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Standard Test Method for Estimating Contribution of Environmental Tobacco Smoke to Respirable Suspended Particles Based on Solanesol[1]

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1. Scope

1.1 This test method covers the sampling/analysis of respirable suspended particles (RSP) and the estimation of the RSP fraction attributable to environmental tobacco smoke (ETS). The test method is based on collection of total RSP on a membrane filter, extraction of the filter in methanol, and determination of solanesol, a C_{45} isoprenoid alcohol, by high performance liquid chromatography (HPLC) with ultraviolet (UV) detection.

1.2 This test method is compatible with the determinations of gravimetric RSP, ultraviolet particulate matter (UVPM), and fluorescent particulate matter (FPM) (see Test ~~Methods~~Method D5955), but does not require them. UVPM and FPM, which are based on the ultraviolet absorbance and fluorescence of the filter extract, are also used to estimate the contribution of ETS to RSP.

1.3 The sampling components consist of a 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. This test method is applicable to personal and area sampling.

1.4 This test method is limited in sample duration only by the capacity of the membrane filter. The test method has been evaluated up to 24-h sample duration; a minimum sample duration of 1 h is recommended.

1.5 Limits of detection (LOD) for this test method at a sampling rate of 2 L/min are 0.042 µg/m^3 for 1-h sample duration and 0.005 µg/m^3 for 8-h sample duration.

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

1.6 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 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]

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 Calibration of Personal Sampling Pumps

D5955 Test Methods for Estimating Contribution of Environmental Tobacco Smoke to Respirable Suspended Particles Based on UVPM and FPM

3. Terminology

3.1 *Definitions:* For definitions of terms used in this test method, refer to Terminology D1356.

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 *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].

3.2.3 *solanesol particulate matter (Sol-PM)*—a tobacco-selective marker for the contribution of ETS particulate matter to RSP.

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[1] This test method is under the jurisdiction of ASTM Committee D22 ~~on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee~~ on Air Quality and is 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] The boldface numbers in parentheses refer to a list of references at the end of this standard.

4. Summary of Test Method

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

4.2 Solanesol is extracted from the filter with methanol in a 4-mL glass vial.

4.3 An aliquot of the extract is injected into an HPLC system equipped with a UV detector (205 nm absorbance).

4.4 The area of the resulting solanesol peak is compared to areas obtained from the injection of standard solutions of solanesol, and the weight of solanesol is determined.

4.5 The concentration of solanesol (µg/m^3) is calculated from the weight of solanesol and the volume of air sampled. If desired, the concentration of RSP can also be calculated according to Test ~~Methods~~Method D5955.

4.6 The concentration of RSP attributable to ETS, referred to as Sol-PM, is calculated from the airborne concentration of solanesol and the experimentally determined weight ratio of solanesol to RSP in ETS **(2,3,4)**.

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..." **(5)**. Solanesol meets this requirement, staying in a constant ratio to the RSP contributed by tobacco smoke over a variety of ventilation conditions and sampling durations **(6)**. UVPM and FPM, which are the tracers or markers employed by Test ~~Methods~~Method D5955, also fulfill this requirement. Airborne solanesol, however, is unique in that it is specific to tobacco smoke and is found only in the particulate phase of ETS. Its high molecular weight and low volatility make it extremely unlikely that any solanesol will be lost from the membrane filter used for sample collection. Solanesol constitutes approximately 3 % by weight of the RSP of ETS **(2,7,8)**, making it suitable for measurement at realistic smoking rates. Of the available ETS particulate phase markers (UVPM, FPM, and solanesol), all are currently used and relied upon, but solanesol is considered to be a better marker for the particulate phase of ETS and, as a result, provides the best way of quantifying the contribution of ETS particulate matter to RSP **(3,4,9,10,11,12,13)**.

5.2 To be able to quantify the contribution of ETS to RSP with a tobacco-specific marker is important because RSP is not specific to tobacco smoke. RSP is 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, RSP emanates from numerous sources **(14)** and has been shown to be an inappropriate tracer of ETS **(7,15,16,17)**. UVPM and FPM are used as more selective markers to estimate the contribution of tobacco smoke to RSP; however, these markers can overestimate the contribution of tobacco smoke to RSP due to potential interference from nontobacco combustion sources. (Refer to Test ~~Methods~~Method D5955 for the protocol on determining UVPM and FPM.) Although UVPM and FPM are useful in investigations of indoor air quality, solanesol is a better indicator of the tobacco smoke contribution to RSP. This test method has been used to apportion RSP into ETS and non-ETS components by determining the weight ratio of solanesol to total RSP **(2,3,4,7,18,19)**.

6. Interferences

6.1 The genus *Nicotiana*, which includes tobacco as one of its species, is a member of the *Solanaceae* family of plants. Like tobacco, many plants in this family, particularly those which also contain trace amounts of nicotine, contain solanesol. Examples are tomato, potato, eggplant, and pepper. With cooking as the only likely source of interference, the potential for interference is negligible. However, if there were an interference of this type, the weight of solanesol would be biased high and the contribution of ETS to RSP would be overestimated. It is anticipated that the only measurable contribution of solanesol to an indoor environment would come from tobacco combustion.

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.~~1.3-cm spacer ring inserted between the top (inlet) and bottom (outlet) pieces.[4] The filter cassette holds the PTFE membrane during sampling. All connections to the filter assembly are made with flexible 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.

[4] The three-piece filter cassette (with a spacer ring in the center) is not always needed. A two-piece filter cassette may be substituted.

