm in length and having an AR (length to width) of at least 3:1 and a width of at least 0.25 μm are characterized by SAED and EDS. These fibers are then classified as nonasbestos or asbestos. The type of asbestos is also determined. A value of percent asbestos is determined and this percentage applied to PCM results of the same sample. No concentration of fibers per cubic centimeter is reported under Method 7402. The ASTM method for the PCM analysis of workplace exposures, D4240, has been removed from official ASTM practice, and a new method with the inclusion of some TEM procedures for the identification of the fibers found in mines and quarries has been published.[19] The ASTM Method, D7200-06: Standard Practice for Sampling and Counting Airborne Fibers, Including Asbestos Fibers, in Mines and Quarries, by PCM and TEM,

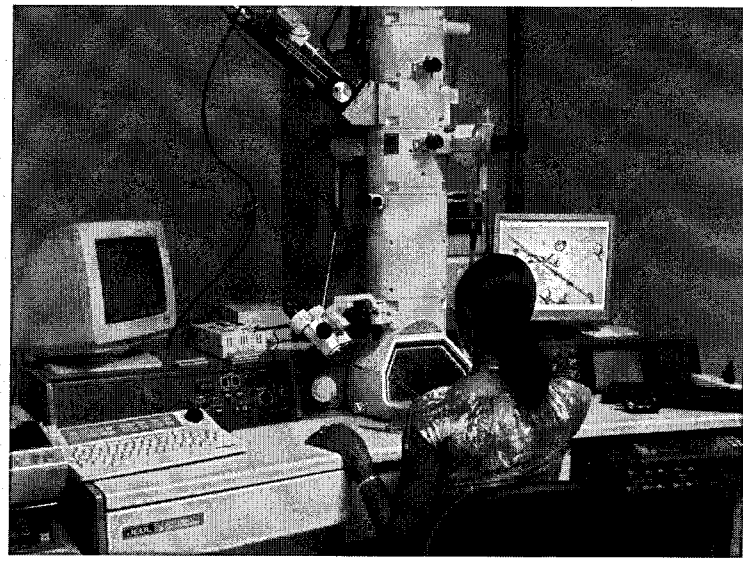

Figure 2.5 Analyst using a TEM for asbestos analysis.

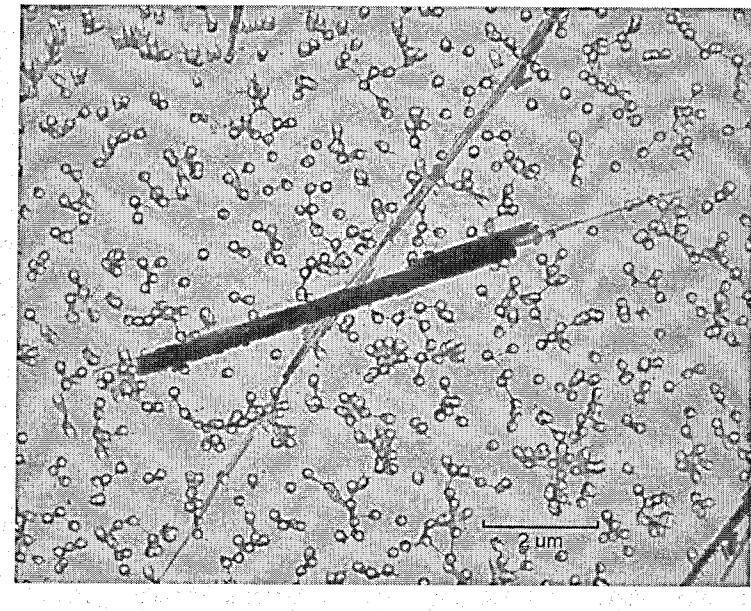

Figure 2.6 Image of crocidolite and chrysotile asbestos fibers as seen with a TEM. Crocidolite is the thicker fiber; chrysotile is longer and thin. The circles are carbon replicas of the PC filter.

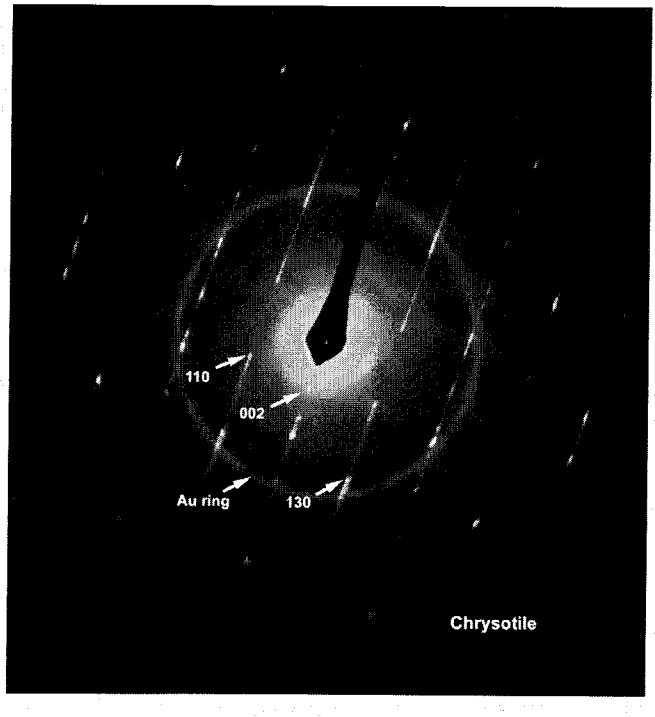

Figure 2.7 Chrysotile SAED pattern. The gold ring results from coating the fiber with a thin layer of gold and is used for calibration.

