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Standard Test Methods for Estimating Contribution of Environmental Tobacco Smoke to Respirable Suspended Particles Based on UVPM and FPM[1]

This standard is issued under the fixed designation D 5955; 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 These test methods pertain to the sampling/analysis of respirable suspended particles (RSP) and the estimation of the RSP fraction attributable to environmental tobacco smoke (ETS). These test methods are based on collection of total RSP on a membrane filter, extracting the collected material in methanol, and measuring total ultraviolet absorbance or fluorescence, or both, of this extract. The corresponding methods of estimation are termed ultraviolet particulate matter (UVPM) and fluorescent particulate matter (FPM), respectively.

1.2 These test methods are compatible with, but do not require the determination of solanesol, which is also used to estimate the contribution of ETS to RSP (see Test Method D 6271).

1.3 The sampling components consist of a preweighed, 1.0-µm pore size polytetrafluoroethylene (PTFE) membrane filter in a filter cassette connected on the inlet end to a particle size separating device and, on the outlet end, to a sampling pump. These test methods are applicable to personal and area sampling.

1.4 These test methods are limited in sample duration only by the capacity of the membrane filter (about 2000 µg). These test methods have been evaluated up to a 24-h sample duration with a minimum sample duration of at least 1 h.

1.5 Limits of detection (LOD) and quantitation (LOQ) for the UVPM test method at a sampling rate of 2 L/min are, respectively, 2.5 µg/m^3 and 8.3 µg/m^3 for a 1-h sample duration and 0.3 µg/m^3 and 1.0 µg/m^3 for an 8-h sample duration. The LOD and LOQ for the FPM test method at a sampling rate of 2 L/min are, respectively, 1.4 µg/m^3 and 4.7 µg/m^3 for a 1-h sample duration and 0.2 µg/m^3 and 0.6 µg/m^3 for an 8-h sample duration.

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

1.7 *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.* Specific precautionary information is given in 13.6.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres

D 1357 Practice for Planning the Sampling of the Ambient Atmosphere

D 3631 Test Methods for Measuring Surface Atmospheric Pressure

D 5337 Practice for Flow Rate for Calibration of Personal Sampling Pumps

D 6271 Test Method for Estimating Contribution of Environmental Tobacco Smoke to Respirable Suspended Particles Based on Solanesol

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology D 1356.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *environmental tobacco smoke* (ETS)—an aged, dilute composite of exhaled tobacco smoke (exhaled mainstream smoke) and smoke from tobacco products (sidestream smoke).

3.2.2 *environmental tobacco smoke particulate matter* (ETS-PM)—the particulate phase of ETS.

3.2.3 *fluorescent particulate matter* (FPM)—an estimation of the contribution of ETS particulate matter to RSP obtained

[1] These test methods are under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and are the direct responsibility of Subcommittee D22.05 on Indoor Air.

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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.

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by comparing the fluorescence intensity of the RSP sample to that of a surrogate standard.

3.2.4 *respirable suspended particles* (RSP)—particles which, when captured by a size-selective sampling device, conform to a collection efficiency curve with a median cutpoint at an aerodynamic diameter of 4.0 µm (**1**).[3]

3.2.5 *surrogate standard*—a chemical whose concentration has been related quantitatively to a known concentration of ETS-PM.

3.2.6 *2,2′,4,4′-tetrahydroxybenzophenone* (THBP)—a UVPM surrogate standard.

3.2.7 *ultraviolet particulate matter* (UVPM)—an estimation of the contribution of ETS particulate matter to RSP obtained by comparing the ultraviolet absorbance of the RSP sample to that of a surrogate standard.

4. Summary of Test Methods

4.1 A known volume of air is drawn through an inertial impactor or cyclone assembly separating at 4.0 µm to separate RSP from total suspended particulate matter and then through a filter assembly. The respirable suspended particulate matter is collected on a PTFE membrane filter contained within the filter assembly.

4.2 The weight of RSP is determined as the difference between the filter weight before and after collection. The concentration of RSP (µg/m^3) is calculated from the RSP weight and volume of air sampled.

4.3 The filter is extracted with methanol in a 4-mL glass vial.

4.4 An aliquot of the extract is injected into a columnless high performance liquid chromatography (HPLC) system equipped with an ultraviolet detector (325 nm) and a fluorescence detector (300-nm excitation; 420-nm emission) connected in series. (Alternatively, absorbance and fluorescence may be measured with bench-top spectrophotometers.)

