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Standard Test Methods for Estimating Contribution of Environmental Tobacco Smoke to Respirable Suspended Particles Based on UVPM and FPM¹

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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. 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 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.

1.3 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.

1.4 Limits of detection (LOD) and limit of quantitation (LOQ) for the UVPM test method at a sampling rate of 2 L/min are, respectively, 2.5 $\mu\text{g}/\text{m}^3$ and 8.3 $\mu\text{g}/\text{m}^3$ for a 1-h sample duration and 0.3 $\mu\text{g}/\text{m}^3$ and 1.0 $\mu\text{g}/\text{m}^3$ 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 $\mu\text{g}/\text{m}^3$ and 4.7 $\mu\text{g}/\text{m}^3$ for a 1-h sample duration and 0.2 $\mu\text{g}/\text{m}^3$ and 0.6 $\mu\text{g}/\text{m}^3$ for an 8-h sample duration.

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 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:

- D 1356 Terminology Relating to Sampling and Analysis of Atmospheres²
- D 1357 Practice for Planning the Sampling of the Ambient Atmosphere²
- D 3631 Test Methods for Measuring Surface Atmospheric Pressure²
- D 5337 Practice for Flow Rate for Calibration of Personal Sampling Pumps²

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 and smoke from tobacco products.

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

3.2.4 *respirable suspended particles* (RSP)—particles which can be deposited in the gas-exchange region of the lung and are defined as particles that pass through a sampler having a 4.0- μm median cutpoint (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.

¹ These test methods are under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and are the direct responsibility of Subcommittee D22.05 on Indoor Air.

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² *Annual Book of ASTM Standards*, Vol 11.03.

³ The boldface numbers in parentheses refer to the list of references at the end of this 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 ($\mu\text{g}/\text{m}^3$) 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 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 particulate-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. In contrast, nicotine (a component of the ETS vapor phase) does not remain in a consistent ratio to ETS-PM (3).

5.2 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 $\mu\text{g}/\text{m}^3$. The RSP emanate from numerous sources (4) and have been shown to be an inappropriate tracer of ETS (5-8). The test methods described herein have been used to estimate more accurately the contribution of ETS to RSP (5-79-13).

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 (14); 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*,⁴ membrane filter with 1.0- μm pore size and 37-mm diameter.

7.1.2 *Filter Sampling Assembly*, consists of monitor case,⁵ PTFE membrane filter, and gasket.⁶ 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*⁷ or *Mass Flowmeter*,⁸ for sample pump calibration.

7.1.5 *Personal Sampling Pump*,⁹ portable constant-flow sampling pump calibrated for a flow rate dependent upon the separating characteristics of the impactor/cyclone in use (see 7.1.6).

7.1.6 *Inertial Impactor*¹⁰ or *Cyclone*,^{11, 12} with nominal cutpoint of 4.0 μm .

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 solvent

⁴ Fluoropore membrane filters, Catalog No. FALP 03700, available from Millipore Corp., 80 Ashby Rd., Bedford, MA 01730, or equivalent, have been found suitable for this purpose.

⁵ Monitor cases available from Millipore Corp., Catalog No. M00003700, or equivalent, have been found suitable for this purpose.

⁶ Gaskets, Catalog No. 0301139-A31, available from Sloan Valve Co., 10500 Seymour Ave., Franklin Park, IL 60131, or equivalent, have been found suitable for this purpose.

⁷ The Gilibrator primary standard airflow calibrator manufactured by Gilian Instrument Corp., 35 Fairfield Pl., W. Caldwell, NJ 07006, or equivalent, has been found suitable for this purpose.

⁸ The Top = Trak mass flowmeter, Model No. 821-1-(Air, 5SLPM), available from Sierra Instruments, Inc., 5 Harris Ct., Monterey, CA 93940, or equivalent, has been found suitable for this purpose.

⁹ Personal sampling pump, Model 224-50, available from SKC Inc., 863 Valley View Rd., Eighty Four, PA 15330, or equivalent, has been found suitable for this purpose.

¹⁰ Inertial impactor manufactured by TSI, Inc., Catalog No. 8508-040, available from Craft Associates, 770 Old Roswell Rd., Ste E500, Roswell, GA 30076, or equivalent, has been found suitable for this purpose.

¹¹ Respirable dust cyclone, Model BGI-4, available from BGI Inc., 58 Guinan St., Waltham, MA 02154, or equivalent, has been found suitable for this purpose.

¹² Cyclone, Catalog No. 2418584-0001, available from Sensidyne, Inc., 16333 Bay Vista Dr., Clearwater, FL 34620, or equivalent, has been found suitable for this purpose.



delivery system,¹³ autosampler,¹⁴ ultraviolet detector,¹⁵ fluorescence detector,¹⁶ peak integration system, and 10-ft stainless steel tubing with 0.009-in. inside diameter. Note that no LC 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*, 4-mL LC vials with screw caps and PTFE-lined septa.

7.2.3 *Microgram Balance*,¹⁷ for weighing filters. (Readability = 1 µg or lower.)

7.2.4 *Filter Forceps*, for handling filters.

7.2.5 *Static Inhibitor*,¹⁸ 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.¹⁹ 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), UVP 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 % grade, (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 Calibrate the personal sampling pump prior to and immediately after sampling. For calibration, connect flowmeter

to the inlet of the inertial impactor or cyclone. Measure flow with the prepared filter assembly in place between the pump and impactor or cyclone. Refer to Practice D 5337 for standard practice in calibrating personal sampling pumps.

9.2.2 Record the barometric pressure and ambient temperature.

9.2.3 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:

$$Q = \frac{V}{R} \quad (1)$$

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.

9.2.4 Adjust the potentiometer on the sampling pump so that the desired flow rate is obtained.

9.2.5 With the filter assembly correctly inserted and positioned between the impactor or cyclone and 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 required flow rate 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 assembly from the sampling system and seal the filter cassette with plugs provided.

9.2.10 Treat a minimum of six filter 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 used filter assemblies 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, they should be stored at 0°C or less. All filters should be analyzed within eight weeks after sample collection. It has been established that samples are stable for at least eight weeks at -10°C storage conditions.

10. Analysis

10.1 *System Description*:

10.1.1 Analysis is performed using a columnless HPLC system equipped with an ultraviolet (UV) detector or a fluorescence detector, or both.

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

10.1.3 Helium is used for continuous purging of the methanol mobile phase.

¹³ Solvent delivery system, Model M-45, available from Waters Chromatography Division, Millipore Corp., 34 Maple St., Milford, MA 01757, or equivalent, has been found suitable for this purpose.

¹⁴ Autosampler available from Waters, Model 717, or equivalent, has been found suitable for this purpose.

¹⁵ Programmable wavelength detector available from Waters, Model 490E, or equivalent, has been found suitable for this purpose.

¹⁶ Fluorescence detector, Model F1000, available from Hitachi, 44 Old Ridgebury Rd., Danbury, CT 06810, or equivalent, has been found suitable for this purpose.

¹⁷ Microgram balance, Model C-31, available from Cahn Instruments, Inc., 16207 S. Carmenita Rd., Cerritos, CA 90701, or equivalent, has been found suitable for this purpose.

¹⁸ Static inhibitor manufactured by NRD, Inc., Catalog No. B2075-1, available from Baxter Diagnostics, Inc., 1430 Waukegan Rd., McGaw Park, IL 60085, or equivalent, has been found suitable for this purpose.

¹⁹ *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.