7.1.5 *Personal Sampling Pump*, portable constant-flow 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.

NOTE 1—If alternate definition of RSP is used (see 3.2.2), 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, UV detector with deuterium source lamp, autosampler, column oven (optional), and data acquisition and peak integration system.

7.2.2 *HPLC Column*, 250 mm by 3.0-mm ID, reversed-phase C_{18} column ~~(300-Å~~(30-nm pore size; 5-µm particle size). C_{18} packing material with low carbon loading has been found to be preferable.

7.2.3 *Guard Cartridge Column*, a guard cartridge with packing material and dimensions compatible with the HPLC column in 7.2.2, placed in front of the analytical column for protecting and prolonging the life of the column.

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

7.2.5 *Filter Forceps*, for handling 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.[5] 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 *Acetonitrile*, HPLC grade, (CAS No. 75-05-8).

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

8.4 *Solanesol*~~, 90+%, (CAS No. 13190-97-1). (See Note 6.)~~ , 90+ %, (CAS No. 13190-97-1).

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

9. Sampling

9.1 *General*—for planning sampling programs, refer to Practice D1357.

9.2 *Procedure*:

NOTE 2—If a gravimetric determination of RSP is to be performed, then weigh the filters according to Test ~~Methods~~Method D5955 prior to 9.2.1.

9.2.1 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 the flow with the prepared filter assembly in place between the pump and the impactor or cyclone. Refer to Practice D5337 for standard practice in calibrating personal sampling pumps.

9.2.2 Record the barometric pressure and ambient temperature.

9.2.3 If using a mass flowmeter, record the volumetric flow rate, Q. If using a bubble flowmeter, 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, in accordance with Eq 1:

$$Q = VR \quad (1)$$

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 3—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 sampling period of 1 h. Turn off the pump at the end of the desired sampling period and record the time elapsed during sample collection.

[5] *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. Pharmaceupeial Convention, Inc. (USPC), Rockville, MD.

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 two 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 4—If the samples are not prepared and analyzed immediately, then store them at 0°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 **(20)**.

10. Analysis

10.1 *System Description*:

10.1.1 Perform analysis using an HPLC system equipped with a UV detector at a wavelength setting of 205 nm.

NOTE 5—A UV detector with a deuterium source is required. A detector with a xenon source is not acceptable because of insufficient lamp energy at 205 nm.

10.1.2 The HPLC column and guard column are as listed in 7.2.2 and 7.2.3.

10.1.3 The mobile phase consists of 95:5 (v/v) acetonitrile: methanol.

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

10.1.5 Pump flow is 0.5 mL/min.

10.1.6 Injection volume is 100 µL.

10.1.7 Run time is 15 min.

10.1.8 Retention time for solanesol is approximately 9 min.

10.1.9 Measure peak areas electronically with a chromatography data acquisition system.

11. Procedure

11.1 *Preparation of Solanesol Standard Solutions*:

11.1.1 Clean all volumetric flasks and screw-cap jars used for the preparation and storage 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 liquid laboratory cleaner designed for cleaning laboratory equipment.

11.1.2 Prepare a primary standard of solanesol (300 µg/mL) by weighing 30 mg of solanesol (assuming 100 % solanesol purity) directly into a 100-mL volumetric flask, diluting to volume with methanol, and shaking to mix.

NOTE 6—Actual concentration of standard solutions will depend on the exact weight and purity of the solanesol reagent used in 11.1.2. Obtain the solanesol purity from the vendor for the specific lot of solanesol reagent used. The vendor specifies the purity of the solanesol reagent for each individual lot number produced.

11.1.3 Prepare a secondary standard of solanesol (15 µg/mL) by transferring 5.00 mL of the primary standard to a 100-mL volumetric flask, diluting to volume with methanol, and shaking to mix.

11.1.4 Prepare a tertiary standard of solanesol (6 µg/mL) by transferring 2.00 mL of the primary standard to a 100-mL volumetric flask, diluting to volume with methanol, and shaking to mix.

11.1.5 Prepare five working standards of solanesol which cover the expected concentration range of the samples. Typical volumes used (diluted to 100 mL in methanol) are 1 mL of tertiary standard; 1, 3, and 7 mL of secondary standard; and 1 mL of primary standard. This provides a calibration range with the following concentrations of solanesol: 0.060, 0.150, 0.450, 1.05, and 3.00 µg/mL. Store all standard solutions in low-actinic borosilicate glass screw-cap jars in a freezer (below 0°C) when not in use. Before transfer and use, allow solutions to reach room temperature, observing a minimum equilibration time of 1 h, and mix thoroughly. Transfer sufficient volume of each working standard (2 to 3 mL) to a clean, 4-mL autosampler vial each day for instrument calibration. Cap and tightly seal the vials.

11.1.6 Prepare working standards and secondary and tertiary standards 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.

11.2 *Extraction of Filter*:

NOTE 7—If a gravimetric determination of RSP is to be performed, then reweigh the filters according to Test ~~Methods~~Method D5955 prior to 11.2.1.

11.2.1 Place each filter in a vial, label the vial, and add 3.00 mL methanol. Prepare field blanks in exactly the same manner as samples. In addition, prepare and analyze two previously unweighed filters as laboratory blanks.

NOTE 8—If high concentration samples are being analyzed, filters may be extracted in 4.00 mL of methanol.

11.2.2 Seal the 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.

11.3 *Loading the Autosampler*: