Designation: D6281 – 15

Standard Test Method for Airborne Asbestos Concentration in Ambient and Indoor Atmospheres as Determined by Transmission Electron Microscopy Direct Transfer (TEM)[1]

This standard is issued under the fixed designation D6281; 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[2] is an analytical procedure using transmission electron microscopy (TEM) for the determination of the concentration of asbestos structures in ambient atmospheres and includes measurement of the dimension of structures and of the asbestos fibers found in the structures from which aspect ratios are calculated.

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

1.1.2 This test method cannot always discriminate between individual fibers of the asbestos and non-asbestos analogues of the same amphibole mineral.

1.2 This test method is suitable for determination of asbestos in both ambient (outdoor) and building atmospheres.

1.2.1 This test method is defined for polycarbonate capillary-pore filters or cellulose ester (either mixed esters of cellulose or cellulose nitrate) filters through which a known volume of air has been drawn and for blank filters.

1.3 The upper range of concentrations that can be determined by this test method is 7000 s/mm^2. The air concentration represented by this value is a function of the volume of air sampled.

1.3.1 There is no lower limit to the dimensions of asbestos fibers that can be detected. In practice, microscopists vary in their ability to detect very small asbestos fibers. Therefore, a minimum length of 0.5 µm has been defined as the shortest fiber to be incorporated in the reported results.

1.4 The direct analytical method cannot be used if the general particulate matter loading of the sample collection filter as analyzed exceeds approximately 10 % coverage of the collection filter by particulate matter.

1.5 *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:*[3]

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D1357 Practice for Planning the Sampling of the Ambient Atmosphere

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D6620 Practice for Asbestos Detection Limit Based on Counts

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 *ISO Standard:*[4]

ISO 10312 Ambient air - Determination of asbestos fibres - Direct-transfer transmission electron microscopy method

3. Terminology

3.1 For definitions of general terms used in this test method, refer to Terminology D1356 (see 2.1).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *acicular*—the shape shown by an extremely slender crystal with cross-sectional dimensions that are small relative to its length, that is, needle-like.

[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] This test method was adapted from International Standard ISO 10312 "Air quality—Determination of asbestos fibres—Direct transfer transmission electron microscopy method."

[3] 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.

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

3.2.2 *amphibole*—a group of more than 60 different silicate minerals with similar crystal structures and complex compositions that conform to the nominal formula:

$$A_{0-1}B_2C_5T_8O_{22}(OH,F,Cl)_2 \quad (1)$$

where:

A = K, Na, Ca,
B = Fe^{2+}, Mn, Mg, Ca, Na,
C = Al, Cr, Ti, Fe^{3+}, Mg, Fe^{2+}, Mn, and
T = Si, Al, Cr, Fe^{3+}, Ti.

In some varieties of amphibole, these elements can be partially substituted by Li, Pb, Zn, Be, Ba, or Ni. Amphiboles are characterized by a complex monoclinic or orthorhombic structure that includes a double chain of T-O tetrahedra with a T:O ratio of approximately 4:11; a variable morphology that ranges from columnar to prismatic to acicular to fibrous; and good prismatic cleavage at angles of about 56 and 124°. The cleavage may not be readily exhibited by small crystals that are bound by irregular growth and fracture surfaces (**1**).[5]

3.2.3 *amphibole asbestos*—amphibole in an asbestiform habit.

3.2.4 *analytical sensitivity*—the calculated airborne asbestos structure concentration in asbestos structures/L, equivalent to the counting of one asbestos structure in the analysis.

3.2.5 *asbestiform*—a specific 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.

3.2.6 *asbestos*—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.

3.2.6.1 *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.

Asbestos	Chemical Abstracts Service Registry No.[6]
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

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

3.2.8 *aspect ratio*—the ratio of length to width of a particle.

3.2.9 *blank*—a structure count made on TEM specimens prepared from an unused filter to determine the background measurement.

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

3.2.11 *chrysotile*—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.

3.2.12 *cleavage*—the breaking of a mineral along one of its crystallographic directions.

3.2.13 *cleavage fragment*—a fragment of a crystal that is bounded in whole or in part by cleavage faces. Some cleavage fragments would be included in the fiber definition used in this method.

3.2.14 *cluster*—a structure in which two or more fibers or fiber bundles are randomly oriented in a connected grouping.

3.2.15 *d-value or interplanar spacing*—the perpendicular distance between identical adjacent and parallel planes of atoms in a crystal.

3.2.16 *decision value, n*—the structure count that must be exceeded to claim that a measurement represents a population of airborne structures that is different than the background population, which is established by analyzing blanks (see 3.2.9 and Practice D6620).

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

3.2.18 *electron scattering power*—the extent to which a substance scatters electrons from their original courses.

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

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

3.2.21 *field blank*—a filter cassette that has been taken to the sampling site, opened, and then closed. Such a filter is used to determine the background structure count for the measurement.

3.2.22 *fibril*—a single fiber of chrysotile that cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearances.

