Designation: D6480 – 19

Standard Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis for Asbestos Structure Number Surface Loading by Transmission Electron Microscopy[1]

This standard is issued under the fixed designation D6480; 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 identify asbestos in samples wiped from surfaces and to provide an estimate of the concentration of asbestos reported as the number of asbestos structures per unit area of sampled surface. The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregated asbestos into fundamental fibrils, fiber bundles, clusters, or matrices. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates nor destroys asbestos, but it may alter the physical form of the mineral fiber aggregates.

1.2 This test method describes the equipment and procedures necessary for wipe sampling of surfaces for levels of asbestos structures. The sample is collected onto a particle-free wipe material (wipe) from the surface of a sampling area that may contain asbestos.

1.2.1 The collection efficiency of this wipe sampling technique is unknown and will vary among substrates. Properties influencing collection efficiency include surface texture, adhesiveness, and other factors.

1.2.2 This test method is generally applicable for an estimate of the surface loading of asbestos structures starting from approximately 1000 asbestos structures per square centimetre.

1.3 Asbestos identification by transmission electron microscopy (TEM) is based on morphology, electron diffraction (ED), and energy dispersive X-ray analysis (EDXA).

1.4 This test method allows determination of the type(s) of asbestos fibers present.

1.4.1 This test method cannot always discriminate between individual fibers of the asbestos and nonasbestos analogues of the same amphibole mineral.

1.4.2 There is no lower limit to the dimensions of asbestos fibers that can be detected. However, in practice, the lower limit to the dimensions of asbestos fibers, that can be detected, is variable and dependent on individual microscopists. Therefore, a minimum length of 0.5 µm has been defined as the shortest fiber to be incorporated in the reported results.

1.5 The values stated in SI units are to be regarded as 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

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

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

2.2 *Government Standard:*[3]

40 CFR 763, USEPA, Asbestos-Containing Materials in Schools: Final Rule and Notice, Appendix A to Sub-part E

[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, Analysis, Management of Asbestos, and Other Microscopic Particles.

Current edition approved June 1, 2019. Published July 2019. Originally approved in 1999. Last previous edition approved in 2010 as D6480 – 05 (2010). DOI: 10.1520/D6480-19.

[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 U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.access.gpo.gov.

2.3 *U.S. Environmental Protection Agency Standards:*[3]
EPA 600/4-83-043 Analytical Method for the Determination of Asbestos in Water
EPA 747-R-95-001 USEPA, Residential Sampling for Lead: Protocols for Dust and Soil Sampling: Final Report

3. Terminology

3.1 *Definitions*—For definitions of general terms used in this test method, refer to Terminology D1356.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *amphibole asbestos, n*—amphibole in an asbestiform habit (**1**).[4]

3.2.2 *analytical sensitivity, n*—the calculated asbestos structure concentration in asbestos structures/square centimetre, equivalent to counting of one asbestos structure in the analysis calculated using Eq 2.

3.2.3 *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 (**1-3**).

3.2.3.1 *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 in accordance with nomenclature of the International Mineralogical Association (**3, 4**).

Asbestos	Chemical Abstracts Service Registry No.[A]
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

[A] The nonasbestiform variations of the minerals indicated in 3.2.3.1 have different Chemical Abstract Service (CAS) numbers.

3.2.4 *asbestos structure, n*—a term applied to isolated fibers or to any connected or overlapping grouping of asbestos fibers or bundles, with or without other nonasbestos particles.

3.2.5 *aspect ratio, n*—the length to width ratio of a particle.

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

3.2.7 *camera length, n*—the equivalent projection length between the specimen and its selection diffraction pattern, in the absence of lens action.

3.2.8 *chrysotile, n*—a group of fibrous minerals of the serpentine group that have the nominal composition $Mg_3Si_2O_5(OH)_4$ and have the crystal structure of either clinochrysotile, orthochrysotile, or parachrysotile. Most natural chrysotile deviates little from this nominal composition. Chrysotile may be partially dehydrated or magnesium-leached both in nature and in building materials. In some varieties of chrysotile, minor substitution of silicon by Al^{3+} may occur. Chrysotile is the most prevalent type of asbestos.

3.2.9 *cluster, n*—a 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 must have more than two points touching.

3.2.10 *d-spacing or inter-planar spacing, n*—the perpendicular distance between identical adjacent and parallel planes of atoms in a crystal.

3.2.11 *electron diffraction, n*—techniques in electron microscopy that include selected area electron diffraction (SAED) and microdiffraction by which the crystal structure of a specimen is examined.

3.2.12 *energy dispersive X-ray analysis, n*—measurement of the energies and intensities of X-rays by use of a solid state detector and multichannel analyzer system.

3.2.13 *eucentric, n*—the condition when the area of interest of an object is placed on a tilting axis at the intersection of the electron beam at that axis and is in the plane of focus.

3.2.14 *fiber, n*—an elongate particle with parallel or stepped sides. For the purposes of this test method, a fiber is defined to have an aspect ratio equal to or greater than 5:1 and a minimum length of 0.5 µm (see 40 CFR 763).

