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

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[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

as shown in Fig. 1. The dried sample is poured into the 19-mm sieve and misted lightly with isopropyl alcohol to neutralize static charges and minimize liberation of particles when the sieve is activated. Proper precautions should be used when starting the sieve shaker when isopropyl alcohol is used. Use of a fume hood with a HEPA filter is the responsibility of the laboratory. ASTM International does not specify local environmental policy.

11.2.1 Secure the sieve shaker top on the sieve as shown in Fig. 1. A lid is placed on the 19-mm (top) sieve before sieving.

11.2.2 Turn on the sieve shaker for 5 min or until the fractions are reasonably separated. When finished, wait at least 5 min before dismantling the sieves to allow particles to settle in each sieve.

11.2.3 Carefully separate each sieve to minimize disturbance of particles. Determine the weight for each of the three remaining sieve fractions. Each fraction may be placed in a tared sealable container.

11.2.4 Between each sample, clean sieves thoroughly by brushing in hot soapy water, rinsing thoroughly, sonicating in a submerged bath for 10 min, and following up with a final rinse and drying. Inspect screen cloths after cleaning for damage.

11.3 *Blanks*—Blanks should be added before each sample sieved to check for contamination. Blank material can consist of asbestos-free materials such as Ottawa sand or known asbestos-free soil.

11.4 The coarse (>2-mm), medium (<2-mm and >106-µm), and fine (<106-µm) fractions are analyzed by stereomicroscopy and PLM using calibrated visual area estimation and identification consistent with EPA 600/R-93/116.

11.4.1 *>19-mm Fraction*—The >19-mm fraction is not considered soil and may be analyzed separately by stereomicroscopy and PLM; the results are not combined or reported with the other three fractions and can be reported separately outside of this method.

11.4.2 *Coarse Fraction (ISO 2 mm)*—Observation by stereomicroscopy and PLM is used to confirm asbestos and quantify based on calibrated visual area estimation. If fibers are observed in matrices or as isolated material, they are teased or extracted and confirmed by PLM. If a single asbestos structure is observed, a value of 0.25 % is used for nominal sensitivity.

11.4.3 *Medium Fraction (ISO 106 µm)*—Observation by stereomicroscopy and PLM is used to confirm asbestos and quantify based on calibrated visual area estimation. If fibers are observed in matrices or as isolated material, they are teased or extracted and confirmed by PLM. If a single asbestos structure is observed, a value of 0.25 % is used for nominal sensitivity.

11.4.4 *Fine Fraction (ISO 106 µm)*—Observation by stereomicroscopy, PLM, is used to confirm if asbestos is present. If fibers are observed in matrices or as isolated material, they are teased or extracted and confirmed by PLM. Recommended additional analysis by TEM is detailed in 11.6.1. If agglomerations of fibrous-containing material are observed by stereomicroscopy, PLM is used to estimate the relative percentage by calibrated visual area estimation or point counting or both.

11.4.5 Any building materials collected at the site are analyzed separately by stereomicroscopy and PLM and reported separately.

11.5 *Point Count*—If asbestos is detected at <1 % in the fine fraction, then perform a point count by preparing eight separate slide mounts and examine at 100× following EPA 600/R-93/116 until 400 points are counted. Additional points may be counted to lower the analytical sensitivity.

11.6 *Optional TEM Analysis of Fine Fraction (Recommended if PLM Results Negative):*

11.6.1 *TEM Drop Mount Screening:*

11.6.1.1 In a scintillation vial or equivalent, approximately 20 to 100 mg of the material is suspended in 2 to 5 mL of ethanol or other alcohol. The suspension is ultrasonicated for 3 min. Shake the suspension and immediately extract a 3-µL aliquot and drop mount it onto a carboncoated grid and allow to dry. Repeat the process for a second aliquot and deposit onto another grid (3-mm diameter, 200 mesh). The drop should remain intact and not leave the grid surface while drying.

11.6.1.2 The sample grid is examined using TEM. At least five openings over two grids are examined on the grid at high magnification (15 000–20 000×). Identify asbestos structures and type using morphology, SAED, and EDXA.

