Designation: D7521 – 16

Standard Test Method for Determination of Asbestos in Soil[1]

This standard is issued under the fixed designation D7521; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure to: (*1*) identify asbestos in soil, (*2*) provide an estimate of the concentration of asbestos in the sampled soil (dried), and (*3*) optionally to provide a concentration of asbestos reported as the number of asbestos structures per gram of sample.

1.2 In this test method, results are produced that may be used for evaluation of sites contaminated by construction, mine and manufacturing wastes, deposits of natural occurrences of asbestos (NOA), and other sources of interest to the investigator.

1.3 This test method describes the gravimetric, sieve, and other laboratory procedures for preparing the soil for analysis as well as the identification and quantification of any asbestos detected. Pieces of collected soil and material embedded therein that pass through a 19-mm sieve will become part of the sample that is analyzed and for which results are reported.

1.3.1 Asbestos is identified and quantified by polarized light microscopy (PLM) techniques including analysis of morphology and optical properties. Optional transmission electron microscopy (TEM) identification and quantification of asbestos is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about fiber size may also be determined. The PLM and TEM methods use different definitions and size criteria for fibers and structures. Separate data sets may be produced.

1.4 This test method has an analytical sensitivity of 0.25 % by weight with optional procedures to allow for an analytical sensitivity of 0.1 % by weight.

1.5 This test method does not purport to address sampling strategies or variables associated with soil environments. Such considerations are the responsibility of the investigator collecting and submitting the sample. Appendix X2 covering elements of soil sampling and good field practices is attached.

1.6 *Units*—The values stated in SI units are to be regarded as the standard. Other units may be cited in the method for informational purposes only.

1.7 *Hazards*—Asbestos fibers are acknowledged carcinogens. Breathing asbestos fibers can result in disease of the lungs including asbestosis, lung cancer, and mesothelioma. Precautions should be taken to avoid creating and breathing airborne asbestos particles when sampling and analyzing materials suspected of containing asbestos.

1.8 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C136 Test Method for Sieve Analysis of Fine and Coarse Aggregates

D1193 Specification for Reagent Water

D3670 Guide for Determination of Precision and Bias of Methods of Committee D22

D6281 Test Method for Airborne Asbestos Concentration in Ambient and Indoor Atmospheres as Determined by Transmission Electron Microscopy Direct Transfer (TEM)

D6620 Practice for Asbestos Detection Limit Based on Counts

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *EPA Standards:*[3]

EPA 600/R-93/116 Method for the Determination of Asbestos in Bulk Building Materials

[1] This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from United States Environmental Protection Agency (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20004, http://www.epa.gov.

2.3 *ISO Standards:*[4]

ISO 10312:1995 Ambient Air -Determination of Asbestos Fibers–Direct Transfer Transmission Electron Microscopy Method (1st Ed. 1995-05-01)

ISO 17025 General requirements for the competence of testing and calibration laboratories

ISO/DIS 22262-1 Bulk materials—Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials

3. Terminology

3.1 *Definitions:*

3.1.1 *asbestiform, n*—type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils.

3.1.1.1 *Discussion*—This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see Steel and Wylie[5] and Zussman.[6]

3.1.2 *asbestos, n*—a collective term that describes a group of naturally occurring, inorganic, highly-fibrous, silicate minerals that are easily separated into long, thin, flexible, strong fibers when crushed or processed.

3.1.2.1 *Discussion*—Included in the definition are the asbestiform varieties of serpentine (chrysotile); riebeckite (crocidolite); grunerite (grunerite asbestos [Amosite]); anthophyllite (anthophyllite asbestos); tremolite (tremolite asbestos); and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to the nomenclature of the International Mineralogical Association.

3.1.2.2 *Discussion*—The mineral fibers described in this definition are listed below. This method is also applicable to other mineral fibers of interest not listed in Table 1.

3.1.3 *aspect ratio, n*—ratio of the length of a fibrous particle to its average width.

3.1.4 *bundle, n*—structure composed of two or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.

3.1.5 *cluster, n*—structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers shall have more than two points touching.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[5] Steel, E., and A. Wylie, "Mineralogical Characteristics of Asbestos," in *Geology of Asbestos Deposits*, P. H. Riordon, Ed., SME-AIME, 1981, pp. 93–101.

