Designation: D5955 – 02 (Reapproved 2017)ε1

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 D5955; 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.

ε[1] NOTE—Reapproved with editorial changes and warning notes editorially updated throughout in March 2017.

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 D6271).

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

1.8 *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]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D1357 Practice for Planning the Sampling of the Ambient Atmosphere

D3631 Test Methods for Measuring Surface Atmospheric Pressure

D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps

D6271 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 D1356.

[1] These test methods are under the jurisdiction of ASTM Committee D22 on Air Quality 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.

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 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.

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

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 D6271, 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 D6271 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 1.27-cm 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).

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 3.05-m stainless steel tubing with 0.2-mm 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 D1357.

9.2 *Procedure:*

9.2.1 Adjust 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 D5337 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 built-in elapsed time meters 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.

10.1.4 Use helium for the continuous purging of the methanol mobile phase.

10.1.5 HPLC pump flow is 0.4 mL/min.

10.1.6 Injection volume is 50 µL.

10.1.7 Run time is 2 min.

10.1.8 Retention time for UVPM is approximately 0.5 min and for FPM (with the fluorescence detector connected in series downstream from the UV detector) is approximately 0.7 min.

10.1.9 Measure peak areas electronically using any appropriate chromatography data acquisition system or digital electronic integrator.

11. Procedure

11.1 *Gravimetric Determination of RSP and Filter Sampling Assembly Preparation:*

11.1.1 Prepare 80.0 % (w/w) aqueous solution of glycerol by mixing 800 g of 99.5 % glycerol with 200 g distilled, deionized water. Prepare solution at least every 12 months.

11.1.2 Prepare humidity-controlled chamber at approximately (50 ± 2) % RH by placing a 80.0 % (w⁄w) aqueous solution of glycerol (see 11.1.1) in a tray in the bottom of a desiccator cabinet. (23)

11.1.3 Remove the top covers of individual boxes of membrane filters and place the boxes in humidity-controlled chamber for at least 12 h prior to weighing.

11.1.4 Calibrate and zero the microgram balance according to the manufacturer's instructions.

11.1.5 Place the filter on a dust- and lint-free surface under an antistatic device for approximately 15 s.

11.1.6 Weigh the filter to the nearest microgram on a microgram balance containing another antistatic device attached to the wall inside the weighing chamber.

NOTE 4—Handle the filter with clean forceps only.

11.1.7 Repeat 11.1.5 and 11.1.6 until three weights are obtained for each filter, ensuring that the balance is zeroed between each individual weighing.

NOTE 5—If any of the three weights appear to be outliers, then establish a range of acceptable weights that is appropriate for the individual laboratory.

11.1.8 Record the mean of the three replicate weighings as the tare weight.

11.1.9 Place the weighed filter inside the three-piece filter cassette, with the filter backing facing the cassette outlet (bottom piece), and with the spacer ring (center piece of the cassette) in place between the filter and the cassette inlet (top piece).

11.1.10 Tightly seal the filter cassette containing the weighed filter and, if desired, seal the cassette with a cassette-sealing band as a precaution against leaks or tampering, or both. Allow the band to dry thoroughly. If the prepared filter sampling assembly is not to be used immediately, then plug the inlet and outlet ports of the cassette with plastic plugs.

11.1.11 After sample collection, return the filter cassette containing the sample collected on the weighed filter to the weighing area.

NOTE 6—If the sample was stored below room temperature, then allow the filter cassette containing the sample to equilibrate to room temperature prior to removing the inlet and outlet plugs.

11.1.12 Remove the plugs and place the filter cassette containing the sample in the humidity-controlled chamber for at least 12 h prior to reweighing.

11.1.13 Reweigh the filter following the procedure described in 11.1.4 – 11.1.7.

11.1.14 Record the mean of the three replicate weighings as the final weight.

11.1.15 Transfer the filter to a clean sample vial and seal, then label. Begin UVPM or FPM determination, or both, immediately or store the sealed vial at –10°C or less until analysis.

11.2 *Preparation of UVPM Surrogate Standard Solutions:*

11.2.1 Clean all volumetric flasks and screw-cap jars used for the preparation of standard solutions with detergent, thoroughly rinse with tap water, followed by distilled water, followed by methanol, and allow to air dry. (**Warning—**In cleaning the glassware, avoid the use of dishwashing detergents because some have been found to leave unacceptably high absorbance backgrounds. Use a cleaner designed for cleaning laboratory equipment.)