11.6.2 *TEM Gravimetric Reduction and Filter Preparation (Recommended for Quantitative Analysis; Required if Drop Mount Negative):*

11.6.2.1 Approximately 100 to 250 mg of the material from the fine fraction is weighed and gravimetrically reduced as follows:

(1) Place the sample into a tared crucible or vial and record weight. Place an aluminum lid on the vial containing the sample and place it in a muffle furnace with the temperature held accurately at 480 ± 5°C. Leave in the furnace for at least 1 to 12 h or until the weight stabilizes. Remove the vial from the furnace and allow to cool to room temperature. Remove the vial lid and weigh the vial and residual ash. Since some components of the residual ash may be hygroscopic, this weighing shall be done before the ash absorbs moisture from the air (within 3 to 5 min after cooling unless the sample is placed in a desiccator).

(2) Set up a 25- or 47-mm filtration assembly using a vacuum flask and water aspirator. Weigh and install the MCE 0.22-µm-size filter or 0.4 polycarbonate filter on top of the 5-µm backing filter. Add approximately 0.5 mL of ultra-pure water to the crucible with the ashed residue and grind the material to disperse it.

(3) Slowly add approximately 2 mL of concentrated hydrochloric acid. Calcite and dolomite will dissolve with the evolution of CO_2. After 15 min, dilute the suspension with more ultra-pure water and pour into a filtration apparatus with the 0.22-µm-size filter or 0.4 polycarbonate filter. During filtration, estimate the particle loading of approximately 20 % on the filter using a stereomicroscope at 10 to 100×.

(4) Dry filters and prepare for TEM examination using a direct method consistent with Test Method D6281.

(5) Analysis follows Test Method D6281 and, in general, choose the prepared filter that contains 5 to 25 % particle loading when examined by TEM at 1000×. Continue the count

until completion of the grid opening on which 100 fibers and bundles have been recorded or sufficient area of the specimen has been examined to achieve the desired analytical sensitivity.

(6) If negative, then ten grid openings are examined at 20 000×. Record the structures that contain asbestos fibers meeting 5:1 length/width aspect ratio greater than 0.5 µm in length. Structures are identified using morphology, SAED, and EDXA.

12. Sample Storage

12.1 Sample retention is determined by the laboratory and client.

13. Calculations

13.1 *PLM Analysis:*

13.1.1 Total calculated asbestos content of the soil [the coarse (>2-mm), medium (<2-mm and >106-µm), and fine (<106-µm) fractions] in the soil sample using PLM analysis is determined using:

$$\text{Total Asbestos (\%)} = \frac{[\%_F \quad \text{PLM PC} * W_F] + [\%_M \quad \text{PLM} * W_M] + [\%_C \quad \text{PLM} * W_C]}{W_F + W_M + W_C} \quad (3)$$

where:

$\%_F$ = Percentage of asbestos in the fine fraction determined by PLM point count;
$\%_M$ = Percentage of asbestos in the medium fraction determined by PLM VAE;
$\%_C$ = Percentage of asbestos in the coarse fraction determined by stereomicroscope and PLM, VAE;
W_F = Weight of fine fraction of sample;
W_M = Weight of medium fraction of sample; and
W_C = Weight of coarse fraction of sample.

13.1.1.1 *Analysis of the >19-mm Fraction*—The >19-mm fraction is reported separately and not combined with the other three fractions.

13.1.1.2 *Building Debris*—Building debris is reported separately and not combined with the other analyses.

13.1.2 *TEM Analysis:*

13.1.2.1 *TEM Results of Optional Drop Mount*—The drop mount results are expressed as nondetected or detected with asbestos type(s) identified. If asbestos is observed, the gravimetric reduction and TEM analysis by visual estimation shall be performed.