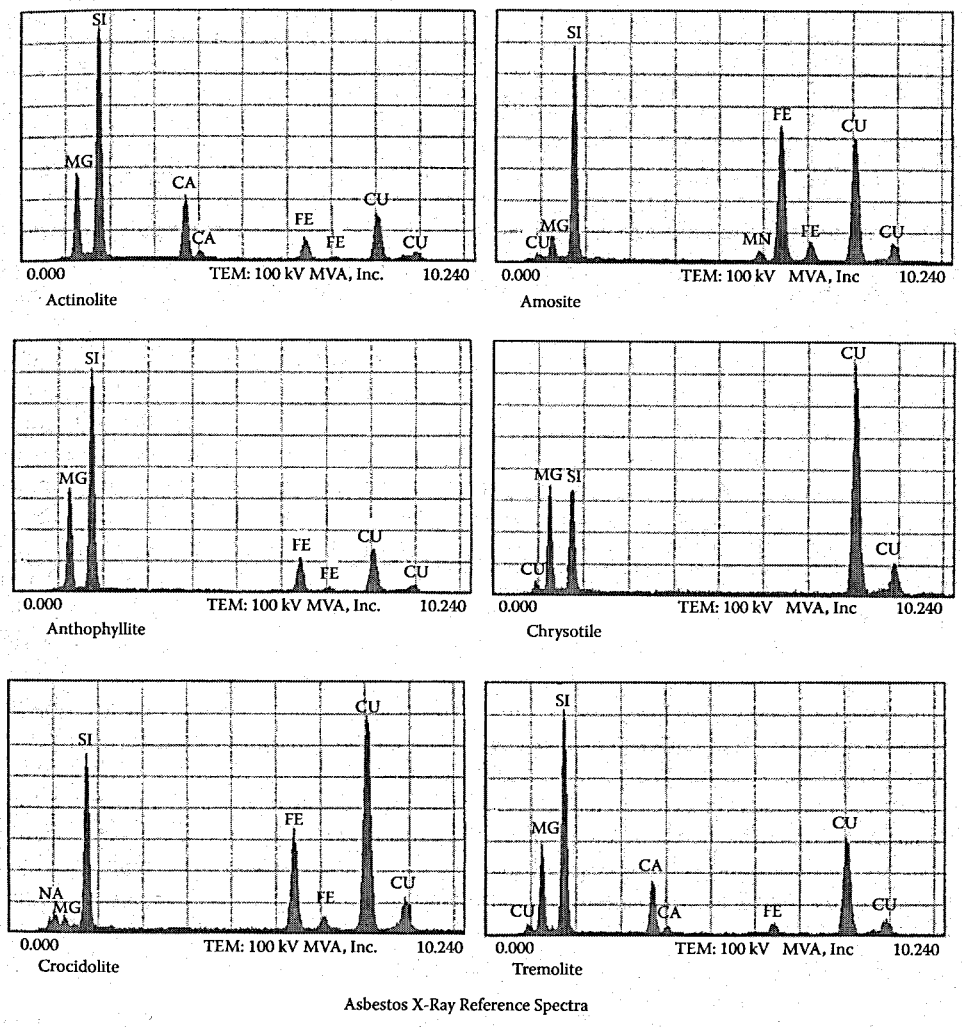

Figure 2.8 EDS x-ray spectra for NIST reference asbestos fibers.

is currently being studied by NIOSH for consideration by OSHA.[20] Under the D7200 method, three classes of elongated particles are established. During PCM analysis, fibers longer than 5 μm with an AR of 3:1 are classified in Class 1 if they show curvature, split ends, or a bundle appearance. Fibers that are greater than 10 μm or less than 1 μm in width are classified as Class 2. All other particles that meet the basic NIOSH definition of a fiber are classified as Class 3. According to the method, if the sum of Classes 1 and 2 are greater than 50% of the total, the data indicate the possibility of an asbestos fiber population. The analyst is to proceed with TEM analysis. Changing the classification criterion of Class 2 to fibers that are greater than 10 μm and less than 1 μm in width is being considered by ASTM.

Early TEM measurements of airborne asbestos such as those used by Nicholson involved the collection of fibers on a membrane filter followed by an indirect-transfer method.[21,22] In the TEM specimen procedure known as the "rubout" method, air samples collected using mixed cellulose ester filters were ashed in a low-temperature plasma asher and the residual ash dispersed in a solution of nitrocellulose. The dispersion was "rubbed out" or spread as uniformly as possible on an

optical microscope slide. After the solvent had evaporated, a portion of the film containing the particles from the filter residue was mounted on a TEM grid for examination. The value of asbestos was reported in terms of nanograms per cubic meter of air. The values were determined by summing the masses of the fibers that were calculated from the TEM dimensions of each fiber and an appropriate density for the type of asbestos found.

In 1978, Samudra et al.[23] published the first methodology for determination of the numerical concentration of asbestos fibers in ambient atmospheres using a direct preparation method. The provisional methodology developed under contract for the U.S. EPA recommended air sampling using a 0.4-μm pore size PC filter and preparation of TEM specimen grids by carbon coating followed closely by chloroform extraction to remove the filter polymer. The Samudra methodology was never taken beyond the provisional status.

In the early 1980s, Yamate et al.[24] at the Illinois Institute of Technology Research Institute were asked under contract by EPA to take the methods that were being used by various laboratories and put together a TEM method for airborne asbestos. Their document, which circulated in draft form in 1984, was never officially adopted by EPA. Although it remained in draft form, it became the generally accepted method for TEM analysis of airborne asbestos. As a fiber definition, it used the minimum AR of 3:1 from the NIOSH and OSHA methods but had no minimum fiber length. However, fibers less than 1 mm at the fluorescent screen magnification level were characterized as being 1 μm. At the analysis magnification of ×20,000, the 1-mm size corresponded to 0.5 μm. In addition to asbestos fibers, the method classified asbestos-containing objects as bundles, clusters, and matrices (see Table 2.2 for a comparison of fiber definitions used by several airborne asbestos analysis methods). Yamate et al.[24] also included the concept of levels of analysis because he realized that analytical tools available with the analytical electron microscope provided progressively more specific identification of asbestos fibers depending on the amount of time devoted to the task. The method's levels are known among the TEM asbestos analytical community as Yamate Level 1, Level 2, and Level 3. Level 1, requiring the least amount of identification, was designed for those situations where the airborne particulate was well characterized. If a particular process was known to emit only chrysotile, Level 1 permitted identification on the basis of morphology alone. For Level 2, asbestos identification was determined by morphology and visual diffraction characteristics for chrysotile. For amphiboles, Level 2 included some x-ray elemental information. Asbestos identification in Yamate Level 3 began with the identification steps in Level 2 and added diffraction pattern indexing to more specifically identify the amphibole.

The Yamate method also contained a section for the situation where an air filter was overloaded. The preparation was an indirect procedure where a portion of the filter was ashed and the ash suspended in water. A second filter was prepared with a portion of the suspension and then processed using the same direct procedures described in the main method.

On October 22, 1986, President Reagan signed into law the Asbestos Hazard Emergency Response Act (AHERA).[25] The Act required that EPA describe the methods used to determine completion of response actions such as the abatement of school buildings. Following the deliberations of a panel of asbestos analysis experts, the "Interim TEM Analytical Methods" were published in the Federal Register on October 30, 1987, as Appendix A to Subpart E of the EPA's "Asbestos-containing Materials in Schools; Final Rule and Notice." Following an asbestos abatement and before the protective plastic barriers are removed, leaf blowers and fans are used to aggressively stir the air and resuspend any settled dust while five area air samples are collected. For abatement clearance, the five area air samples collected inside the containment were to be compared with five or more area air samples collected outside the containment. No aggressive disturbance of the air outside the containment was to be done. If there was no statistical difference between the two sets of samples, the abated area was cleared and prepared for reoccupancy. A simplified version of the Yamate draft method was needed to create a rapid method for the clearance of school buildings. The AHERA method maintained many of the method particulars of the Yamate method but

Table 2.2 Comparison of Fiber Definitions Used in Measuring Asbestos in Air

Fiber—NIOSH 7400 (PCM)	Longer than 5 μm with a length to width ratio equal to or greater than 3:1.
Fiber—NIOSH 7402 (TEM)	All particles with a diameter greater than 0.25 μm that meet the definition of a fiber (AR greater than or equal to 3:1, longer than 5 μm).
Fiber—OSHA ID-160 (PCM)	A particle that is 5 μm or longer, with a length to width ratio of 3:1 or longer.
Fiber—Yamate (TEM)	Particle with an AR of 3:1 or greater and with substantially parallel sides.
Fiber—AHERA (TEM)	A structure greater than or equal to 0.5 μm in length with an AR (length to width) of 5:1 or greater and having substantially parallel sides.
Fiber (fiber)—ISO 10312 (TEM)	An elongated particle that has parallel or stepped sides. For the purposes of this international standard, a fiber is defined to have an AR equal to or greater than 5:1 and a minimum length of 0.5 μm.
Bundle—NIOSH 7400 (PCM)	Not defined in method.
Bundle—NIOSH 7402 (TEM)	Not defined in method.
Bundle—OSHA ID-160 (PCM)	Not defined in method.
Bundle—Yamate (TEM)	Paniculate composed of fibers in a parallel arrangement, with each fiber closer than the diameter of one fiber.
Bundle—AHERA (TEM)	A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
Bundle—ISO 10312 (TEM)	A structure composed of parallel, smaller diameter fibers attached along their lengths. A fiber bundle may exhibit diverging fibers at one or both ends.
Cluster—NIOSH 7400 (PCM)	Not defined in method.
Cluster—NIOSH 7402 (TEM)	Not defined in method.
Cluster—OSHA ID-160 (PCM)	Not defined in method.
Cluster—Yamate (TEM)	Particulate with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group.
Cluster—AHERA (TEM)	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.
Cluster—IS010312 (TEM)	A structure in which two or more fibers, or fiber bundles, are randomly oriented in a connected grouping.
Matrix—NIOSH 7400 (PCM)	Not defined in method.
Matrix—NIOSH 7402 (TEM)	Not defined in method.
Matrix—OSHA ID-160 (PCM)	Not defined in method.
Matrix—Yamate (TEM)	Fiber or fibers with one end free and the other end embedded or hidden by a particulate.
Matrix—AHERA (TEM)	Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the (AHERA) fiber definition.
Matrix—ISO10312 (TEM)	A structure in which one or more fibers, or fiber bundles, touch, are attached to or partially concealed by a single particle or connected group of nonfibrous particles.

simplified the counting and recording for a rapid clearance procedure. As in the Yamate method, structures were counted. A structure was defined as a microscopic bundle, cluster, fibers, or matrix that may contain asbestos. A matrix was defined as a 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. Under the AHERA method, an asbestos fiber was defined as a structure greater than or equal to 0.5 μm in length with an AR (length to width) of 5:1 or greater and having substantially parallel sides. Individual dimensions of structures or fibers are not recorded under the AHERA method, but information about the overall structure size is classified as either between 0.5 and 5.0 μm or greater than 5.0 μm. The size data are not used to determine compliance with the AHERA regulations but is included so that if an area does not pass, the project manager might infer something about the source of the contamination. Many large structures found in the air would suggest improper cleaning, whereas small structures could have come from a source external to the cleaning effort. During

the deliberations of the expert panel, the question was raised about whether all 10 samples needed to be analyzed if no asbestos structures were found on the five inside-the-containment samples. On the basis of experience of some of the panel in finding occasional asbestos fibers on blank (unused) PC filters, it was decided that a sample was clearly above the blank filter level if it had a filter loading greater than 70 structures per millimeter square (str/mm^2). In the real-world abatement industry, the 70 str/mm^2 became the generally recognized clearance level, and contractors were and still are normally instructed to reclean if the average of the five inside samples exceeded that value. Only rarely today is the comparison made of the five inside and five outside samples. Those few cases are usually where a contractor believes that asbestos contamination outside the containment area is contributing to the air within the abatement area.

2.7 SCANNING ELECTRON MICROSCOPY

In 1987 when the AHERA method mandated the use of TEM, the scanning electron microscope was determined to be inadequate for building clearance (Figure 2.9). The reasons given in the AHERA document were as follows: (1) currently available methodologies were not validated for the analysis of asbestos fibers, (2) SEM was limited in its ability to identify the crystalline structure of a particular fiber, (3) the National Bureau of Standards found that the image contrast of the microscopes was difficult to standardize between individual scanning electron microscopes, and (4) no current laboratory accreditation program existed for accrediting SEM laboratories.[25] NBS had determined that the only SEM method recognized at that time, the Asbestos International Association protocol,[26] had inherent difficulty when examining certain types of asbestos. As of 2009, there are still no laboratory accreditation programs for SEM laboratories. In the United States, no standard SEM method is in use for asbestos, although it is mentioned in the OSHA ID-160 method. However, there is interest internationally, and the ISO 14966 method for SEM analysis of inorganic fibrous particles that includes asbestos (Figure 2.10), ceramic fibers, and glass fibers in air was approved in 2002.[27]

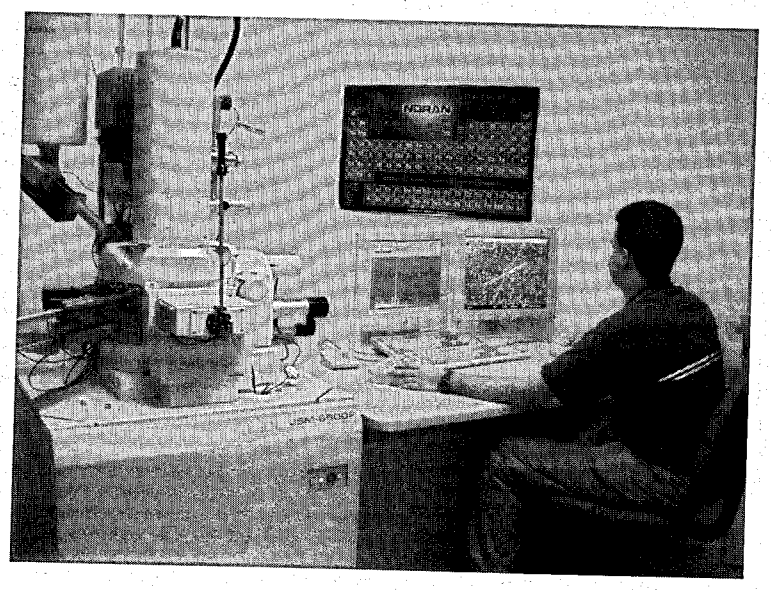

Figure 2.9 Analyst using an SEM for asbestos analysis.