11.2.2 Prepare a primary standard of THBP (1000 µg/mL) by weighing 100 mg of THBP directly into a 100-mL volumetric flask, diluting to the mark with methanol, and shaking to mix.

11.2.3 Prepare a secondary standard of THBP (16 µg/mL) by transferring 4.00 mL of the primary standard to a 250-mL volumetric flask, diluting to the mark with methanol, and shaking to mix.

11.2.4 Prepare five working standards covering the expected concentration range of the samples. Typical volumes used (diluted to 100 mL in methanol) are 1, 2, 5, 10, 20, and 40 mL (of the secondary standard), which yield UVPM standards of 0.16, 0.32, 0.80, 1.60, 3.20, and 6.40 µg/mL THBP, respectively. Of these, select either the five lowest or the five highest in concentration to cover the expected range of samples.

11.2.5 Store all standard solutions in low-actinic borosilicate glass screw-cap jars in a refrigerator (approximately 4°C) when not in use. Allow standards to reach room temperature and transfer sufficient volume (2 mL to 3 mL) of each working standard to a clean sample vial each day for instrument calibration. Cap and tightly seal the vials.

11.2.6 Prepare a methanol blank by transferring neat methanol to a clean sample vial. Analyze this blank as a *zero* standard.

NOTE 7—Prepare the *zero* standard for each run from the methanol used for extracting samples (that is, do not prepare it in advance and store with the other standard solutions).

11.2.7 Prepare working standards from the secondary standard and secondary standard from the primary standard as needed. Prepare primary standard at least every 12 months. Deterioration of the primary standard has not been observed and no definitive time interval has been established for its replacement; however, storage and use for more than 12 months is not recommended.

11.3 *Preparation of FPM Surrogate Standard Solutions:*

11.3.1 Clean all volumetric flasks used for the preparation of standard solutions with detergent, thoroughly rinse with tap water, followed by distilled water, followed by methanol, and allow to air dry.

11.3.2 Prepare a primary standard of scopoletin (350 µg/mL) by weighing 35 mg of scopoletin (assuming 100 % scopoletin purity) directly into a 100-mL volumetric flask, diluting to the mark with methanol, and shaking to mix.

NOTE 8—The concentrations of the standard solutions will depend on the purity of the scopoletin reagent. Use the actual purity of the scopoletin reagent when calculating the concentrations of the standard solutions.

11.3.3 Prepare a secondary standard of scopoletin (3.50 µg/mL) by transferring 1.00 mL of the primary standard to a 100-mL volumetric flask, diluting to the mark with methanol, and shaking to mix. The secondary standard is also the highest level working standard.

11.3.4 Prepare a tertiary standard of scopoletin (0.350 µg/mL) by transferring 10.00 mL of the secondary standard to a 100-mL volumetric flask, diluting to the mark with methanol, and shaking to mix. The tertiary standard is also one of the working standards.

11.3.5 Prepare five working standards covering the expected concentration range of the samples. Typical volumes used (diluted to 100 mL in methanol) are 1 and 3 mL (of the tertiary standard) and 1, 3, and 30 mL (of the secondary standard), which yield FPM standards of 0.0035, 0.0105, 0.035, 0.105, 0.350 (the tertiary standard), 1.05, and 3.50 (the secondary standard) µg/mL scopoletin. Of these, select either the five lowest or the five highest in concentration to cover the expected range of samples.

11.3.6 Store all standard solutions in low-actinic borosilicate glass screw-cap jars in a refrigerator (approximately 4°C) when not in use. Allow standards to reach room temperature and transfer sufficient volume (2 mL to 3 mL) of each working standard to a clean sample vial each day for instrument calibration. Cap and tightly seal the vials.

11.3.7 Prepare a methanol blank by transferring neat methanol to a clean sample vial. Analyze this blank is as a *zero* standard.

NOTE 9—Prepare the *zero* standard for each run from the methanol used for extracting samples (that is, do not prepare it in advance and store with the other standard solutions).

11.3.8 Prepare standards from scopoletin at least every six months. Deterioration of the standards has been observed in standards stored for more than six months.

11.4 *Extraction of Filter:*

11.4.1 If samples and field blanks stored in the sealed vials were stored in a freezer (see 11.1.15), allow them to reach room temperature. Add 3.00 mL of methanol to each sample vial. Prepare field blanks in exactly the same manner as samples. In addition, prepare and analyze two unweighed filters as laboratory blanks.