4.5 The area of the resulting UV peak is compared to areas obtained from the injection of standard solutions of THBP (a surrogate standard for ETS-PM). The area of the resulting fluorescence peak is compared to areas obtained from the injection of standard solutions of scopoletin (a surrogate standard for ETS-PM). The results, which are estimates of the contribution of ETS-PM to RSP, are reported as UVPM and FPM, respectively.

5. Significance and Use

5.1 Environmental tobacco smoke consists of both vapor- and particle-phase components. Due to the nature of vapor and particulate phases, they rarely correlate well, and an accurate assessment of ETS levels in indoor air requires determining good tracers of both phases. Among the attributes of an ideal ETS tracer, one critical characteristic is that the tracer should "remain in a fairly consistent ratio to the individual contaminant of interest or category of contaminants of interest (for example, suspended particulates) under a range of environmental conditions..." (**2**). The UVPM and FPM fulfill this requirement, staying in a constant ratio to RSP from tobacco smoke under a variety of ventilation conditions and sampling durations. Solanesol (a C_{45} isoprenoid alcohol specific to tobacco), determined in accordance with Test Method D 6271, is an ETS tracer or marker that also meets this requirement. In contrast, nicotine (a component of the ETS vapor phase) does not remain in a consistent ratio to ETS-PM (**3**).

5.2 To be able to quantify the contribution of ETS to RSP is important because RSP is not specific to tobacco smoke. The RSP are a necessary indicator of overall air quality; the Occupational Safety and Health Administration (OSHA) has previously set a PEL (permissible exposure level) for respirable dust in the workplace of 5000 µg/m^3. However, the RSP emanate from numerous sources (**4**) and have been shown to be an inappropriate tracer of ETS (**5-13**). In the test methods described herein, UVPM and FPM are used as more selective markers to estimate more accurately the contribution of ETS to RSP (**5-7, 9-18**). Of the available ETS particulate phase markers (UVPM, FPM, and solanesol), all are currently used and relied upon in investigations of indoor air quality, although UVPM and FPM can overestimate the contribution of tobacco smoke to RSP due to potential interference from nontobacco combustion sources. Solanesol, because it is tobacco-specific and ETS particle phase-specific, may be the best indicator of the ETS particulate phase contribution to RSP (**9-13, 19-21**). Refer to Test Method D 6271 for the protocol on determining solanesol.

6. Interferences

6.1 Because the measured spectral properties are not unique to ETS-PM, these test methods will always be a conservative measure of (that is, they overestimate) the contribution of ETS to indoor RSP. Combustion sources are known to add significantly to the UVPM measure (**19**); FPM is considered to be less prone to, but not free from, interferences. Due to the potential presence of unquantifiable interferences, these test methods provide only an indication of, and not the absolute level of, the contribution of ETS to indoor RSP.

7. Apparatus

7.1 *Sample Collection*:

7.1.1 *PTFE Filter,* membrane filter with 1.0-µm pore size and 37-mm diameter. The PTFE membrane is bonded to a high density polyethylene support net, referred to as the filter backing, to improve durability and handling ease.

7.1.2 *Filter Sampling Assembly*, consists of the PTFE membrane filter and a black, opaque, conductive polypropylene filter cassette in a three-piece configuration with a 0.5-in. spacer ring inserted between the top (inlet) and bottom (outlet) pieces. All connections to the filter assembly are made with flexible (for example, plastic) tubing.

7.1.3 *Barometer and Thermometer*, for taking pressure and temperature readings at the sampling site.

7.1.4 *Bubble Flowmeter or Mass Flowmeter,* for calibration of the sampling pump.

7.1.5 *Personal Sampling Pump,* portable constant-flow air sampling pump calibrated for a flow rate dependent upon the separating characteristics of the impactor or cyclone in use (see 7.1.6).

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

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7.1.6 *Inertial Impactor or Cyclone,* with nominal cutpoint of 4.0 µm at the specified flow rate.

NOTE 1—If alternate definition of RSP is used (see 3.2.4), ensure that the impactor or cyclone is compatible with this definition.

7.1.7 *Stopcock Grease*, for coating impactor plates.

7.2 *Analytical System*:

7.2.1 *Liquid Chromatography System*, consists of HPLC pump, autosampler, ultraviolet detector, fluorescence detector, peak integration system, and 10-ft stainless steel tubing with 0.009-in. inside diameter. Note that no HPLC analytical column is used. If this analysis is attempted using an ultraviolet spectrophotometer, a cell with a path length of at least 40 mm is recommended.