[6] Zussman, J., "The Mineralogy of Asbestos," in *Asbestos: Properties, Applications and Hazards*, John Wiley and Sons, 1979, pp. 45–67.

TABLE 1 Asbestos

Asbestos	Chemical Abstract Service No.
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Amosite	12172-73-5
Anthophyllite asbestos	77536-67-5
Tremolite asbestos	77536-68-6
Actinolite asbestos	77536-66-4
Asbestos	1332-21-4

3.1.6 *fiber (transmission electron microscopy, TEM), n*—structure having a minimum length of 0.5 um, an aspect ratio of 5:1 or greater, and substantially parallel sides.

3.1.7 *fibril, n*—single fiber that cannot be separated into smaller components without losing its fibrous properties or appearance.

3.1.8 *fibrous (polarized light microscopy, PLM), adj*—mineral composed of parallel, radiating, or interlaced aggregates of fibers from which the fibers may or may not be separable, that is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers but has that distinct appearance.

3.1.8.1 *Discussion*—The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. The term fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals can have a fibrous habit, not all minerals having fibrous habits are asbestos.

3.1.9 *free fibers, n*—during sample collection, these are fibers that are not associated with discrete pieces of building material or debris in the soil.

3.1.9.1 *Discussion*—Free fibers may or may not be visible to the unaided eye. Their source (for example, weathered asbestos-cement products) may or may not be present in the soil in an amount sufficient to collect a bulk sample, if at all.

3.1.10 *matrix, n*—structure in which one or more fibers, or fiber bundles that are touching, are attached to or partially concealed by a single particle or connected group of nonfibrous particles.

3.1.10.1 *Discussion*—The exposed fiber shall meet the fiber definition (*see* fiber (TEM)).

3.1.11 *point count, n*—quantitative regimen with definitions that can be found under EPA 600 R-93/116. A technique used to determine the relative projected areas occupied by separate components in a microscope slide preparation of a sample. For asbestos analysis, this technique is used to determine the relative concentrations of asbestos minerals to non-asbestos sample components.

3.1.12 *soil, n*—for this test method, soil is considered material of variable particle size and composition generally less than 19 mm in size.

3.1.12.1 *Discussion*—Examples may include loosely consolidated sediments, building materials, and other accumulated materials at the surface. Other materials larger than 19 mm may also be submitted at the collector's discretion as separate bulk samples.

3.1.13 *structures (TEM), n*—term that is used to categorize all the types of asbestos particles which are recorded during the analysis (such as fibers, bundles, clusters, and matrices).

3.1.14 *visual area estimate, VAE, n*—quantitative estimate of the amount of asbestos present most readily obtained by visual comparison of the bulk sample and slide preparations to other slide preparations and bulk samples with known amounts of asbestos present in them.

3.1.14.1 *Discussion*—Given that soils are typically

heterogeneous, sieving the soil helps to achieve similar particle size and facilitates subsequent VAE on the three sieved fractions.

3.2 *Descriptions for TEM Analysis Using Test Method D6281:*

3.2.1 *asbestos fiber or bundle longer than 5 µm, n*—any asbestos fiber or any width, bundle, or such fibers that has a length exceeding 5 µm.

3.2.2 *asbestos structure larger than 5 µm, n*—any fiber, bundle, cluster, or matrix for which the largest dimension exceeds 5 µm; does not necessarily contain asbestos fibers or bundles longer than 5 µm.

3.2.3 *compact matrix (Type C), n*—structure consisting of a particle or linked group of particles in which fibers or bundles can be seen either within the structure or projecting from it, such that the dimensions of individual fibers and bundles cannot be unambiguously determined.

3.2.4 *disperse matrix (Type D), n*—structure consisting of a particle or linked group of particles with overlapping or attached fibers or bundles in which at least one of the individual fibers or bundles can be separately identified and its dimensions measured.

3.2.4.1 *Discussion*—In practice, matrices can occur in which the characteristics of both types of matrix occur in the same structure. When this occurs, the structure should be assigned as a disperse matrix, and then a logical procedure should be followed by recording structure components according to the counting criteria.