NOTE 10—If high concentration samples are being analyzed, then filters may be extracted in larger volumes of methanol (4.00 mL can be accommodated in the specified vials), or initial extracts may be quantitatively diluted.

11.4.2 Seal each vial tightly with the septum/cap assembly and place in a holding tray. After all samples have been prepared, transfer the vials or trays to a wrist-action shaking device and extract under agitation for 60 min.

11.5 *Loading the Autosampler:*

11.5.1 Load UVPM standards at the beginning of the autosampler queue, followed by FPM standards (if performing UVPM and FPM determinations simultaneously; otherwise, omit standards for analysis not being conducted).

11.5.2 Load the *zero* standard, samples, field blanks, and laboratory blanks in queue following the standards.

11.5.3 Make duplicate injections of each solution and obtain integrated peak area counts for each by way of the peak integration system. Compare the peak areas of samples and standards and use the corresponding calibration curve to calculate the concentrations of UVPM or FPM, or both, in the samples.

NOTE 11—It is acceptable to use either the mean peak area (obtained from duplicate injections) for quantitation or to obtain individual results from each injection and report the results for each sample as the mean of the duplicate injections.

11.6 *Constructing the UVPM Calibration Curve*—Calculate the mean peak area counts obtained from duplicate injections of each standard (*y*-axis, including the *zero* standard) and, together with surrogate standard (THBP) concentrations (*x*-axis, in micrograms per millilitre, including the *zero* standard), construct a linear regression model, and obtain the slope and *y*-intercept.

NOTE 12—If detector nonlinearity is significant, a weighted regression (for example, $1/x$ weighting) or a second-order polynomial regression, or both, may be more appropriate; if so, substitute the appropriate regression equation in the calculations in 12.3.1.

11.7 *Constructing the FPM Calibration Curve*—Calculate the mean peak area counts obtained from duplicate injections of each standard (*y*-axis, including the *zero* standard) and, together with surrogate standard (scopoletin) concentrations (*x*-axis, in micrograms per millilitre, including the *zero* standard), construct a linear regression model, and obtain the slope and *y*-intercept.

NOTE 13—If detector nonlinearity is significant, a weighted regression or a second-order polynomial regression, or both, may be more appropriate; if so, substitute the appropriate regression equation in the calculations in 12.5.1. Also, especially for FPM, ensure that detector response for all

standards is within the operating range of the instrument. If not, alter the detector sensitivity settings accordingly or delete higher-level standards as necessary.

12. Calculation

12.1 *Calculation of RSP Weight:*

12.1.1 Record the weight of RSP, in micrograms, as the difference between the tare weight of the filter and the final weight after sampling in accordance with Eq 2:

$$RSP = X_2 - X_1 \quad (2)$$

where:

RSP = weight of RSP, µg,
X_2 = mean of 3 replicate weighings of filter after sampling, recorded as the final weight, µg (see 11.1.14), and
X_1 = mean of 3 replicate weighings of filter prior to sampling, recorded as the tare weight, µg (see 11.1.8).

12.1.2 Blank-correct all values obtained for RSP by subtracting the mean weight difference determined for the field blanks.

12.2 *Calculation of RSP Concentration:*

12.2.1 Calculate the volume of air sampled in accordance with Eq 3:

$$V = \text{Time} \times Q \quad (3)$$

where:

V = volume of air sampled, L,
Time = time elapsed during sample collection, min, and
Q = pump flow rate, L/min, that was determined during initial calibration (see 9.2.4) or the average (before and after sampling) pump flow rate (see 9.2.8).

12.2.2 Calculate the RSP concentration in accordance with Eq 4:

$$[RSP] = \frac{RSP \times 1000}{V} \quad (4)$$

where:

$[RSP]$ = concentration of RSP, µg/m^3,
RSP = weight of RSP, µg (see 12.1),
1000 = conversion factor, L/m^3, and
V = volume of air sampled, L.

12.2.3 Adjust the RSP concentration found in the sampled air to standard conditions of temperature and pressure in accordance with Eq 5 (optional):

$$[RSP]^{stp} = [RSP] \times \frac{101.325}{P} \times \frac{(T+273)}{298} \quad (5)$$

where:

$[RSP]^{stp}$ = concentration of RSP corrected to standard temperature and pressure, µg/m^3,
$[RSP]$ = concentration of RSP calculated in 12.2.2, µg/m^3,
P = barometric pressure of atmosphere sampled, kPa,
T = temperature of atmosphere sampled, °C,
101.325 = standard pressure, kPa, and
298 = standard temperature, K.

12.3 *Calculation of UVPM Concentration:*

12.3.1 Convert the mean peak area counts obtained from duplicate injections of samples and blanks to [UVPM^{sse}] (UVPM expressed as surrogate standard equivalents in micrograms per millilitre) in accordance with Eq 6 (using the slope and intercept values obtained in 11.6):

$$[UVPM^{sse}] = \frac{(\text{mean area count}) - (y - \text{intercept})}{\text{slope}} \quad (6)$$

assuming the calibration data were fit to a linear model.

12.3.2 Correct each sample for the sample blank with the following equation:

$$[UVPM^{sse}]_{corr} = \text{sample} - \text{average blank} \quad (7)$$

where:

$[UVPM^{sse}]_{corr}$ = blank-corrected $[UVPM^{sse}]$ concentration, µg/mL,
sample = $[UVPM^{sse}]$ concentration found in 12.3.1, µg/mL, and
average blank = average of $[UVPM^{sse}]$ concentration found in all field blanks, µg/mL.

12.3.3 Calculate $[UVPM]$ from $[UVPM^{sse}]_{corr}$ in accordance with Eq 8:

$$[UVPM] = [UVPM^{sse}]_{corr} \times 7.5 \quad (8)$$

where:

$[UVPM]$ = UVPM concentration in ETS equivalents, µg/mL,
$[UVPM^{sse}]_{corr}$ = blank-corrected UVPM concentration in surrogate standard equivalents found in 12.3.2, µg/mL, and
7.5 = conversion factor from surrogate standard to ETS equivalents (that is, 7.5 µg of ETS-PM has absorbance equivalent to 1.0 µg of THBP).

NOTE 14—This conversion factor is an aggregate of factors determined empirically in an environmental test chamber where the only RSP present was that generated from the normal smoking of selected cigarettes. Individual factors include: 8.0 determined for the Kentucky 1R4F reference cigarette (**6**), 7.5 for the leading 50 cigarette brand styles in the United States (**21**), 8.2 for the leading six cigarette brand styles in each of ten countries in Europe and Asia (**24**), and 7.2 for six leading cigarette brand styles in each of eight countries in other regions of the world (**25**). It should also be noted that, if the ETS-PM being measured is from a specific tobacco product with a known conversion factor, then this factor should be substituted. The applicability of this factor has not been determined for tobacco smoke not meeting the definition of ETS as given in 3.2.1 (for example, machine-generated sidestream smoke).

12.3.4 Calculate $UVPM$ from $[UVPM]$ in accordance with Eq 9:

$$UVPM = [UVPM] \times \text{extract volume} \quad (9)$$

where:

$UVPM$ = UVPM weight in ETS equivalents, µg/filter,
$[UVPM]$ = UVPM concentration found in 12.3.3, µg/mL, and
extract volume = volume of methanol, mL, used to extract filter (from 11.4.1; typically either 3 mL or 4 mL).

12.3.5 Calculate the airborne concentration of UVPM from volume of air sampled (see 12.2.1) and UVPM weight (see 12.3.4) by the relationship shown in Eq 4 (see 12.2.2).

12.3.6 Adjust the UVPM concentration found in the sampled air to standard conditions of temperature and pressure by the relationship shown in Eq 5 (see 12.2.3) (optional).

12.4 *RSP Apportionment as Estimated by UVPM:*

12.4.1 Calculate the RSP fraction that is estimated to be attributable to ETS-PM, based on the determination of UVPM, in accordance with Eq 10 (optional):

$$ETS - PM^{UV} = \frac{UVPM}{RSP} \times 100 \quad (10)$$

where:

$ETS\text{-}PM^{UV}$ = portion of RSP attributable (estimate) to ETS, based on UVPM measurement, %,
$UVPM$ = UVPM weight found in 12.3.4, µg, and
RSP = RSP weight found in 12.1, µg.

12.5 *Calculation of FPM Concentration:*

12.5.1 Convert the mean peak area counts obtained from duplicate injections of samples and blanks to $[FPM^{sse}]$ (FPM expressed as surrogate standard equivalents in micrograms per millilitre) in accordance with Eq 11 (using the slope and intercept values obtained in 11.7):

$$[FPM^{sse}] = \frac{(\text{mean area count}) - (y - \text{intercept})}{\text{slope}} \quad (11)$$

assuming the calibration data were fit to a linear model.

12.5.2 Correct each sample for the sample blank with Eq 12:

$$[FPM^{sse}]_{corr} = \text{sample} - \text{average blank} \quad (12)$$

where:

$[FPM^{sse}]_{corr}$ = blank-corrected $[FPM^{sse}]$ concentration, µg/mL,
sample = $[FPM^{sse}]$ concentration found in 12.5.1, µg/mL, and
average blank = average of $[FPM^{sse}]$ concentration found in all field blanks, µg/mL.

12.5.3 Calculate $[FPM]$ from $[FPM^{sse}]_{corr}$ in accordance with Eq 13:

$$[FPM] = [FPM^{sse}]_{corr} \times 39.0 \quad (13)$$

where:

$[FPM]$ = FPM concentration in ETS equivalents, µg/mL,
$[FPM^{sse}]_{corr}$ = blank-corrected FPM concentration in surrogate standard equivalents found in 12.5.2, µg/mL, and
39.0 = conversion factor from surrogate standard to ETS equivalents (that is, 39.0 µg of ETS-PM has fluorescence intensity equivalent to 1.0 µg of scopoletin).

NOTE 15—This conversion factor is an aggregate of factors determined empirically in an environmental test chamber where the only RSP present was that generated from the normal smoking of selected cigarettes. Individual factors include: 33.6 determined for the Kentucky 1R4F reference cigarette (**6**), 39.0 for the leading 50 cigarette brand styles in the United States (**21**), 44.2 for the leading six cigarette brand styles in each of 10 countries in Europe and Asia (**24**), and 41.8 for six leading cigarette brand styles in each of eight countries in other regions of the world (**25**). It should also be noted that, if the ETS-PM being measured is from a specific tobacco product with a known conversion factor, then this factor should be substituted. The applicability of this factor has not been determined for tobacco smoke not meeting the definition of ETS as given in 3.2.1 (for example, machine-generated sidestream smoke).

12.5.4 Calculate FPM from $[FPM]$ in accordance with Eq 14:

$$FPM = [FPM] \times \text{extract volume} \quad (14)$$

where:

FPM = FPM weight in ETS equivalents, µg/filter,
$[FPM]$ = FPM concentration found in 12.5.3, µg/mL, and
extract volume = volume of methanol, mL, used to extract filter (from 11.4.1; typically either 3 mL or 4 mL).

12.5.5 Calculate the airborne concentration of FPM from volume of air sampled (see 12.2.1) and FPM weight (see 12.5.4) by the relationship shown in Eq 4 (see 12.2.2).

12.5.6 Adjust the FPM concentration found in the sampled air to standard conditions of temperature and pressure by the relationship shown in Eq 5 (see 12.2.3) (optional).

12.6 *RSP Apportionment as Estimated by FPM:*

12.6.1 Calculate the RSP fraction that is estimated to be attributable to ETS-PM, based on the determination of FPM, in accordance with Eq 15 (optional):

$$ETS - PM^{F} = \frac{FPM}{RSP} \times 100 \quad (15)$$

where:

$ETS\text{-}PM^{F}$ = portion of RSP attributable (estimate) to ETS, based on FPM measurement, %,
FPM = FPM weight found in 12.5.4, µg, and
RSP = RSP weight found in 12.1, µg.

13. Performance Criteria and Quality Assurance

13.1 This section summarizes required quality assurance measures and provides guidance concerning performance criteria that should be achieved within each laboratory.

13.2 *Standard Operating Procedures (SOPs):*

13.2.1 Users should generate SOPs describing and documenting the following activities in their laboratory:

13.2.1.1 Assembly, calibration, leak-check, and operation of the specific sampling system and equipment used,

13.2.1.2 Preparation, storage, shipment, and handling of samples,

13.2.1.3 Assembly, leak-check, calibration, and operation of the analytical system, addressing the specific equipment used, and

13.2.1.4 All aspects of data recording and processing, including lists of computer hardware and software used.

13.2.2 The SOPs should provide specific, step-by-step instructions and should be readily available to, and understood by, the laboratory personnel conducting the work.

13.2.3 Sample blanks should contain less than the equivalent of 0.5 µg of ETS particulate matter (UVPM or FPM, or

both). Larger quantities would be evidence of contamination during sampling or analysis.

13.2.4 Periodically, the inertial impactor's surface is wiped clean, and a thin coat of stopcock grease is applied. If a cyclone is used, empty the grit pot prior to each use, and ensure that the cyclone remains upright (that is, it should never turn past horizontal) during sampling.

