Designation: D5756 – 02 (Reapproved 2008)

Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Surface Loading[1]

This standard is issued under the fixed designation D5756; 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 (*a*) identify asbestos in dust and (*b*) provide an estimate of the surface loading of asbestos in the sampled dust, reported as either the mass of asbestos per unit area of sampled surface or as the mass of asbestos per mass of sampled dust.

1.1.1 If an estimate of asbestos structure counts is to be determined, the user is referred to Test Method D5755.

1.2 This test method describes the equipment and procedures necessary for sampling, by a microvacuum technique, non-airborne dust for levels of asbestos. The non-airborne sample is collected inside a standard filter membrane cassette from the sampling of a surface area for dust which may contain asbestos.

1.2.1 This procedure uses a microvacuuming sampling technique. The collection efficiency of this technique is unknown. Variability of collection efficiency for any particular substrate and across different types of substrates is also unknown. The effects of sampling efficiency differences and variability on the interpretation of dust sampling measurements have not been determined.

1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.

1.4 This test method is generally applicable for an estimate of the surface loading of asbestos starting from approximately 0.24 pg of asbestos per square centimetre (assuming a minimum fiber dimension of 0.5 µm by 0.025 µm, see 17.8), but will vary with the analytical parameters noted in 17.8.

1.4.1 The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disaggregate and disperse asbestos into fibrils and fiber bundles that can be more accurately identified, counted, and sized by transmission electron microscopy. However, as with all indirect sample preparation techniques, the asbestos observed for quantitation may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates not destroys asbestos, but it may alter the physical form of the mineral fibers.

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *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]

D1193 Specification for Reagent Water

D3195 Practice for Rotameter Calibration

D5755 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading

D6620 Practice for Asbestos Detection Limit Based on Counts

E832 Specification for Laboratory Filter Papers

2.2 *ISO Standards:*

ISO/10312 Ambient Air: Determination of Asbestos Fibers; Direct Transfer Transmission Electron Microscopy Procedure[3]

ISO/CD13794 Ambient Air: Determination of Asbestos Fibres; Indirect-Transfer Transmission Electron Microscopy Procedure[3]

3. Terminology

3.1 *Definitions:*

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

3.1.1 *asbestiform*—a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see references (**1**), (**2**) and (**3**).[4]

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

3.1.2.1 *Discussion*—Included in the definition are the asbestiform varieties of: serpentine (chyrsotile); riebeckite (crocidolite); grunerite (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**).

Asbestos	Chemical Abstract Service No.[5]
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Grunerite Asbestos (Amosite)	12172-73-5
Anthophyllite Asbestos	77536-67-5
Tremolite Asbestos	77536-68-6
Actinolite Asbestos	77536-66-4

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aspect ratio*—the ratio of the length of a fibrous particle to its average width.

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

3.2.3 *cluster*—an aggregate of two or more randomly oriented fibers, with or without bundles. Clusters occur as two varieties—disperse clusters and compact clusters.

3.2.3.1 *compact cluster*—a complex and tightly bound network in which one or both ends of each individual fiber or bundle are obscured, such that the dimensions of individual fibers or bundles cannot be unambiguously measured.

3.2.3.2 *disperse cluster*—a disperse and open network in which both ends of one of the individual fibers or bundles can be separately located and its dimensions measured.

3.2.4 *debris*—materials that are of an amount and size (particles greater than 1 mm in diameter as defined by a 1.0 by 1.0 mm screen) that can be visually identified (by color, texture, etc.) as to their source.

3.2.5 *dust*—any material composed of particles in a size range of <1 mm.

3.2.6 *fiber*—a structure having a minimum length of 0.5µ m with an aspect ratio of 5 to 1 or greater and substantially parallel sides (**4**). Fibers are assumed to have a cylindrical shape (**5**).

3.2.7 *fibrous mineral*—a mineral that is composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable.

3.2.7.1 *Discussion*—The crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance. 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. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.

3.2.8 *indirect preparation*—a method in which a sample passes through one or more intermediate steps prior to final filtration.

3.2.9 *matrix*—a structure in which one or more fibers, or fiber bundles, touch, are attached to, or partially concealed by a single particle or connected group of non-fibrous particles. The exposed fiber must meet the fiber definition (see section 3.2.6). Matrices occur as two varieties—disperse matrices and compact matrices.

3.2.9.1 *compact matrix*—a 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.

3.2.9.2 *disperse matrix*—a 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.

3.2.10 *structures*—a 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).

4. Summary of Test Method

4.1 The sample is collected by vacuuming a known surface area with a standard 25 or 37 mm air sampling cassette using a plastic tube that is attached to the inlet orifice which acts as a nozzle. The sample is transferred from inside the cassette to a 50/50 alcohol/water solution and screened through a 1.0 by 1.0 mm screen. The fine dust is filtered onto a membrane filter and ashed in a muffle furnace. The ash is mixed with distilled water to a known volume. Aliquots of the suspension are then filtered through a membrane. A section of the membrane is prepared and transferred to a TEM grid using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using SAED and EDXA at a magnification dependent on the size range of asbestos structures present.

5. Significance and Use

5.1 This microvacuum sampling and indirect analysis method is used for the general testing of non-airborne dust samples for asbestos. It is used to assist in the evaluation of dust that may be found on surfaces in buildings, such as ceiling tiles, shelving, electrical components, duct work, carpet, etc. This test method provides an estimate of the mass surface loading of asbestos in the dust reported as either the mass of

[4] The boldface numbers refer to the list of references at the end of the test method.

[5] The non-asbestiform variations of the minerals indicated in 3.1.2 have different Chemical Abstract Service (CAS) numbers.

asbestos per unit area or as the mass of asbestos per mass of sampled dust as derived from a quantitative TEM analysis.

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

5.1.2 At present, no relationship has been established between asbestos-containing dust as measured by this test method and potential human exposure to airborne asbestos. Accordingly, the users should consider other available information in their interpretation of the data obtained from this test method.

5.2 This definition of dust accepts all particles small enough to pass through a 1 mm screen. Thus, a single, large asbestos-containing particle(s) (from the large end of the particle size distribution) disassembled during sample preparation may result in anomalously large asbestos surface loading results in the TEM analyses of that sample. Conversely, failure to disaggregate large particles may result in anomalously low asbestos mass surface loadings. It is, therefore, recommended that multiple independent samples be secured from the same area, and that a minimum of three samples be analyzed by the entire procedure.

6. Interferences

6.1 The following minerals have properties (that is, chemical or crystalline structure) which are very similar to asbestos minerals and may interfere with the analysis by causing false positives to be recorded during the test. Therefore, literature references for these materials must be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 *Antigorite.*
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 not listed in 5.1.3.

6.2 Collection of any dust particles greater than 1 mm in size in this test method may cause an interference and, therefore, should be avoided.

7. Apparatus

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

7.2 *Energy Dispersive X-ray System (EDXA).*

7.3 *High Vacuum Carbon Evaporator,* with rotating stage.

7.4 *High Efficiency Particulate Air (HEPA),* filtered negative flow hood.

7.5 *Exhaust or Fume Hood.*

7.6 *Particle-Free Water* (ASTM Type II, see Specification D1193).

7.7 *Glass Beakers,* 50 mL.

7.8 *Glass Sample Containers,* with wide mouth screw cap (200 mL), or equivalent sealable container (height of the glass sample container should be approximately 13 cm high by 6 cm wide).

7.9 *Waterproof Markers.*

7.10 *Forceps (tweezers).*

7.11 *Ultrasonic Bath,* table top model (100 W, approximate, see 22.5).

7.12 *Graduated Pipettes,* 1, 5, and 10 mL sizes, glass or plastic.

7.13 *Filter Funnel,* 25 mm or 47 mm (either glass or disposable). Filter funnel assemblies, either glass or disposable plastic, and using either a 25 mm or 47 mm diameter filter.

7.14 *Side Arm Filter Flask,* 1000 mL.

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

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

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

7.18 *Glass Slides.*

7.19 *Scalpel Blades.*

7.20 *Cabinet-type Desiccator,* or low temperature drying oven.

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 where such specifications are available.[6] 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 *Acetone.*

8.3 *Dimethylformamide (DMF).*

8.4 *Chloroform.*

8.5 *1-methyl-2-pyrrolidone.*

8.6 *Glacial Acetic Acid.*

[6] *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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.