3.2.5 *fibers that extend outside the field of view, n*—during scanning of a grid opening, count fibers that extend outside the field of view systematically so as to avoid double counting.

3.2.5.1 *Discussion*—In general, a rule should be established so that fibers extending outside the field of view in only two quadrants are counted. Measure the length of each of these fibers by moving the specimen to locate the other end of the fiber and then return to the original field of view before continuing to scan the specimen. Fibers without terminations within the field of view shall not be counted.

3.2.6 *other-structure-counting criteria, n*—Test Method D6281 structure-counting criteria may be used for TEM and PCM equivalent analysis of structures in the fine fraction.

3.2.7 *phase contrast microscope (PCM) equivalent fiber, n*—any particle with parallel or stepped sides with an aspect ratio of 3:1 or greater, longer than 5 µm that has a diameter between 0.2 and 3.0 µm (according to Test Method D6281).

3.2.7.1 *Discussion*—For chrysotile, PCM-equivalent fibers will always be bundles.

3.2.8 *PCM-equivalent structure, n*—any fiber, bundle, cluster, or matrix with an aspect ratio of 3:1 or greater, longer than 5 µm, that has a diameter between 0.2 and 3.0 µm.

3.2.8.1 *Discussion*—PCM-equivalent structures do not necessarily contain fibers or bundles longer than 5 µm or PCM-equivalent fibers.

3.2.8.2 *Discussion*—Record the dimensions of the structure such that the obscured portions of components are taken to be equivalent to the unobscured portions. For example, the length of a fiber intersecting a grid bar is taken to be twice the unobscured length. Structures intersecting either of the other two sides shall not be included in the count.

4. Summary of Test Method

4.1 The sample is dried and sieved with sieves arranged from top to bottom: 19 mm, 2 mm, 106 µm, and collection pan. The sieve fractions are designated coarse fraction (<19 to >2 mm), medium fraction (<2 mm to >106 µm), and fine fraction (<106 µm). Weights for each fraction are measured and recorded. During analysis, the >19-mm fraction may be analyzed using stereomicroscopy and polarized light microscopy (PLM) and reported separately but are not considered part of this method. The results are not included in the final result of the other three sieves fractions. Any building material debris collected from the field along with the soil sample may also be analyzed and reported separately. The coarse, medium, and fine fractions are all analyzed by stereomicroscopy and PLM visual area estimation (VAE). Initial results for the PLM analyses are expressed in calibrated visual area estimated percent and results for the fine fraction using point count values if below detection limit (see also 11.4.2-11.4.4). In addition, if PLM results indicate none detected, then the fine fraction of the sample may be analyzed for asbestos using transmission electron microscopy (TEM) drop mount as outlined in 11.6.1. If the TEM drop mount is negative or a quantitative result is desired, then it is recommended that the sample be gravimetrically reduced and visually estimated by TEM to derive a quantitative result expressed as an estimated weight percent.

4.2 *Optional TEM Analysis by Test Method D6281*—Additional analysis of the fine fraction may be performed to provide size data and concentration of asbestos reported as the number of asbestos structures per gram of sample.

4.3 The nominal quantity of soil sieved and analyzed is a 250-cm^3 sample. A larger amount (no more than 500 cm^3) may be required for different types of soil or other reasons determined by the laboratory and investigator. Any amount greater than 500 cm^3 will be discarded. The remainder of the sieved samples may be reserved for repeat additional testing or quality assurance testing. The laboratory shall assume that the investigator has ensured that the entire sample submitted is sufficiently homogeneous for his purposes.

5. Significance and Use

5.1 This analysis method is used for the testing of soil samples for asbestos. The emphasis is on detection and analysis of sieved particles for asbestos in the soil. Debris identifiable as bulk building material that is readily separable from the soil is to be analyzed and reported separately.

5.2 The coarse fraction of the sample (>2 to <19 mm) may contain large pieces of asbestos-containing material that may release fibers and break down during the sieving process into smaller pieces that pass through the 2-mm sieve into the medium fraction. If this alteration of the original sample is not desired by the investigator, these pieces should be removed from the sample before sieving and returned to the coarse fraction before analysis.

5.3 This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings or outdoor areas potentially contaminated with asbestos-containing materials or compliance with federal, state, or local regulations or statutes. It is the investigator's responsibility to make these determinations.

5.4 Whereas this test method produces results that may be used for evaluation of sites contaminated by construction, mine, and manufacturing wastes; deposits of natural occurrences of asbestos; and other sources of interest to the investigator, the application of the results to such evaluations and the conclusions drawn there from, including any assessment of risk or liability, is beyond the scope of this test method and is the responsibility of the investigator.

6. Interferences

6.1 The following minerals have properties (that is, chemical or crystalline structure) that are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials shall be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals. If this test method is used for the determination of the presence of nonregulated fibrous minerals, the following interferences may not apply:

6.1.1 Antigorite, picrolite;
6.1.2 Palygorskite (attapulgite);
6.1.3 Halloysite;
6.1.4 Pyroxenes;
6.1.5 Sepiolite;
6.1.6 Vermiculite scrolls;
6.1.7 Fibrous talc;
6.1.8 Hornblende and other amphiboles;
6.1.9 Other clays such as chlorite associated with talc deposits;
6.1.10 Scrolled minerals (lizardite); and
6.1.11 Non-asbestiform analogues of those listed in the first Discussion of 3.1.2.

7. Apparatus

7.1 In this section, equipment used for preparation and analysis of the samples in the laboratory is described. Materials and equipment used for sample collection are described in 11.2.

7.2 *Analytical Balance*—Balances or scales used in testing medium and coarse aggregate shall have readability and accuracy to two decimal places (0.01 g). For the fine fraction, an analytical balance with sensitivity to four decimal places (0.0001 g) shall be used.

7.3 *Sieves*—The sieve meshes and standard sieve frames shall conform to the requirements of Specification E11 (7.6- or 20–cm diameter); ASTM type; ¾ in. (ISO 19 mm), No. 10 (2 mm), No. 140 (106 µm), and collection pan (with drain outlet when using the wet sieve procedure).

7.4 *Mechanical Sieve Shaker*—A mechanical sieving device capable of creating motion of the sieves to cause the particles to bounce, tumble, or otherwise turn so as to present different orientations to the sieving surface. More information on sieving can be found in Test Method C136.

7.5 *Laboratory Oven or Equivalent*—An oven of appropriate size capable of maintaining a uniform temperature of 110 ± 5°C.

7.6 *TEM,* 80- to 120-kV, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM shall be equipped with an energy dispersive X-ray spectrometer (EDXA), and it shall have a scanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover.

7.7 *EDXA*—The EDXA system (detector and multichannel analyzer), under routine analysis conditions, meets the following specifications: <175 eV or better resolution at Mn Kα peak, proven detection of Na peak in standard crocidolite or equivalent, capable of obtaining statistically significant Mg and Si peaks from a single fibril of chrysotile, and consistent relative sensitivity factors over large areas of the specimen grid.

7.8 *High-Vacuum Carbon Evaporator,* with rotating stage.

7.9 *Exhaust or Fume Hood,* capable of 25-linear m/min (80-fpm) flow rate.

7.10 *Stereo Microscope,* approximately 10 to 45×, with light source.

7.11 *Side-Arm Filter Flask,* 1000 mL.

7.12 *Cabinet-Type Desiccator,* or low-temperature drying oven.

7.13 *Scintillation Tube,* or equivalent.

7.14 *Vacuum Pump,* which can maintain a pressure of 92 kPa.

7.15 *PLM,* binocular or monocular with crosshair reticule (or functional equivalent); low (≥5 and ≤15×), medium (>15 and <40×), and high (≥40×) objectives; light source; 360° rotatable stage; substage condenser with iris diaphragm, polarizer, and analyzer that can be placed at 90° to each other; accessory slot at 45° to polarizers for wave plates and compensators; wave retardation plate (~550-nm retardation); dispersion-staining objective complete with accessories (optional); and test slide (or a standard such as NIST SRM 1867/anthophyllite) for aligning the crosshairs with the privileged directions of the polarizer and analyzer.

7.16 *Ultrasonic Bath,* tabletop model (100 W).