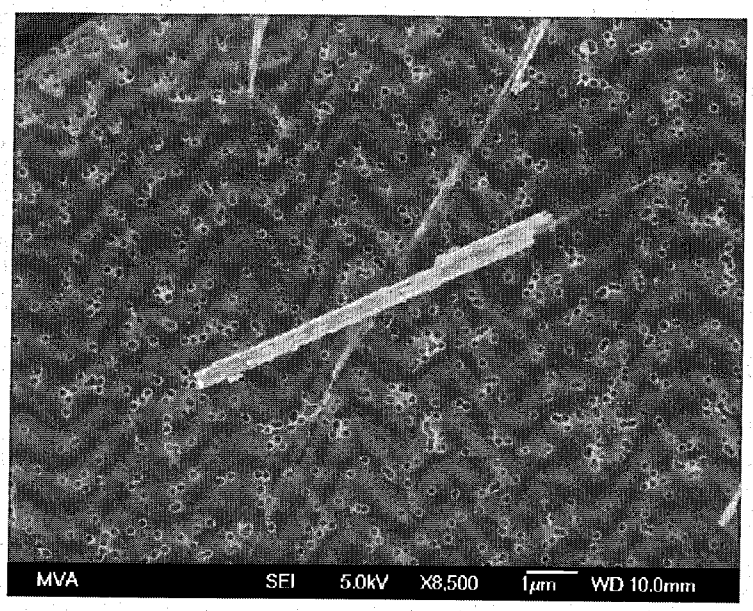

Figure 2.10 Image of crocidolite and chrysotile asbestos fibers as seen with an SEM. Same fibers as shown in Figure 2.6.

2.8 TEM BEYOND AHERA

In 1987 when the AHERA method was published in the Federal Register as an interim method, it contained a provision that the method would be updated by the National Institute of Standards and Technology (NIST). As of 2009, no updated version of the method has been published by NIST or any other federal agency. The AHERA method became the generally accepted TEM method for the analysis of asbestos in air. However, its lack of specific size data for individual asbestos structures was considered a deficiency for some situations. A Yamate Level 2 analysis was requested on occasions when information about fiber size was needed. In March 1988, the CARB issued Method 427 for the determination of paniculate asbestos emissions from stationary sources using stack sampling, light microscopy, and electron microscopy.[28] Although the NIOSH 7400 PCM method may be used with the CARB 427 method, it is evident that the TEM portion is the focus of the method. Recording of fiber size data is done on the basis of the Yamate method.

A more complete TEM airborne asbestos analysis procedure developed largely by Dr. Chatfield of Chatfield Technical Consulting was released in 1995 by the ISO.[29] The International Standard 10312 contains counting rules which expand on the Yamate and AHERA concept of asbestos structures. Clusters and matrices are subdivided into dispersed and compact structures. A dispersed cluster contains asbestos fibers that can be measured and reported separately, whereas a compact cluster has fibers too intertwined to be reported individually. In this method, cluster and matrix components are identified, measured, and recorded separately up to a maximum of nine substructures. The ISO 10312 was followed in 1998 by the ASTM Standard Test Method D6281-98, which was a translation of the ISO 10312 method into ASTM format with a few improvements and changes.[30] The ASTM Method D6281 was reapproved in 2002 as D6281-02. For samples that contain any appreciable amount of asbestos, analysis by either ISO 10312 or ASTM D6281 is considerably more time consuming than an AHERA analysis and therefore more expensive. The data produced by ISO 10312/ASTM D6281 were designed to allow another analyst to review the data of the original analyst and understand how the asbestos structures were present on the filter grid. The method of

data recording was designed to allow reevaluation of the counting data as new medical evidence or regulatory requirements become available. From the results of an ISO 10312 (or ASTM D6281) analysis, it should be possible to determine several different airborne asbestos structure concentration values on the basis of a number of fiber size classifications. For instance, it should be possible to extract what a structure per cubic centimeter concentration would have been if the sample had been analyzed by AHERA counting rules. Some precision data have been determined for the ASTM D6281 method (also applicable to ISO 10312). The ASTM interlaboratory studies found relative standard deviations of 0.1 for amphibole fibers (amosite) and 0.5 for chrysotile fibers.

Both ISO 10312 and ASTM D6281 have an annex, which describes procedures for the determination of concentrations of asbestos fibers and bundles longer than 5 μm and of PCM-equivalent (PCME) asbestos fibers. For improved analytical sensitivity and statistical precision, the larger fiber counts are done at lower magnifications so more area of the filter may be examined. The U.S. EPA has set a risk-based cleanup benchmark for asbestos in air for their World Trade Center (WTC) Test and Clean Program. The EPA risk-based benchmark for PCME asbestos fibers analyzed by TEM (ISO 10312 or ASTM D6281) is 0.0009 s/cc.[31] A comparison of four common asbestos methods for the analysis of air samples is shown in Table 2.3.

In 1999, ISO 13794 (indirect air) was published.[32] The asbestos structure and the fiber counting procedures in this method are the same as those presented in ISO 10312 and ASTM D6281. ISO 13794 provides an indirect-transfer procedure so overloaded filters can be analyzed. The filter preparation methods described in both ISO 10312 and ASTM D6281 are direct-transfer procedures. In steps similar to the Yamate indirect preparation procedure, a portion of the original filter is ashed and the ash suspended in water. A second filter is prepared with a known portion of the suspension and then processed using the same direct procedures described in ISO 10312 and ASTM D6281. Although the method states "This International Standard is applicable to measurement of airborne asbestos in a wide range of ambient air situations, including the interior atmospheres of buildings, and for detailed evaluation of any atmosphere in which asbestos fibers are likely to be present," the user is cautioned that comparison of results using this indirect-transfer procedure with those from a direct-transfer procedure may not be done *a priori*.[32] The best study of the differences between direct and indirect air sample preparation remains the study by Chesson and Hatfield.[33] Their findings supported the generally accepted opinion that TEM analysis of air samples using indirect-transfer methods provides estimates of the total airborne asbestos structure concentration that are higher than those using direct-transfer methods. They concluded that no single factor can be used to convert measurements made by one method to a value that is comparable with measurements made by the other. They also concluded that the breakdown of larger structures into smaller ones

Table 2.3 Comparison of Common Methods for Measuring Asbestos in Air

	NIOSH 7400	NIOSH 7402	AHERA	ISO
Instrument	PCM	TEM	TEM	TEM
Filter preparation	Direct	Direct	Direct	Direct: 10312; Indirect: 13794
Magnification	×450	×10,000	~×20,000	~×20,000
Fiber length	$L > 5$ μm	$L > 5$ μm	$L > 0.5$ μm	$L > 0.5$ μm
Fiber diameter	$W > 0.25$ μm	$W > 0.25$ μm	$W > 0.002$ μm	$W > 0.002$ μm; PCME: $L > 5$ μm, $W > 0.25$ μm
AR	>3:1	>3:1	>5:1	>5:1 or 3:1
Counting	Fibers	Fibers	Structures	Structures and fibers
Identification	None	Morphology, crystal structure, elements	Morphology, crystal structure, elements	Morphology, crystal structure, elements
Reporting	Fibers/cm^3	% asbestos	All asbestos str/cm^3 and >5 μm structures	All asbestos str/cm^3 and >5 μm fibers and PCME fibers/cm^3

during indirect preparation does not appear to be sufficient to explain the difference in measured concentrations. Interference by debris and association of unattached structures may also be important. They recommended additional research was needed to determine which transfer method more accurately reflects biologically meaningful airborne asbestos concentrations.