3.2.15 *fibril, n*—a single fiber, that cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearances.

3.2.16 *fibrous mineral, n*—a mineral composed of parallel, radiating, or interlaced aggregates of fibers from which the fibers are sometimes 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. 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.

3.2.17 *fibrous structure, n*—a fiber, or connected grouping of fibers, with or without other particles.

3.2.18 *field wipe blank, n*—a clean, unused, moistened wipe from the same supply that is used for sampling. Field wipes shall be processed in the same manner used to collect field samples with the exception that no surface is wiped. Each wipe designated as a field wipe should be removed from the bulk pack, moistened, and folded in the same manner as the field samples and placed in a sample container labeled as field wipe.

3.2.19 *filter blank, n*—an unused, unprocessed filter of the type used for liquid filtration.

3.2.20 *filtration blank, n*—a filter prepared from 250 mL of water.

3.2.21 *habit, n*—the characteristic crystal growth form or combination of these forms of a mineral, including characteristic irregularities.

3.2.22 *indirect preparation, n*—a method in which a sample passes through one or more intermediate steps prior to final

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

filtration. The particles are removed from the original medium and deposited on a second filter prior to analysis.

3.2.23 *limit of detection, n*—the limit of detection for a measurement by this test method is 2.99 multiplied by the analytical sensitivity for the measurement.

3.2.23.1 *Discussion*—This limit of detection is based on the assumption that the count resulting from potential filter contamination, sample preparation contamination, and other uncontrollable background sources is no greater than 0.05 structures per sample. At this time, however, this subcommittee has no empirical data to confirm this rate.

3.2.24 *matrix, n*—a 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. The exposed fiber must meet the fiber definition.

3.2.25 *process blank, n*—an unused wipe (that has not been taken into the field) processed in accordance with the entire preparation and analytical procedure.

3.2.26 *replicate sampling, n*—one of several identical procedures or samples.

3.2.27 *serpentine, n*—a group of common rock-forming minerals having the nominal formula: $Mg_3Si_2O_5(OH)_4$. For further information see Ref (**4**).

3.2.28 *structure, n*—a single fiber, fiber bundle, cluster, or matrix.

3.2.29 *structure number concentration, n*—concentration expressed in terms of asbestos structure number per unit of surface area.

3.2.30 *zone-axis, n*—the crystallographic direction of a crystal that is parallel to the intersecting edges of the crystal faces defining the crystal zone.

3.3 *Symbols:*

eV = electron volt
h = hour
J = joule
kV = kilovolt
min = minute(s)
mL = millilitre (10^{-3} litre)
µL = microlitre (10^{-6} litre)
mm = millimetre (10^{-3} metre)
µm = micrometre (10^{-6} metre)
nm = nanometre (10^{-9} metre)
s = second(s)
W = watt
Pa = pascals

3.4 *Acronyms:*

DMF = dimethyl formamide
ED = electron diffraction
EDXA = energy dispersive X-ray analysis
FWHM = full width, half maximum
HEPA = High Efficiency Particulate Air
MCE = mixed cellulose ester and also refers to pure cellulose nitrate filters
PC = polycarbonate
TEM = transmission electron microscope

4. Summary of Test Method

4.1 Wiping a surface of known area with a wipe material collects a sample. The sample is transferred from the wipe material to an aqueous suspension of known volume. Aliquots of the suspension are then filtered through a membrane filter. A section of the membrane filter is prepared and transferred to a TEM grid, using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using ED and EDXA at a magnification from 15 000 to 20 000×.

5. Significance and Use

5.1 This wipe sampling and indirect analysis test method is used for the general testing of surfaces for asbestos. It is used to assist in the evaluation of surfaces in buildings, such as ceiling tiles, shelving, electrical components, duct work, and so forth. This test method provides an index of the concentration of asbestos structures per unit area sampled as derived from a quantitative measure of the number of asbestos structures detected during analysis.

5.1.1 This test method does not describe procedures or techniques required for the evaluation of 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, a single direct relationship between asbestos sampled from a surface and potential human exposure does not exist. Accordingly, the user should consider these data in relationship to other available information (for example, air sampling data) in their evaluation.

5.2 One or more large asbestos-containing particles dispersed during sample preparation may result in large asbestos surface loading results in the TEM analyses of that sample. It is, therefore, recommended that multiple replicate independent samples be secured in the same area, and that a minimum of three such samples be analyzed by the entire procedure.

6. Interferences

6.1 The following materials have properties (that is, chemical composition 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 with asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 Antigorite,
6.1.2 Fibrous talc,
6.1.3 Halloysite,
6.1.4 Hornblende and other amphiboles,
6.1.5 Palygorskite (attapulgite),
6.1.6 Pyroxenes,
6.1.7 Sepiolite, and
6.1.8 Vermiculite scrolls.

7. Apparatus

7.1 *Equipment and Materials for Sampling:*
7.1.1 *Disposable Wet Towels.*