13.1.2.2 *TEM Results of the Fine Fraction where asbestos structures are counted*—The sensitivity of the analysis for this portion of the test method may be calculated using the following formula. The minimum area analyzed should be no less than 0.2 mm^2.

$$\text{Sensitivity} = \frac{\text{EFA}}{\text{Area Analyzed}} \times \frac{\text{SV}}{\text{FV}} \times \frac{1 \text{ Str}}{\text{SS}} = \text{str}/\mu\text{g} \quad (4)$$

where:

EFA = effective filter area in square millimetres,
G.O. = grid openings,
AA (area analyzed) = number of G.O. × grid opening area in square millimetres,
SV (suspension volume) = original suspended volume of material in millilitres,
FV (filtered volume) = aliquot filtered in millilitres, and
SS (subsample) = will be quantitatively weighed from a fraction of the sieved fines. This result will be recorded in micrograms.

13.1.2.3 For purposes of demonstration/discussion:

(1) Grid opening size = 0.0104 mm^2. (Note that this may vary depending on the grids used).

(2) Grid openings counted = 20. (Note that this may change to achieve the 0.2-mm^2 sensitivity level required by the analysis).

(3) Effective filter area (EFA) of 47-mm filter = 1320 mm^2. (Note that this may vary depending on the funnel system used).

(4) Fibers counted = 2 (chrysotile).

- A subsample will be quantitatively weighed from a fraction of the sieved fines. This result will be recorded in micrograms. For example, 248 mg = 248 000 µg of fine fraction.
- This 248 000-µg amount is suspended in 100 mL of deionized water and subsequent aliquots (for example, 10 mL) are filtered. For the sake of this example, assume that two fibers were counted during TEM examination.
- Resulting equation to get structures/µg (str/µg):

$$\text{Sensitivity} = \frac{1320 \text{ mm}^2}{(20 \text{ G.O.} \times 0.0104 \text{ mm}^2)} \times \frac{100 \text{ mL}}{10 \text{ mL}} \times \frac{1 \text{ Str}}{248\,000 \ \mu\text{g}} = 0.2538 \text{ str}/\mu\text{g} \quad (5)$$

- Structures/µg = 2 fibers × 0.2538 = 0.5076 structures/µg.

13.1.2.4 *TEM Results of the Fine Fraction in which Asbestos is Visually Estimated*—The minimum area analyzed should be no less than 0.2 mm^2. Results can be expressed as a weight percent in which the visual estimate of asbestos on the TEM is multiplied by the remaining weight percent of the sample prepared.

14. Report

14.1 *PLM Reporting*—Report the following information for each soil sample analyzed:

14.1.1 Concentration of asbestos in each sieved fraction and total sample asbestos concentration determined by PLM in percent (%);

14.1.2 Analytical sensitivity of each sieved fraction in weight % in which analytical sensitivity is determined by multiplying the weight of fine fraction by the single-fiber PLM detection limit of 0.25 %.

14.1.3 Type(s) of asbestos present.

14.2 *TEM Drop Mount Reporting*—Report the drop mount sample analysis as non-detected or detected for asbestos. If detected, report the sample type(s) present. Other fibrous material may be reported if relevant.

14.3 *TEM Filter Analysis Reporting*—Report the following information for each soil sample analyzed:

14.3.1 Number of asbestos structures counted and length and width of each structure (if counted), 14.3.2 Effective filtration area,

14.3.3 Size of the TEM grid openings,

14.3.4 Number of grid openings examined,

14.3.5 Weight of subsample used for TEM (micrograms),

14.3.6 Magnification(s) used for TEM analysis,

14.3.7 Amount of original suspension (millilitres) and amount filtered/analyzed (millilitres),

14.3.8 Type(s) of asbestos present,

14.3.9 Total structures per microgram of sample,

14.3.10 Dry or wet sieving for preparation of samples will be noted on the report, and

14.3.11 Notation on final report will express the percent loss of the total sample from sieving.

NOTE 1—Reporting of the >19-mm fraction and building debris are conducted separately and are not considered part of this reporting.

15. Quality Assurance

15.1 In general, the laboratory's quality control checks are used to verify that a system is performing according to specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. However, because of the difficulties in preparing known quantitative asbestos samples, routine quality control testing focuses on reanalysis of samples. Laboratory process blanks should be incorporated into the quality program to ensure sieves are asbestos free.

15.2 In addition, quality assurance programs shall follow ISO 17025 guidelines and Test Method D6281 for TEM, where applicable.