2.9 WATER ANALYSIS

There are three standard methods available for the analysis of drinking water for asbestos: the EPA 100.1, the EPA 100.2, and the AWWA 2570.[34–36] These methods are all TEM methods and are compared in Table 2.4.[37] The EPA has set a maximum contaminant level of seven million fibers longer than 10 μm/L of drinking water and has listed both the 100.1 and 100.2 methods as acceptable for the analysis of water-borne asbestos. The EPA 100.1 method is a research report produced in 1984 before the EPA drinking water regulations and describes counting procedures that include asbestos fibers longer than 0.5 μm. The EPA 100.2 describes counting only those fibers longer than 10 μm. Guidance as to the modifications of EPA 100.1 necessary to comply with the EPA drinking water regulations was published by Feige et al.[38] Some precision data for the water method were described in EPA 100.1. An interlaboratory comparison study with six laboratories found relative standard deviations for amphibole fibers (crocidolite) of 25% and for chrysotile fibers of 29%.[34] The ELAP Certification Manual Item 198.2 describes a modification to Method 100.2 required for New York State Department of Health compliance.[39] In the modification, the ozone generator is considered optional *only* if all samples are filtered within 48 h.

2.10 SURFACE DUST ANALYSIS

In 1989, the ASTM subcommittee D22.07 began work on methods for the analysis of asbestos in settled dust.[40] Three ASTM methods are currently available for the analysis of surface dust for asbestos. These methods include two microvacuum methods: ASTM D5755-02 (structure count) and D5756-02 (mass) and one wipe method, ASTM D6480-99.[41–43] An EPA carpet method, EPA/600/J-93/167, was developed during a research study that was published as an article in 1993.[44] The EPA number was assigned in 2001. The three ASTM methods are nondestructive, whereas the carpet method requires that a piece be cut from the carpet and sent to the laboratory. Some precision data have been determined for the ASTM D5755 method. The ASTM interlaboratory study found a relative standard deviation of 0.5 for samples of WTC dust containing chrysotile fibers. A comparison of the four surface (settled) dust methods is shown in Table 2.5.

Table 2.4 Comparison of Common Methods of Measuring Asbestos in Water

	EPA 100.1	EPA 100.2	AWWA 2570
Instrument	TEM	TEM	TEM
Filter preparation	Indirect PC	Indirect (PC and MCE)	Indirect (PC and MCE)
Magnification	~x20,000	~x20,000	~x20,000
Fiber length	$L > 0.5$ μm	$L > 10$ μm	$L > 0.5$ μm
Fiber diameter	$W > 0.002$ μm	$W > 0.002$ μm	$W > 0.002$ μm
AR	>5:1	>5:1	>5:1
Counting	Fibers	Fibers	Fibers
Identification	Morphology, crystal structure, elements	Morphology, crystal structure, elements	Morphology, crystal structure, elements
Reporting	Millions of asbestos fibers per liter (MFL)	MFL > 10 μm	MFL

Table 2.5 Comparison of Common Methods for Measuring Asbestos in Surface Dust

	ASTM D5755-02	ASTM D5756-02	ASTM D6480-99	EPA/600/J-93/167
Instrument	TEM	TEM	TEM	TEM
Sample preparation	Microvacuum (indirect)	Microvacuum (indirect)	Wipe (indirect)	Piece of carpet (indirect)
Magnification	~x20,000	~x20,000	~x20,000	~x20,000
Fiber length	$L > 0.5$ μm	$L > 0.5$ μm	$L > 0.5$ μm	$L > 0.5$ μm
Fiber diameter	$W > 0.002$ μm	$W > 0.002$ μm	$W > 0.002$ μm	$W > 0.002$ μm
AR	>5:1	>5:1	>5:1	>5:1
Counting	Asbestos structures	Asbestos structures	Asbestos structures	Asbestos structures
Identification	Morphology, crystal structure, elements	Morphology, crystal structure, elements	Morphology, crystal structure, elements	Morphology, crystal structure, elements
Reporting	Asbestos s/cm^2	Asbestos μg/cm^2	Asbestos s/cm^2	Asbestos s/cm^2 of carpet

Because dust particles can be arranged in layers more than one particle thick, direct preparation techniques are of limited value for TEM because the electron beam must be able to penetrate the sample. Indirect preparation procedures are used for all four methods. The results of the analysis are expressed in numbers or mass of asbestos structures per square centimeter of surface sampled. The number count methods were originally designed with an analytical sensitivity of approximately 1000 str/cm^2 but can achieve much better sensitivities on clean surfaces. A nominal analytical sensitivity for the mass determination is 0.24 pg of asbestos/cm^2. There is some disagreement on how to interpret the surface dust asbestos data.[45–53] The U.S. EPA has set risk-based cleanup benchmarks for asbestos in settled dust for their WTC Test and Clean Program. The EPA risk-based benchmarks for asbestos analyzed according to the ASTM D5755 method (microvacuum count) are 5000 s/cm^2 for accessible areas (e.g., floors) and 50,000 s/cm^2 for infrequently accessed areas (e.g., behind a bookshelf). At EPA's Libby, Montana site, an action level of 5000 s/cm^2 in generally accessible areas has been established for triggering cleanup in a residential dwelling.

Because the amount and the type of surface dust collected by each method differ, it is clear that results of one settled dust method cannot be necessarily compared directly with data from another. For instance, the bulk carpet method, EPA/600/J-93/167, is an analysis of the total amount of dust in a carpet. Because carpets are known to be excellent traps for dust and dirt, the amount of asbestos in the carpet may be considerably higher than that collected from the surface of the same carpet using the D5755 microvacuum method. It is not appropriate to compare bulk carpet values with results of the D5755 method or with the EPA risk-based cleanup benchmarks, although both are given in terms of structures per square centimeter. In one set of tests, the EPA/600/J-93/167 results were found to be approximately 100 times higher than that of the D5755 type analysis because the bulk carpet method involves all dirt trapped in the carpet and the microvacuum method only analyzed the top, readily releasable dust.[44] Asbestos in dust deep in the carpet may not be releasable under normal activities and may only be of concern when the carpet is being removed. Asbestos fibers that are in a sticky film on a surface and therefore not readily releasable are collected by the D6480 wipe method. The wipe method gives an index of all asbestos fibers on a surface regardless of how stuck they are, whereas the microvacuum method gives an index of the readily releasable fibers.

2.11 SOIL ANALYSIS

Soil is a difficult medium for the analysis of asbestos because soil minerals are not easily separated from the asbestos fibers. In a method used by the U.S. EPA Region 1, sieving is used to enhance the ability to find asbestos fibers that are then identified using essentially the standard PLM bulk analysis procedure.[54] The Australian Standard Bulk Method for the qualitative identification of asbestos

in bulk samples has a section specifically for soil samples.[55] The entire sample is screened through a 10-mm (1 cm) sieve. The less than 10-mm fraction is then sieved through a 2-mm sieve. The less than 2-mm fraction is spread to a thickness of no more than 1 to 3 mm. Using a combination of low- and high-power stereomicroscopy, all fractions are examined for fibers that are then identified by PLM with dispersion staining. The fibrous particles that are found are weighed, or the length and widths of each fiber bundle are estimated. The method describes a reporting limit of 0.1 g/kg or 0.01%.

The chrysotile flotation method described by Falini et al.[56] in 2003 appeared to be a method that could separate asbestos from soil without drastically changing the fiber size distribution. However, studies of the Falini et al.[56] method concluded that the chrysotile flotation method does not provide an effective and efficient way to completely and cleanly separate asbestos with a range of fiber sizes from soils. Although the method may be gentle enough to maintain the integrity of the asbestos fibers (i.e., without fiber length reduction), the processing lost the longer and thicker fibers, thus not maintaining the integrity of the fiber size distribution. In the CARB 435 method, "Determination of Asbestos Content of Serpentine Aggregate," the sample aggregates are crushed to produce a material with a nominal size of less than three-eighths of an inch.[57] The samples are further crushed using a Braun mill or equivalent to produce a material of which the majority shall be less than 200 Tyler mesh (76-μm diameter). Asbestos identification is done by PLM and the determination of the amount of asbestos is done using a 400-point count. Crushing of the samples is necessary because all particles must fit under the coverslip on the microscope slide and be of a uniform size for point counting. The lower detection limit is described as 0.25% (1 asbestos fiber/400 nonempty points). However, there is considerable statistical uncertainty in this value.[12]

Currently, soil methods designed around both grinding and sieving are under consideration. It is thought that crushing or grinding the soil sample will produce a more homogeneous mixture, which should improve the precision of the analysis. However, interlaboratory comparison studies done among West Coast laboratories have shown that the type of crusher and other factors need more investigation to achieve the level of precision needed. Although the sieving approach is thought to be more difficult in terms of achieving good quantitative results than the grinding approach, it should provide information about the fiber sizes, which is lost during a repertory grinding step. A soil method that uses sieving, PLM, and TEM is currently under development by the ASTM Subcommittee D22.07.

A more complicated procedure, which looks at airborne asbestos fibers that might be released from soil, is called the Superfund method.[58,59] The soil sample is placed in a rotating drum, and air samples are collected in a vertical elutriator. The samples are analyzed by TEM according to procedures on the basis of the ISO 10312 method. The counting procedure may be modified to count "protocol" fibers. Protocol fibers are asbestos fibers with certain length and width characteristics as determined by studies in biological systems. At one point in time, fibers longer than 40 μm were thought to be of greatest interest, and the method was modified to count more grid openings at a lower magnification for better counting statistics. A comparison of the two soil methods is shown in Table 2.6.

Table 2.6 Comparison of Common Methods for Measuring Asbestos in Soil

	EPA Superfund	EPA Region 1 CARB 435
Instrument	TEM	PLM
Sample preparation	Elutriator	Sieving crushing
Magnification	~x20,000	x10–1000
Fiber length; diameter	$L > 0.5$ μm; $W > 0.002$ μm	$W > \sim 1$ μm
AR	>5:1	>3:1
Counting	Structures	Areal % point count
Identification	~ISO 10312	Optical
Reporting	Various	% asbestos

2.12 VERMICULITE ANALYSIS

Vermiculite is also a special case for bulk asbestos analysis. Sometimes referred to as "The Cincinnati Method," the EPA research method for the sampling and analysis of fibrous amphibole in vermiculite attic insulation uses a flotation step to separate the vermiculite from the more dense amphiboles.[60] The fibrous amphiboles found in Libby, Montana, vermiculite can be hand picked from the "sinks" using a stereomicroscope and weighed to get a direct weight percent estimate. The method includes a TEM portion for the analysis of amphibole fibers that might be present in the "suspended particle" fraction of the water used in the flotation step. Criteria for examination of the TEM specimens are specified in ISO 10312 or ISO 13794. Early in 2004, EPA held a day and a half workshop for a panel of experts to meet and propose a method to determine whether Libby amphibole is present in a sample of vermiculite attic insulation. The objective of the method is to be accurate with respect to identifying Libby amphibole, to be affordable to the average homeowner, and to be adaptable to most current commercial fiber analysis laboratories. This more routine vermiculite method, on the basis of the Cincinnati research method, was expected to be released in late 2004. The routine method is still currently under development.

2.13 METHODS FOR ASBESTOS ANALYSIS IN OTHER MEDIA

In addition to media such as air, water, soil, and dust, methods for analyzing asbestos in clothing, talc, and biological specimens have appeared in the scientific literature.[61–64] Only a few of the many scientific papers that contain descriptions of asbestos analysis methods are referenced here. Sample preparation procedures are generally different for each type of sample matrix, but the type of microscopy to be used and the counting rules are usually borrowed from one of the standard methods described earlier.

2.14 ASBESTOS DEFINITIONS AND TERMINOLOGY

The definition of a "Federal Asbestos Fiber" depends on the federal agency involved. The OSHA uses a definition of a fiber that is at least 5 μm long with an AR (length to width) of 3:1. The EPA uses a definition of a fiber that is at least 0.5 μm long with a 5:1 AR. The ISO and the ASTM TEM methods use a definition of a fiber that is 0.5 μm long with a 5:1 AR definition in their main procedure and provide an annex, which describes counting fibers greater than 5 μm long with an AR of 3:1. Other ARs such as 10:1 and 20:1 have been suggested for defining an asbestos fiber but have not been adopted.

From the microscopic analyst's point of view, an asbestos fiber is defined by the counting method being used. Under the AHERA counting rules, a fiber is a structure having a minimum length greater than 0.5 μm and an AR (length to width) of 5:1 or greater and substantially parallel sides. The appearance of the end of the fiber, that is, whether it is flat, rounded, or dovetailed, is to be noted. However, AHERA does not use information about fiber ends, nor does it say whether to record this information. Under Section 3.22 of the ISO 10312 counting rules (and a similar section in ASTM D6281), a fiber is defined as an elongated particle that has parallel or stepped sides.