7.17 *Plastic Sample Containers,* with wide-mouth screw cap (500 mL) or equivalent sealable container.

7.18 *Waterproof Markers.*

7.19 *Forceps (Tweezers).*

7.20 *Carbon-Coated Finder Grids (Filter Substrate),* 200 mesh.

7.21 *Graduated Pipets (1-, 5-, or 10-mL Sizes),* glass or plastic.

7.22 *Filter Funnel Assemblies,* either glass or disposable plastic and using either a 25- or 47- mm diameter filter.

7.23 *Mixed Cellulose Ester (MCE) Membrane Filters,* 25- or 47-mm diameter, 0.22- and 5- µm pore size.

7.24 *Polycarbonate (PC) Filters,* 25- or 47-mm diameter, 0.2-µm pore size.

7.25 *Storage Containers,* for the 25- or 47-mm filters (for archiving).

7.26 *Glass Slides,* approximately 76 by 25 mm in size.

7.27 *Scalpel Blades,* No. 10 or equivalent.

7.28 *Cover Slips,* 18 by 18 mm.

7.29 *Nonasbestos Mineral,* references as outlined in 6.1.

7.30 *Asbestos Standards,* National Institute of Standards and Technology (NIST) traceable as outlined in 3.1.2 if available or documented reference materials.

7.31 *Petri Dishes,* large glass, approximately 90 mm in diameter.

7.32 *Jaffe Washer,* stainless steel or aluminum mesh screen, 30 to 40 mesh, approximately 75 by 50 mm.

7.33 *Carbon Rods,* for evaporation of carbon film onto samples.

7.34 *Lens Tissue.*

7.35 *Ashless Filter Paper Filters,* 90-mm diameter.

7.36 *Wash Bottles,* plastic (100 mL suggested).

7.37 *Reagent Alcohol,* high-performance liquid chromatography (HPLC) grade (Fisher A995 or equivalent).

7.38 *Diffraction Grating Replica,* 2160 lines/mm.

7.39 *Disposable Aluminum Pans.*

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society[7] where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D1193.

9. Sampling

9.1 Sample collection is the responsibility of the field investigator. For a discussion of sample collection, see nonmandatory Appendix X2.

[7] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

10. Calibration

10.1 Perform calibrations of the instrumentation on a regular basis and retain these records in the laboratory in accordance with the laboratory's quality assurance program.

10.2 Record calibrations in a log book or laboratory information management system (LIMS) along with dates of calibration and the attached backup documentation.

10.3 *PLM Calibration:*

10.3.1 The laboratory shall ensure that each microscope is in proper working condition. The optical system, including objectives, condensers, polarizers, and so forth, shall not be damaged or modified in any way that would affect microscope resolution or depolarize the light (that is, the lens is relatively free of scratches, nicks, corrosion, signs of impact, and so forth and there is no stop in the back focal plane other than for dispersion-staining objectives).

10.3.2 The laboratory shall have written procedures for aligning the PLM daily (or before use) in such a way that:

10.3.2.1 The privileged directions of the substage polarizer and the analyzer shall be oriented at 90° to one another. The orientations of the privileged direction of the polarizers shall be known. The accessory slot shall be at 45° to these privileged directions;

10.3.2.2 The ocular crosshairs coincide with the privileged directions of the polarizer and the analyzer and this condition shall be verified with a test slide (or similar standard);

10.3.2.3 The objectives or stage or both shall be centered to prevent grains from leaving the fields of view during stage rotation;

10.3.2.4 The substage condenser, which is visualized through the image of the field diaphragm, shall be centered on the optic axis; and

10.3.2.5 An alignment check before use shall be performed and recorded.

10.3.3 The laboratory shall have calibrated refractive index solids, or a refractometer (or access to one), for calibrating refractive index liquids.

10.3.4 The laboratory shall have written procedures for calibrating refractive index (RI) liquids, including the lot number for each of the measured oils, to determine whether their actual or calibrated RI value at 589 nm and 25°C, are within ±0.004 of their nominal values. The procedures shall include:

10.3.4.1 If the calibrated RI value at 589 nm and 25°C deviates more than ±0.004 from the nominal value, the liquid shall not be used.