15.3 Analysis should be supplemented by intralaboratory and interlaboratory replicate analysis and repeat analyses of numerous subsamples from the original soil sample.

15.4 Reanalyze samples at a rate of 1/10 of the sample sets (one out of every ten samples analyzed not including laboratory blanks). The reanalysis shall consist of a second sample preparation of sieved fractions.

16. Precision and Bias

16.1 *Precision*—The precision of this test method is based on an interlaboratory study conducted to support this test method, conducted in 2014. Nine laboratories participated in this study. Each of the nine labs reported three replicates of a single chrysotile material, being tested by PLM, PLM Point Count, and TEM. Every "test result" reported represents an individual determination. Except for the use of only a single material, Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. D22-1036.[8]

16.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

16.1.1.1 Repeatability can be interpreted as the maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

16.1.1.2 Repeatability limits are listed in Tables 2-4.

16.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

16.1.2.1 Reproducibility can be interpreted as the maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

16.1.2.2 Reproducibility limits are listed in Tables 2-4.

16.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

16.1.4 Any judgment in accordance with statements 16.1.1 and 16.1.2 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of materials tested and laboratories reporting results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. The repeatability limit and the reproducibility limit should be considered as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected.

16.2 *Bias*—Bias was determined using a reference material formulated by RTI International with 0.5 % NIST SRM chrysotile asbestos mixed by gravimetric addition in North Carolina sandy loam. The material was homogenized using

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D22-1036. Contact ASTM Customer Service at service@astm.org.

TABLE 2 Total Percentage by Visual Estimation (Optical Microscopy including Stereomicroscopy and PLM)

Material (% in original sample)	Average,[A] $\bar{x}$	Average Reported Recovery, %	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	Reproducibility Limit, R
0.5 % Chrysotile in Soil	5.30	1060	1.75	4.11	4.89	11.52

[A] The average of the laboratories' calculated averages.

TABLE 3 Total Percentage by PLM Point Count

Material	Average,[A] $\bar{x}$	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	ReproducibilityLimit, R
0.5 % Chrysotile in Soil	0.83	0.36	0.59	1.01	1.66

[A] The average of the laboratories' calculated averages.

TABLE 4 Total Percentage by TEM Drop Mount

Material	Average,[A] $\bar{x}$	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	ReproducibilityLimit, R
0.5 % Chrysotile in Soil	1.50	0.56	1.31	1.57	3.68

[A] The average of the laboratories' calculated averages.

blending and Turbula mixing, and then split for individual lab packaging using a spinning riffler. Individual subsamples were checked by RTI to confirm uniformity of fiber distribution in the 27-batch set of samples prior to shipment. Average percent recoveries are reported in Table 2.

NOTE 2—The chrysotile used in this study is generally representative of long fiber chrysotile used in commerce, however, as recognized in Test Method D7521 – 13, chrysotile from some sources may have different characteristics.

16.3 The precision statement was determined through statistical examination of 81 results, from nine laboratories, on one material.

17. Keywords

17.1 asbestos; PLM; polarized light microscopy; sieves; soil; TEM; transmission electron microscopy

APPENDIXES

(Nonmandatory Information)

X1. SAMPLE PREPARATION—WET SIEVING

X1.1 The sample shall be dried in a laboratory drying oven at no more than 110°C until the sample stabilizes to a constant weight.

X1.2 After sample is properly dried, measure and record the sample weight to 0.1 g.

X1.3 Assemble a sieve stack from top to bottom as follows: ASTM 19-mm sieve, ASTM 2-mm sieve, ASTM 106-µm sieve, and collection pan with drain outlet (see Fig. X1.1). A clean receiving container (2 L or larger is recommended) is placed below the sieve stack to receive all water passing through the sieves. This will receive the fine fraction that passes through the 106-µm sieve.

X1.4 In a HEPA-filtered hood, transfer the dried sample onto the top (19-mm) sieve in the assembled sieve stack.

FIG. X1.1 Assemble a Sieve Stack from Top to Bottom as follows: ASTM 19-mm Sieve, ASTM 2-mm Sieve, ASTM 106-µm Sieve, and Collection Pan with Drain Outlet

TABLE X1.1 Examples of Sieve Sizes

Opening/Designation			Brass Frame Brass Cloth		Brass Frame Stainless Cloth		Stainless Frame Stainless Cloth	
ISO	ASTM	Tyler	Full Ht. Part No.	Half Ht. Part No.	Full Ht. Part No.	Half Ht. Part No.	Full Ht. Part No.	Half Ht. Part No.
19.0 mm	¾ in.	0.742-in opg	---	---	4847	5013	5179	---
2 mm	No. 10	9 mesh	4530	4696	4862	5028	5194	5360
106 µm	No. 140	150 mesh	4547	47	4879	5045	5211	5377

FIG. X1.2 Gently Wet the Sample with a Mist of Particle-Free Water

FIG. X1.3 Gently Wash the Course Fraction with a Gentle Stream of Particle-Free Deionized Water

FIG. X1.4 Repeat Steps X1.8 and X1.9 for the >2-mm and >106-µm Fractions

FIG. X1.5 Repeat Steps X1.8 and X1.9 for the >2-mm and >106-µm Fractions

X1.5 Gently wet the sample with a mist of particle-free water from a spray bottle to minimize the chance for particle release during transfer to a lab sink (see Fig. X1.2).

X1.6 Set up the sieve stack on a tripod stand or similar so that the water that passes through the sieves is captured in the receiving container.

X1.7 Place a clean, 2-L beaker or other wide-mouth, straight-walled container under the drain outlet of the receiving pan.

X1.8 Gently wash the coarse fraction with a gentle stream of particle-free deionized water (see Fig. X1.3).

X1.8.1 Use the water sparingly so as not to overflow the 2-L receiving container. If necessary, however, the 2-L beaker can be emptied into another larger straight-walled container for consolidation at the end of the process.

X1.8.2 While wetting the sample, use a gloved hand to "work" the sample. This helps to loosen the soil, break up clumps, and facilitate the separation of the coarse fraction from the rest of the sample.

X1.9 Once the coarse fraction has been completely washed and no more material is visibly passing through the top sieve, carefully remove the top sieve and set aside. Note that this fraction is very often nonexistent (all material <19 mm).

X1.10 Repeat X1.8 and X1.9 for the >2-mm and >106-µm fractions (see Fig. X1.4 and Fig. X1.5).

X1.11 Once all sieve fractions have been separated, the material from each sieve can be transferred into separate, preweighed, disposable aluminum pans for drying. Disposable wooden tongue depressors are useful tools for this transfer.

NOTE X1.1—Alternatively, the samples can be dried in their respective sieves and then transferred for weighing or even weighed right in the sieves provided they have been preweighed empty.

FIG. X1.6 Fine Fraction (<106 µm) that is in Suspension in the 2-L (or Larger) Straight-Walled Container is Covered and Set Aside for at Least 12 h to Allow the Particulate to Settle Out

X1.12 Any material that remains on the 19-mm sieve (see Fig. X1.3) is not considered part of the sample and can optionally be analyzed separately.

X1.13 Dry all fractions following the drying procedure in X1.11, then weigh each fraction and record in grams.

X1.14 The fine fraction (<106 µm) that is in suspension in the 2-L (or larger) straight-walled container is covered and set aside for at least 12 h to allow the particulate to settle out (see Fig. X1.6).