Individual chrysotile fibers, called fibrils, are too thin to be seen by the light microscope during the PCM analysis by NIOSH 7400. The fibers of chrysotile that are seen in the light microscope are actually bundles of fibrils. During the analysis by TEM using the NIOSH 7402 method that considers only elongated particles longer than 5 μm in length and greater than 0.25 μm in width with a 3:1 AR, the chrysotile "fibers" are more correctly listed as bundles. As stated in the ISO 10312 method: "For chrysotile, PCME fibers will always be bundles."[29] During the analysis by TEM using the AHERA method, chrysotile fibrils are listed as fibers. These AHERA chrysotile "fibers" (actually

fibrils less than 0.05 μm in diameter) are not visible with the light microscope. Similar terminology is used in the water and dust methods. With the exception of the NIOSH 7402 method, all TEM chrysotile fibers are actually fibrils and not visible with the light microscope.

2.15 PCM EQUIVALENCY

The U.S. NIOSH 7400 standard method uses PCM and involves counting only those fibers that can be seen with the light microscope (thicker than 0.25 μm) and longer than 5 μm. The TEM companion method NIOSH 7402 considers the same fiber characteristics as the 7400 method, but because the TEM can resolve thin asbestos fibers, 7402 analysis is restricted to fibers greater than 0.25 μm. The TEM fibers analyzed under NIOSH 7402 are then PCME fibers. However, the NIOSH 7402 method is not established to provide concentrations of asbestos fibers. The determination and reportable value from 7402 is a percentage of asbestos fibers of all fibers in the PCME range in the sample. This percentage can thereby be applied to 7400 values to determine asbestos fiber concentrations in fibers per cubic centimeter. Other TEM methods (primarily ISO 10312 and occasionally AHERA) have been used to determine PCME concentrations. It is important when interpreting the data to understand the differences in counting rules between methods. Appendix B of Method NIOSH 7400 contains a description of the asbestos fiber counting rules (referred to as "A" rules) as they apply to labeled objects in Figure 2.2 of the 7400 method. For Object 2 in Figure 2.2, the method states: "Although the object has a relatively large diameter (>3 μm), it is counted as a fiber under the rules. There is no upper limit on the fiber diameter in the counting rules." The ISO 10312 method defines a PCME fiber as "any particle with parallel or stepped sides, with an AR of 3:1 or greater, longer than 5 μm and which has a diameter between 0.2 and 3.0 μm." Using the ISO 10312 method for PCME counting will, therefore, not provide a count of PCM fibers equivalent to the NIOSH 7400 method, unless it is modified so that fibers of all diameters are included.

More serious cautions are appropriate for the attempt to use AHERA counts to estimate PCME concentrations. It is important to realize that the NIOSH 7400 method includes fibers associated with other particles. For Object 6 in Figure 2.2, the NIOSH 7400 method states: "A fiber partially obscured by a particle is counted as one fiber. If the fiber ends emanating from a particle do not seem to be from the same fiber and each end meets the length and AR criteria, they are counted as separate fibers." The AHERA method counts all asbestos objects as structures. Objects that contain one or more fibers partially obscured by a particle are counted as matrices. Under the NIOSH PCM method, several fibers meeting the length and AR criteria, which are overlapping but do not seem to be part of the same bundle, would be counted as separate fibers. Under the AHERA TEM method, these would all be counted as one cluster. If an analyst tries to use the AHERA data to estimate a PCME fiber count and chooses only those structures identified as bundles greater than 5 μm, they will miss PCME fibers that are parts of matrices or clusters. Because AHERA uses a 5:1 AR while the PCM method uses a 3:1 ratio, an AHERA count would not have included fibers more than 5 μm with only a 3:1 AR. Considering the differences in the two methods, it is not appropriate to attempt to estimate PCME fiber concentrations from AHERA data. However, an AHERA analysis in which no asbestos structures are found is considered to be consistent with no PCME fibers detected. It would be a most unusual sample to have no AHERA countable asbestos structures but still have some large fibers with ARs between 3:1 and 5:1.

2.16 CLEAVAGE FRAGMENTS

Most asbestos methods dictate the counting of the asbestos forms of six minerals: one serpentine type (chrysotile) and five amphiboles (amosite, anthophyllite, actinolite, crocidolite, and tremolite).

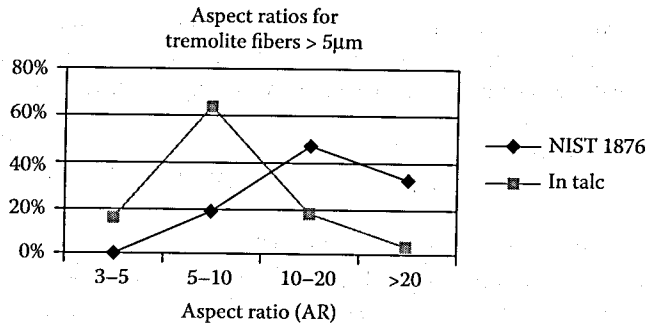

Figure 2.11 Comparison of ARs for tremolite fibers from the Standard Reference Material 1876, tremolite asbestos, and tremolite from a talc sample.

Elongated particles with ARs greater than 3:1 or 5:1 that did not come from a population of asbestos fibers are sometimes called cleavage fragments. The distinction of how to tell an asbestos fiber from a cleavage fragment is currently being debated within the scientific community. A population of fibers as observed in a bulk sample having the asbestiform habit is generally recognized by several characteristics.[5] These include mean ARs in the range from 20:1 to 100:1 or higher for fibers longer than 5 µm. Asbestos is characterized by very thin fibrils, usually less than 0.5 µm in width, and two or more of the following:

- Parallel fibers occurring in bundles
- Fiber bundles displaying splayed ends
- Matted masses of individual fibers
- Fibers showing curvature

It is more difficult to classify individual fibers as to asbestiform or cleavage fragments because individual fibers do not exhibit all the characteristics of a population. With the exception of the requirements given in the TEM standard methods that the asbestos fibers have substantially parallel or stepped sides, there is little specific information for the analyst in the way of asbestos or cleavage fragment differentiation. Research has shown that a population of cleavage fragment particles has a smaller mean AR than a population of commercial asbestos fibers. However, the AR distributions of the two populations can overlap and, on an individual basis, some fibers could be classified either way. In Figure 2.11, the ARs of tremolite fibers found in a talc sample are compared with the ARs determined from the NIST standard reference tremolite asbestos sample SMR 1876. The population of tremolite fibers in talc is considered to be nonasbestiform because the mean AR is less than 20:1. However, some individual tremolite fibers in talc like the one shown in Figure 2.12 would be counted as an asbestos fiber under standard methods if found by itself. There is particular difficulty in using bulk characteristics of amphibole asbestos fibers found in air samples. Air sample filters produced from standard reference amosite asbestos fibers contain many fibers but very few parallel fibers occurring in bundles, fiber bundles displaying splayed ends, matted masses of individual fibers, or fibers showing curvature.