13.2.5 In the event that an initial sample result is above the calibration range, prepare and analyze additional standards, or quantitatively dilute and reanalyze the sample.

13.3 *Calibration of Personal Sampling Pumps:*

13.3.1 Calibrate sampling pumps at the beginning and at the conclusion of each sampling period.

13.3.2 Set the pump flow controller using a bubble or mass flowmeter at the appropriate sampling rate (depending on the separating characteristics of the impactor or cyclone in use) with the filter sampling assembly in place.

13.3.3 For conversion of measured flows to standard flows, record barometric pressure and ambient temperature during both pump calibration and sampling (see Test Methods D3631).

13.4 *Method Sensitivity, Precision, and Linearity:*

13.4.1 The sensitivity of these test methods is demonstrated by the detection limits of 2.5 μg/m^3 and 1.4 μg/m^3 for RSP attributable to ETS by UVPM and FPM, respectively, for a 1-h sample duration.

13.4.2 The precision of these test methods is determined by the coefficients of variation of repeatability, *a*, and the coefficients of variation of reproducibility, *A*.

13.4.3 Nonlinearity in the calibration curve may occur at concentrations near the upper usable range of the UV or fluorescence detector in use. Also, it is not unusual (especially for FPM) for samples to be outside the dynamic range of the detector in which case additional dilution of the sample extract and reanalysis are required.

13.5 *Test Method Modification:*

13.5.1 The sampling time described in these test methods may be extended beyond 24 h provided that the capacity of the filter is not exceeded. Also, a sampling time of less than 1 h may be used in areas of very high ETS-PM concentration (for example, in an environmental test chamber).

13.5.2 The flow rate of air through the filter may be increased up to 5 L/min and beyond provided that the chosen flow rate is within the range specified for the given particle size separator (impactor or cyclone) in use.

13.5.3 The sample extracts resulting from the procedures described herein are also compatible with the determination of solanesol (**9-13, 19-21**), which is also used as a tracer of the particulate phase of ETS (see Test Method D6271).

13.6 *Safety:*

13.6.1 If spilling of solvent or any of the reagents occurs, take quick and appropriate cleanup action. (See Material Safety Data Sheets that are provided by the seller of the chemicals as prescribed by law.)

13.6.2 When preparing standards, as with handling any chemicals, avoid contact with skin and eyes.

14. Precision and Bias

14.1 For these test methods, coefficients of variation of repeatability, *a*, and reproducibility, *A*, have been calculated for RSP, UVPM, and FPM in a collaborative study (**26**). The precision data were determined from an experiment organized and analyzed in accordance with ISO 5725-1 and ISO 5725-2 guidelines in 1998 involving ten laboratories for RSP, eleven laboratories for UVPM and FPM, and six levels. Data from one laboratory for RSP and FPM, and data from two laboratories for UVPM contained outliers. These outliers were not included in the calculation of the repeatability standard deviations and the reproducibility standard deviations. Precision data were determined to vary linearly with mean level over the range 71 μg to 219 μg per sample for RSP, 7.8 μg to 28.1 μg per sample (in surrogate standard equivalents) for UVPM, and 1.7 μg to 8.7 μg per sample (in surrogate standard equivalents) for FPM. These relationships are the following:

$$s_r = a \times m \tag{16}$$

and

$$s_R = A \times m \tag{17}$$

where:

s_r = repeatability standard deviation, μg/sample,
s_R = reproducibility standard deviation, μg/sample,
m = mean sample level, μg/sample,
a = 0.072 for RSP, 0.018 for UVPM, and 0.048 for FPM, and
A = 0.089 for RSP, 0.086 for UVPM, and 0.114 for FPM.

Similar results were obtained for the coefficient of variation of reproducibility for the UVPM test method in a previous collaborative study (**27**) and, for the coefficient of variation of repeatability for UVPM, results obtained in the new collaborative study are lower in value thus indicating better precision than in the previous study.

14.2 Recovery of THBP from the PTFE membrane filter was found to average 94.4 % (**27**). The THBP, however, is not known to be a component of environmental tobacco smoke. Recovery of scopoletin from the PTFE membrane filter was found to average 97.6 % (**28**). Scopoletin is known to be a component of environmental tobacco smoke.

15. Keywords

15.1 environmental tobacco smoke (ETS); fluorescent particulate matter (FPM); indoor air quality; respirable suspended particles (RSP); ultraviolet particulate matter (UVPM)

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