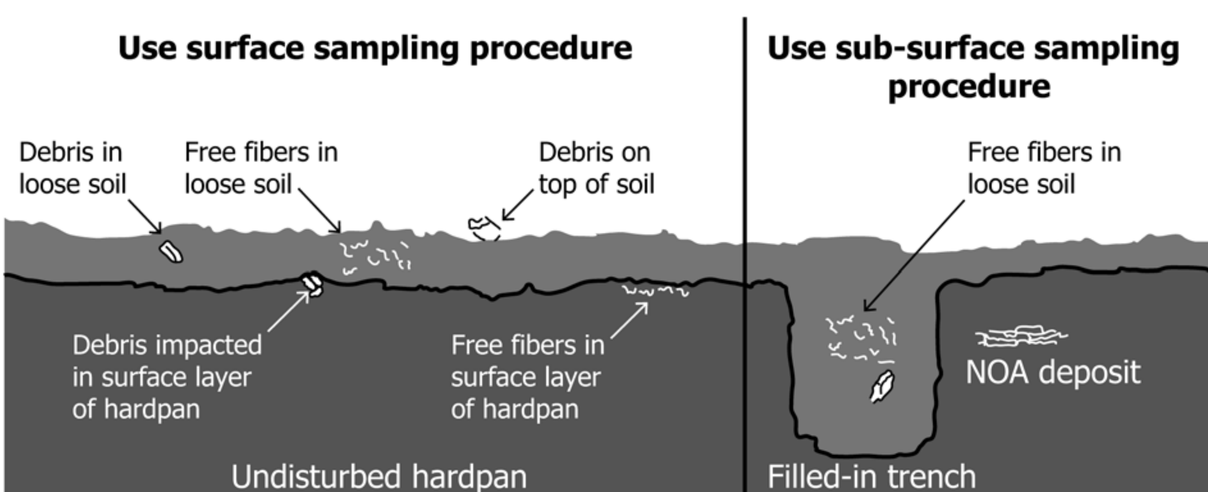
X1.15 The supernatant can then be decanted or aspirated off to be discarded or optionally filtered and analyzed by TEM as the "super fine" fraction.

X2. SAMPLE COLLECTION

X2.1 A representative portion of the soil sample material is collected in a sealable and nonbreakable container. Soil samples shall be collected in a manner that meets the requirements of this test method and the needs of the investigator for whom the soil is being analyzed. It is the responsibility of the investigator to determine the necessary homogeneity of the samples and to ensure that the homogeneity of each sample collected meets this requirement.

X2.2 This appendix describes surface sampling from a relatively flat horizontal surface and subsurface (core) sampling. See Fig. X2.1. It is the responsibility of the investigator to determine which sampling procedure to use and also whether conditions exist at the site requiring appropriate modifications to be made to either procedure.

X2.3 These collection methods are applicable for soils in which the source of asbestos fibers is unknown, external, or considered natural occurrences of asbestos (NOA), or combination thereof, in which the asbestos fibers originated in the soil. Examples of the former include debris from disposal of building materials and mine waste tailings. The latter may include veins of asbestos in construction excavations, deposits of tailings concentrated by runoff, and detritus caused by weathering of rock and soil and other natural occurrences or deposits from quarries or mining activities. The methods are



NOTE 1—Adapted from *Manual on Asbestos Control: Surveys, Removal and Management*: A. Oberta, *Manual on Asbestos Control: Surveys, Removal and Management*, second edition, ASTM International, West Conshohocken, PA, March 2005.

FIG. X2.1 Section through Loose Soil, Hardpan, and Trench

intended to collect soil containing free fibers as well as discrete pieces of building material debris and pieces of NOA in the soil.

X2.4 Sample Collection Equipment

X2.4.1 *Sample Containers*—Rigid, sealable, non-breakable containers should be of sufficient size to hold up to 500-cm^3 of dry soil.

X2.4.2 *Scoop*—the scoop should be a rigid metal or plastic scoop with a sharp edge. For sampling the surface of hardpan, use a metal scoop with a pointed tip capable of scratching the surface.

X2.4.3 *Spray Bottle*—A plastic bottle filled with soapy water with the nozzle set to spray a fine mist.

X2.4.4 *Tape*—To seal sample container.

X2.4.5 *Soil Auger*—For collection of subsurface samples.

X2.5 Surface Sample Collection Procedure

X2.5.1 Loose soil (see X2.5.4) or soil from the top surface of intact undisturbed hardpan (see X2.5.5) may be collected.

X2.5.2 *Sample Area Selection*—If possible, select an area for sampling that is representative of the type of asbestos material or contamination event or both under investigation, such as abandoned or discarded pipe insulation. If there is visual evidence of potentially asbestos-containing material in the form of debris from building material or free fibers from mine or manufacturing wastes, note its presence. However, as the objective is collection of the contaminated soil and not visible debris or waste, select an area in which these objects are not abundant. Select an area that is free of contamination by solvents, oils, lead paint, grease, or other hazardous or interfering substances.

X2.5.3 *Sample Area Preparation and Precleaning*—Secure the area against unauthorized entry and inadvertent disturbance. If pieces of debris larger than 19 mm (see 5.2) that may or may not be identifiable from sources such as pipe insulation, asbestos-cement pipe, and mine or manufacturing waste are present in the sampling area, remove them in a manner that does not release fibers into the soil and cause additional contamination of the soil. Pieces of nonfriable debris should be lightly misted with soapy water and picked out of the soil inside the template. Pieces of friable debris or waste should be lightly misted with soapy water and removed by scooping them up with the underlying loose soil or hardpan, not by trying to pick the debris or waste out of the soil or hardpan. The pieces of each type of debris or waste should be put in sample containers for analysis as bulk samples separately from the soil samples. Friable debris should be placed in rigid, nonbreakable sealable containers. During this precleaning, note any removal of rock, wood, concrete, metal, vegetation, and other obvious nonfibrous materials.

X2.5.4 *Collection of Loose Soil*—Lightly mist the surface of the ground with soapy water—do not saturate the soil. Scrape the edge of the scoop across the surface of the ground and pour the collected contents into the sample container. Repeat this process over the entire sampling area until the required amount of soil—at least 250 cm^3—has been collected. Close the container, wipe it with a moist paper towel, and seal it with tape. Mark the sample number and other information on the container, not on the lid or tape. See Fig. X2.2 for an example of collecting a loose soil sample.

X2.5.5 *Collection of Soil from Hardpan*—If loose soil has been removed down to the hardpan, examine the surface for visible debris from building materials or mine and manufacturing wastes. If these objects are present in the form of debris, remove them per the precleaning procedure in X2.5.3. Debris and fibers will not penetrate intact hardpan as readily as loose soil, so contamination may be limited to a shallow region near the surface. To avoid sampling undisturbed and potentially uncontaminated soil, a larger area will have to be sampled to collect the same sample volume. It may be necessary to remove loose soil to expose enough hardpan to collect the sample. Lightly mist the surface of the hardpan and use the pointed tip of the scoop to scratch the top and then collect the soil with the scoop as described in X2.5.4.

X2.6 Subsurface Sample Collection Procedure

X2.6.1 Debris or free fibers may migrate into porous soil deeper than into intact undisturbed hardpan or previous excavation may have buried debris and free fibers. The investigator may need to sample natural occurrences of asbestos below the surface in loose soil or undisturbed hardpan.

X2.6.2 *Sample Area Selection*—If possible, select an area for sampling that is representative of the type of asbestos material or contamination event or both under investigation.

X2.6.3 *Sample Area Preparation*—Secure the area against unauthorized entry and inadvertent disturbance. Determine the depth of loose soil, if any, and the depth at which the core sample(s) will be taken.

X2.6.4 *Sample Area Precleaning*—Perform precleaning as described in X2.5.3 as applicable to the area being sampled.

X2.6.5 *Collection of Subsurface Sample*—Position the soil auger in the area where free fibers and possibly building material debris or NOA may exist below the surface. Drive the auger through the loose soil, or into the hardpan, to the specified depth. More than one penetration of the soil may be required to reach the desired depth. Remove the auger from the

FIG. X2.2 Collection of Surface Sample: Loose Soil

soil, extract the sample, and place it in the sample container. If necessary and possible, maintain the integrity of any discrete layers in the sample.

X2.7 *Cleanup*—After collection of each sample, clean the scoop or soil auger with soapy water and paper towels, then dispose of the towels as asbestos-contaminated waste material.

X2.8 *Personal Protective Equipment (PPE)*—Because the soil will be collected in an essentially dry condition and pieces of asbestos-containing debris and waste may be handled, the use of PPE is mandatory. At a minimum, respiratory protection shall be worn. Disposable foot coverings may be required to avoid tracking contaminated soil into vehicles.

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