2.17 AMPHIBOLES

For most standard asbestos methods, "asbestos" means chrysotile and the asbestiform varieties of the five amphiboles: crocidolite (riebeckite), amosite (cummingtonite–grunerite), anthophyllite, tremolite, and actinolite. The ISO 10312 method is more open-ended in its definition of asbestos

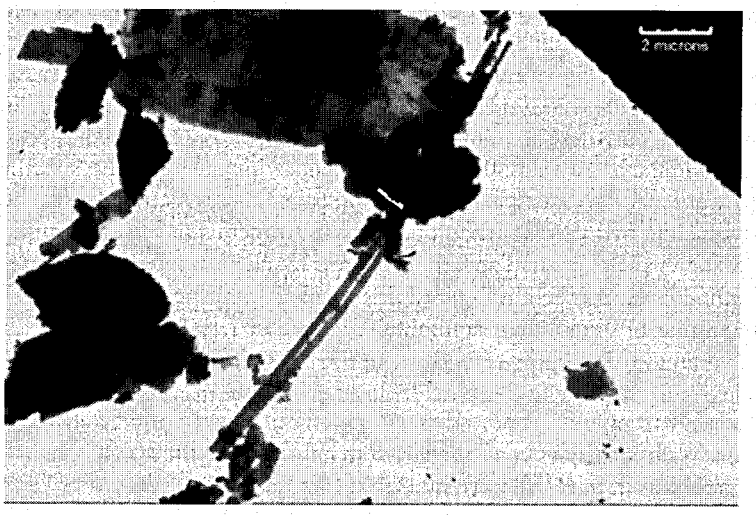

Figure 2.12 TEM image of a tremolite fiber found in a talc sample.

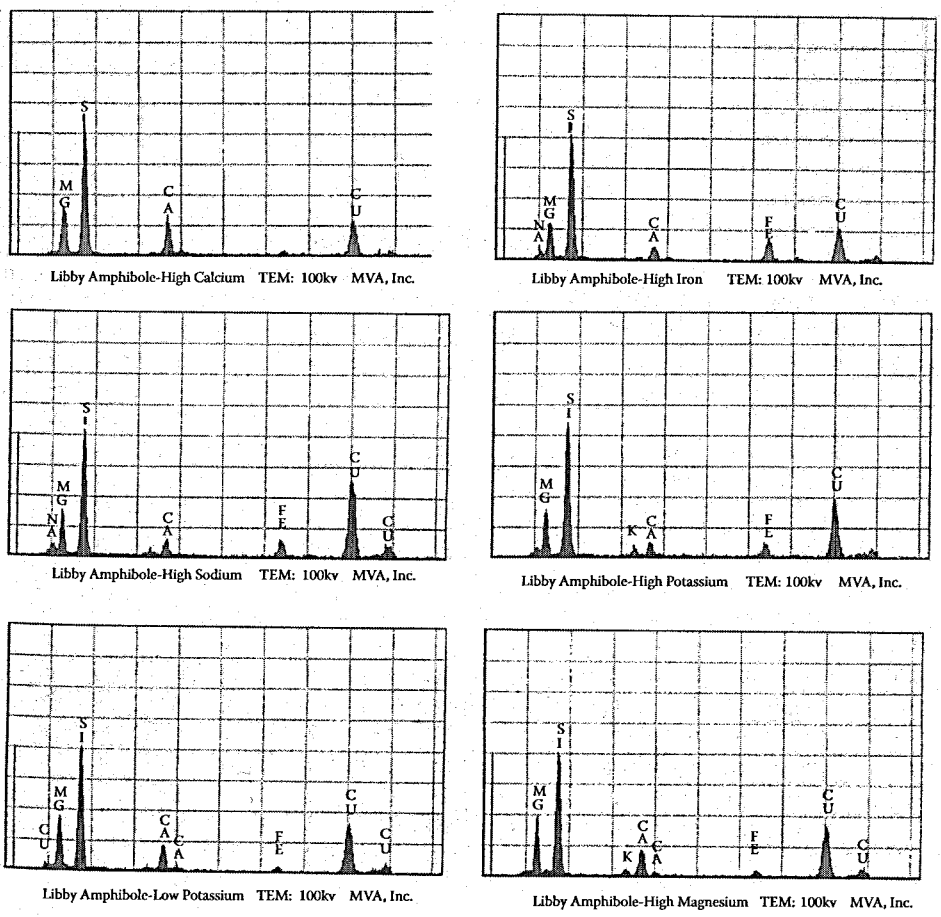

Figure 2.13 EDS x-ray spectra for Libby amphiboles.

with the statement "the most common asbestos varieties are:" (the varieties listed on page 42). Other amphiboles can also exhibit asbestiform habits. The difference between nonregulated asbestiform amphiboles and those that are regulated is the amount of elemental substitution that occurred when the mineral was formed in the earth. The different amphibole names as defined by different elemental compositions are described in Leake et al.[65] Among the amphiboles present in vermiculite from Libby, Montana are tremolite, richterite, and winchite.[66–69] The specific mineralogical determinations were made after extensive mineralogical studies. Distinguishing between tremolite, richterite, and winchite by PLM is difficult because the minerals have very similar optical properties. Figure 2.8 shows the elemental spectra produced by NIST reference asbestos materials using TEM–EDS methodology. As seen in Figure 2.13, the elemental spectra from several Libby amphibole fibers are similar to tremolite or actinolite reference materials but differ in small amounts of sodium and potassium.

The association of amphibole fibers with some chrysotile ores was reported by Addison and Davies.[70] Ilgren and Chatfield[71] reported finding tremolite in the ore from the Jeffrey Mine in Quebec, Canada. Williams-Jones et al.[72] reported that the bulk of the amphibole in the Jeffery Mine in Quebec, Canada was in the form of tremolite and actinolite.

The quantitative analysis for low levels of amphibole fibers in chrysotile or chrysotile-containing products requires that the sample be prepared in a way that eliminates the chrysotile and concentrates the amphibole fibers so they may be detected. This is done first with an ashing at 600°C to aid digestion by opening the chrysotile fibers and to eliminate any organic material such as cellulose fibers. The ashing is followed by an acid digestion step, which is followed by a base digestion step. The residue can then be analyzed by x-ray diffraction or TEM. Using this analysis, low levels (less than 1%) of tremolite and/or actinolite have been found in a number of chrysotile-containing products including, sheet gaskets, packing, brakes, and dryer felt.

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