3.2.23 *fiber*—an elongated particle that has parallel or stepped sides. For the purposes of this test method, a fiber is defined as having an aspect ratio equal to or greater than 5:1 and a minimum length of 0.5 μm.

3.2.24 *fiber bundle*—a structure composed of parallel, smaller-diameter fibers attached along its length. A fiber bundle may exhibit diverging fibers at one or both ends.

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

[6] The non-asbestiform variations of the minerals indicated in 5.2.6 have different Chemical Abstracts Service (CAS) numbers.

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

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

3.2.27 *limit of detection*—the mean count for a population of structures that has been determined, based on a measurement or average of measurements, to be different than the background population of structures (see 3.2.16 and Practice D6620). The limit of detection may be restated in units of structures/L by multiplying the mean count by analytical sensitivity (see 3.2.4).

3.2.28 *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 nonfibrous particles.

3.2.29 *miller index*—a set of three integer numbers used to specify the orientation of a crystallographic plane in relation to the crystal axes.

3.2.30 *PCM equivalent fiber*—a particle of aspect ratio that is greater than or equal to 3:1, is longer than 5 µm, and that has a diameter between 0.2 and 3.0 µm

3.2.31 *PCM equivalent structure*—a fibrous structure of aspect ratio that is greater than or equal to 3:1, is longer than 5 µm, and has a diameter between 0.2 and 3.0 µm.

3.2.32 *primary structure*—a fibrous structure that is a separate entity in the TEM image.

3.2.33 *replication*—a procedure in electron microscopy specimen preparation in which a thin copy, or replica, of a surface is made.

3.2.34 *residual structure*—matrix or cluster material containing asbestos fibers that remains after accounting for the prominent component fibers or bundles, or both.

3.2.35 *serpentine*—a group of common rock-forming minerals having the nominal formula: $Mg_3Si_2O_5(OH)_4$.

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

3.2.37 *twinning*—the occurrence of crystals of the same species joined together at a particular mutual orientation, and such that the relative orientations are related by a definite law.

3.2.38 *unopened fiber bundle*—a large-diameter asbestos fiber bundle that has not been separated into its constituent fibrils or fibers.

3.2.39 *zone-axis*—the crystallographic direction parallel to the intersection edges of the crystal faces defining the crystal zone.

3.3 *Symbols:*

eV = electron volt
kV = kilovolt
L/min = liters per minute
µg = micrograms (10^{-6} g)
µm = micrometer (10^{-6} m)
nm = nanometer (10^{-9} m)
W = watt
Pa = Pascals

3.4 *Abbreviations:*

DMF = dimethyl formamide
ED = electron diffraction
EDXA = energy dispersive X-ray analysis
FWHM = full width, half maximum
HEPA = high-efficiency particle absolute
MCE = mixed cellulose ester; also refers to pure cellulose nitrate filters
PC = polycarbonate
PCM = phase contrast optical microscopy
ED = selected area electron diffraction
SEM = scanning electron microscope
STEM = scanning transmission electron microscope
TEM = transmission electron microscope
UICC = Union Internationale Contre le Cancer

4. Summary of Test Method

4.1 A sample of airborne particulate matter is collected by drawing a measured volume of air through either a capillary-pore polycarbonate membrane filter of maximum pore size 0.4 µm or a cellulose ester (either mixed esters of cellulose or cellulose nitrate) membrane filter of maximum pore size 0.45 µm by means of a battery-powered or mains-powered pump. TEM specimens are prepared from polycarbonate filters by applying a thin film of carbon to the filter surface by vacuum evaporation. Small areas are cut from the carbon-coated filter, supported on TEM specimen grids, and the filter medium is dissolved away by a solvent extraction procedure. This procedure leaves a thin film of carbon that bridges the openings in the TEM specimen grid and that supports each particle from the original filter in its original position. Cellulose ester filters are chemically treated to collapse the pore structure of the filter, and the surface of the collapsed filter is then etched in an oxygen plasma to try to expose particles embedded in the collapsed filter. A thin film of carbon is evaporated onto the filter surface and small areas are cut from the filter. These sections are supported on TEM specimen grids, and the filter medium is dissolved by a solvent extraction procedure.

4.2 The TEM specimen grids from either preparation method are examined at both low and high magnifications to check that they are suitable for analysis before carrying out a quantitative structure count on randomly-selected grid openings. In the TEM analysis, electron diffraction (ED) is used to examine the crystal structure of a fiber, and its elemental composition is determined by energy dispersive X-ray analysis (EDXA). For a number of reasons, it is not possible to identify each fiber unequivocally and fibers are classified according to the techniques that have been used to identify them. For each fiber, a simple code is used to record the manner in which it was classified. The fiber classification procedure is based on successive inspection of the morphology, the ED pattern, and the qualitative and quantitative EDXA. Confirmation of the identification of chrysotile is only by quantitative ED, and confirmation of amphibole is only by quantitative EDXA and quantitative zone axis ED.

4.3 In addition to isolated fibers, ambient air samples often contain more complex aggregates of fibers, with or without other particles. Some particles are composites of asbestos