Designation: D8526 – 23

Standard Test Method for Analytical Procedure Using Transmission Electron Microscopy for the Determination of the Concentration of Carbon Nanotubes and Carbon Nanotube-containing Particles in Ambient Atmospheres[1]

This standard is issued under the fixed designation D8526; 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 is an analytical procedure using transmission electron microscopy (TEM) for the determination of the concentration of carbon nanotubes and carbon nanotube-containing particles in ambient atmospheres.

1.1.1 This test method is suitable for determination of carbon nanotubes in both ambient (outdoor) and building atmospheres.

1.2 This test method is defined for polycarbonate capillary pore filters through which a known volume of air has been drawn and for blank filters.

1.3 The direct analytical method cannot be used if the general particulate matter loading of the sample collection filter as analyzed exceeds approximately 25 % coverage of the collection filter by particulate matter.

1.4 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *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

D1357 Practice for Planning the Sampling of the Ambient Atmosphere

D5337 Practice for Setting and Verifying the Flow Rate of Personal Sampling Pumps

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

D7712 Terminology for Sampling and Analysis of Asbestos

E2456 Terminology Relating to Nanotechnology

2.2 *NIOSH Standards:*[3]

NIOSH 7400 Asbestos and Other Fibers by PCM

NIOSH 7402 Asbestos by TEM

3. Terminology

3.1 *Definitions*—For definitions of general terms used in this test method, refer to Terminologies D1356, D7712, and E2456.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *analytical sensitivity, n*—the calculated airborne nanotube structure concentration in nanotube structures per liter, equivalent to the counting of one nanotube structure in the analysis.

3.2.2 *carbon nanotube, n*—an allotrope of carbon structurally defined by a size of less than 100 nm in two or more

[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 Sept. 1, 2023. Published September 2023. DOI: 10.1520/D8526-23.

[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 National Institute for Occupational Health and Safety (NIOSH), 400 7th Street S.W., Suite 5W, Washington, D.C. 20024, https://www.cdc.gov/niosh/index.htm.

dimensions and a tubular morphology often comprised of one or more coaxial cylinders of graphene (that is, walls).

3.2.2.1 *Discussion*—This method is also applicable to other micrometer or nanometer size carbon fibers, including amorphous carbon nanotubes, carbon nanofibers, carbon nanorods, cellulose nanofibers, and carbon tubes with diameters greater than or equal to 100 nm.

3.2.3 *carbon nanotube structure, n*—a term applied to isolated carbon nanotubes or to any connected or overlapping grouping of carbon nanotubes or bundles of carbon nanotubes, with or without other non-nanotube particles.

3.2.3.1 *Discussion*—See Annex A1 for terminology related to reported structure types.

4. Summary of Test Method

4.1 A sample of airborne particulate matter is collected by drawing a measured volume of air through a capillary-pore polycarbonate membrane filter of maximum pore size 0.4 µm by means of a battery-powered or mains-powered pump. TEM specimens are prepared from 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. The TEM specimen grids 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, the elemental composition of a nanotube structure may be confirmed by energy dispersive X-ray analysis (EDXA).

4.2 In addition to isolated single carbon nanotubes, ambient air samples often contain more complex aggregates of single carbon nanotubes, with or without other particles. Some particles are composites of nanotube structures with other materials. Individual carbon nanotubes and these more complex structures are referred to as nanotube structures. Any continuous grouping of particles in which a single carbon nanotube with a length greater than or equal to 250 nm is detected shall be recorded on the count sheet. These will be designated nanotube structures and classified according to the counting rules specified in Annex A1. The number of nanotube structures found on a known area of the microscope sample, together with the equivalent volume of air filtered through this area, is used to calculate the airborne concentration in nanotube structures per liter of air.

4.2.1 For this method, a minimum length of 250 nm has been defined as the shortest carbon nanotube to be incorporated in the reported test results. However, fibers identified as shorter than 250 nm may be reported separately by the analyst.

4.3 The upper range of concentrations that can be determined by this test method is 7000 structures per square millimeter. The air concentration represented by this value is a function of the volume of air sampled.

5. Significance and Use

5.1 This test method is applicable to the measurement of airborne carbon nanotubes in a wide range of ambient air situations and for evaluation of any atmosphere for carbon nanotube structures. Single carbon nanotube structures in ambient atmospheres have diameters below the resolution limit of the light microscope. This test method is based on transmission electron microscopy, which has adequate resolution to allow detection of small thin single carbon nanotubes and is currently a reliable technique capable of unequivocal identification of the majority of nanotube structures. Carbon nanotubes are often found, not as single carbon nanotubes, but as very complex, aggregated structures, which may or may not be aggregated with other particles.

5.2 This test method applies to the analysis of a single filter and describes the precision attributable to measurements for a single filter. Multiple air samples are usually necessary to characterize airborne nanotube structure concentrations across time and space. The number of samples necessary for this purpose is proportional to the variation in measurement across samples, which may be greater than the variation in measurement for a single sample.

6. Apparatus

6.1 *Air Sampling Equipment and Consumable Supplies:*

6.1.1 *Carbon Rods,* for use in vacuum coating unit during carbon coating of filters.

6.1.1.1 Use of carbon fiber type evaporators should be avoided since they may not be capable of preparing clean laboratory blanks and may introduce carbon fibers during the coating process.

6.1.2 *Filter Cassette,* 25 mm to 50 mm diameter, commercially manufactured, non-reusable, three-piece cassettes, with cowls in front of the filter surface, used for sample collection. Electrically conductive cowls are preferred.

6.1.2.1 Cassette should be loaded with a capillary pore polycarbonate filter of maximum pore size 0.4 µm. Back filter with a 5 µm pore size mixed cellulose ester (MCE) filter and support it by a cellulose back-up pad. Apply a shrink cellulose band or adhesive tape when the filters are in position to prevent air leakage. Ensure that the filters are tightly clamped in the assembly so that significant air leakage around the filter cannot occur.

6.1.2.2 A cassette with the same design, filter media, and pore size as the cassettes to be used for collecting air samples should be reserved and used exclusively for adjusting the flow rate of sampling equipment.

6.1.2.3 It is recommended that representative filters from the filter lot be analyzed as described in Section 12 for the presence of nanotube structures before any are used for air sample collection.

6.1.2.4 Alternatively, a cassette as described above loaded with a 0.45 µm pore size (or less) MCE filter in place of the polycarbonate filter (commonly used for asbestos TEM air clearance sampling) may be used if standard MCE filter preparation techniques, such as those described by NIOSH (Method 7402) or Test Method D6281 without plasma etching, are used to prepare the TEM grids.