7.2.2 *Sample Containers*, low-actinic borosilicate glass autosampler vials, 4-mL capacity, with screw caps and PTFE-lined septa.

7.2.3 *Microgram Balance,* for weighing filters. (Readability = 1 µg or lower.)

7.2.4 *Filter Forceps*, for handling filters.

7.2.5 *Static Inhibitor,* for removing static charge from filters.

7.2.6 *Wrist-action Shaking Device*, for solvent extraction.

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. 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 *Methanol*, HPLC grade, (CAS No. 67-56-1).

8.3 *2,2′,4,4′-Tetrahydroxybenzophenone*, 99 %, (CAS No. 131-55-5), UVPM surrogate standard.

8.4 *Scopoletin*, 95 %, (CAS No. 92-61-5), FPM surrogate standard.

8.5 *Glycerol*, 99.5 %, (CAS No. 56-81-5).

8.6 *Water*, distilled and deionized, (CAS No. 7732-18-5).

8.7 *Helium*, 99.995 %, (CAS No. 7440-59-7), for continuous purging of methanol mobile phase.

9. Sampling

9.1 *General*—For planning sampling programs, refer to Practice D 1357.

9.2 *Procedure*:

9.2.1 Adjust the potentiometer on the sampling pump to obtain the flow rate specified for the particular type of inertial impactor or cyclone being used (see 7.1.6).

9.2.2 Calibrate the personal sampling pump prior to and immediately after sampling. For calibration, connect the flowmeter to the inlet of the inertial impactor or cyclone. Measure flow with the prepared filter sampling assembly in place between the pump and the impactor or cyclone. Refer to Practice D 5337 for standard practice in calibrating personal sampling pumps.

9.2.3 Record the barometric pressure and ambient temperature.

9.2.4 If using a mass flowmeter, record the volumetric flow rate, Q, of the sampling pump. Generate several soap-film bubbles in the flowmeter and allow them to thoroughly wet the surface before recording any actual measurements. Measure the time for a soap-film bubble to travel a known volume with a stopwatch. Obtain five replicate measurements and compute the mean time. Calculate the volumetric flow rate, Q, from Eq 1:

$$Q = \frac{V}{R} \tag{1}$$

where:

Q = pump flow rate, L/min,
V = volume measured with flowmeter, L, and
R = average time for soap-film bubble to flow a known volume (V) in a flowmeter, min.

9.2.5 With the prepared filter sampling assembly correctly inserted and positioned between the impactor or cyclone and the pump, turn on the pump power switch to begin sampling; record the start time.

NOTE 2—Most pumps have microprocessing capabilities or built-in elapsed time meters, or both, for preset sampling periods.

9.2.6 Record the temperature and barometric pressure of the atmosphere being sampled.

9.2.7 Acquire samples at the flow rate required for the impactor or cyclone in use (see 7.1.6), for a minimum time of 1 h. Turn off the pump at the end of the desired sampling period and record the time elapsed during sample collection.

9.2.8 Recheck the flow rate of the pump again after sampling and use the average flow rate (mean of before and after sampling) in later calculations.

9.2.9 Immediately remove the filter cassette containing the sample collected on the membrane filter from the sampling system and seal the inlet and outlet ports of the filter cassette with plastic plugs.

9.2.10 Treat a minimum of six prepared filter sampling assemblies in the same manner as the samples (remove plugs, measure flow, replace plugs, and transport). Label and process these filters as *field blanks*.

9.2.11 Store all filter cassettes containing the samples in a freezer or under dry ice and transport frozen to the laboratory for analysis.

NOTE 3—If the samples are not prepared and analyzed immediately, then store them at -10°C or less. Analyze all the filters within six weeks after sample collection. It has been established that samples are stable for at least six weeks at -10°C storage conditions. (**22**)

10. Analysis

10.1 *System Description*:

10.1.1 Perform analysis using a columnless HPLC system equipped with an ultraviolet (UV) detector (for UVPM) or a fluorescence detector (for FPM), or both (for both UVPM and FPM).

10.1.2 Wavelength settings are: 325 nm for the UV detector and 300-nm excitation and 420-nm emission for the fluorescence detector.

10.1.3 No analytical column is used; pump and detector are connected with tubing as listed in 7.2.1.

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