10.3.4.2 The temperature at the workstation at the time of calibration shall be recorded and, if not 25°C, used to perform temperature correction of the calibrated RI value.

10.4 *TEM Calibrations:*

10.4.1 Check the alignment and the systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

10.4.2 Calibrate the camera length of the TEM in electron diffraction (ED) operating mode before ED patterns of unknown samples are observed.

10.4.3 Perform magnification calibration at the fluorescent screen. This calibration shall be performed at the magnification used for structure counting. Calibration is performed with a grating replica (for example, one containing 2160 lines/mm).

10.4.3.1 Define a field of view on the fluorescent screen. The field of view shall be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

10.4.3.2 Frequency of calibration will depend on the service history of the particular microscope.

10.4.3.3 Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange.

10.4.3.4 The analyst shall ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

10.4.3.5 For instruments that incorporate a eucentric tilting specimen stage, all specimens and the grating replica shall be placed at the eucentric position.

10.4.4 The smallest spot size of the TEM shall be checked.

10.4.4.1 At the crossover point, measure the spot size at a screen magnification of 15 000 to 20 000×.

10.4.4.2 The measured spot size shall be less than or equal to 250 nm.

10.5 *EDXA Calibration:*

10.5.1 The resolution and calibration of the EDXA shall be verified.

10.5.2 Collect a standard EDXA Cu peak and Al peak from a Cu grid with evaporated aluminum or equivalent.

10.5.3 Compare the X-ray energy versus channel number for the Cu peak and theAl peak. Be certain that readings are within ±10 eV.

10.5.4 Select a single fiber of crocidolite with a width less than 1 µm (NIST 1866 or equivalent) and collect an EDXA spectrum from it.

10.5.5 The elemental analysis of the crocidolite shall meet the following condition in which the Na peak is considered statistically significant and not a fluctuation in the background if the number of net counts, N_{net}, exceeds twice of the standard deviation of net counts,$_{net}$, which equals to the square root of (two times the average background counts, N_B, plus the average net counts, N_{net}):

$$N_{net} \geq 2\sigma_{net} \quad (1)$$

where:

$$\sigma_{net} = \sqrt{2N_B + N_{net}} \quad (2)$$

10.5.6 Collect a standard EDXA of chrysotile asbestos (NIST SRM 1866 or equivalent).

10.5.7 The elemental analysis of chrysotile shall determine that the Si and Mg peaks are statistically significant similar to the procedure outlined in 10.4.4 on a single chrysotile fiber with width less than 1 µm.

10.6 *Grid Opening Measurements*—TEM grids shall have a known grid opening area. Determine this area for a lot of TEM grids as follows:

10.6.1 Measure at least 20 grid openings in each of 20 random (200-mesh) copper grids for a total of 400 grid openings for every 1000 grids used by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

10.6.2 Grid area measurements can also be made at the TEM at a calibrated screen magnification. Typically, measure one grid opening for each grid examined. Measure grid openings in both the *x* and *y* directions and calculate the area.

10.6.3 Pre-calibrated TEM grids are also acceptable for this test method.

11. Procedure

11.1 *Sample Preparation*—Dry Sieving (for Wet Sieving, see Appendix X1).

11.1.1 Any building materials collected at the site are analyzed separately by stereomicroscopy and PLM and reported separately. Each soil sample or representative subsample thereof will be dried at the laboratory within 48 h of receipt (recommend prompt shipment after collection to minimize microbial growth) in an oven at 110 ± 5°C until the weight is stable. Record the change in weight. Ensure that sample loss before and after sieving meets requirements set forth in Test Method C136 by weighing before and after sieving. Change in weight should be recorded for moisture content.

11.1.2 For samples with organic or soluble materials, gravimetric reduction of the sample may be performed before sieving using EPA 600/R-93/116.

11.2 Under a hood (high-efficiency particulate air [HEPA] filtered if required), nest the sieves in order of decreasing size of opening from top to bottom on the sieve shaker with the 19-mm sieve on top, 2-mm sieve (coarse), the 106-µm sieve in the middle (medium), and the collection pan on the bottom (fine)

FIG. 1 Configuration of 20-cm Diameter Sieves on the Sieve Shaker