CORESTA RECOMMENDED METHOD N° 51

ENVIRONMENTAL TOBACCO SMOKE —ESTIMATION OF ITS CONTRIBUTION TO RESPIRABLE SUSPENDED PARTICLES — DETERMINATION OF PARTICULATE MATTER BY ULTRAVIOLET ABSORBANCE AND BY FLUORESCENCE

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1. FIELD OF APPLICATION

Environmental tobacco smoke (ETS) is an aerosol consisting of vapor and particulate phase components. Due to the nature of the two aerosol 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 (e.g., suspended particulates) under a range of environmental conditions…" (see Ref. 3.1)

Ultraviolet particulate matter (UVPM) and fluorescent particulate matter (FPM) fulfill this requirement, staying in a consistent ratio to respirable suspended particles (RSP) from tobacco smoke under a variety of ventilation conditions and sampling durations. In contrast, nicotine (a component of the ETS aerosol vapor phase) does not remain in consistent ratio to ETS particulate matter (ETS-PM) (see Ref. 3.2).

RSP, a necessary indicator of overall air quality, emanates from many sources, such as combustion processes (including tobacco smoke), atmospheric dust, talc, insecticide dusts, viruses, bacterias, etc. (see Ref. 3.3). Consequently, RSP is an inappropriate tracer of ETS levels present in any environment. Studies have shown that in most indoor spaces where smoking is permitted without restriction, 50% or less of the RSP (on average) is attributable to tobacco smoke (see Ref. 3.4, 3.5, 3.6, 3.7). The test methods described here have been used effectively to reduce the uncontrollable bias inherent in the use of RSP as a tracer of ETS (see Ref. 3.4, 3.5, 3.6, 3.8, 3.9, 3.10, 3.11, 3.12, 3.13).

2. **DEFINITIONS**

2.1. Environmental Tobacco Smoke (ETS)

A mixture of aged and diluted exhaled mainstream smoke and aged and diluted sidestream smoke.

2.2. Respirable Suspended Particulates (RSP)

Particles which, when captured by a size-selective sampling device, conform to a collection efficiency curve with a median cut point at an aerodynamic diameter of 4.0 μ m (see Ref. 3.14).

2.3. *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 with that of a surrogate standard.

2.4. 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 with that of a surrogate standard.

- **2.5.** *Environmental Tobacco Smoke Particulate Matter (ETS-PM)* The particulate phase of ETS.
- **2.6.** *Surrogate Standard*

A chemical whose concentration has been related quantitatively to a known concentration in the solution of ETS-PM; 2,2',4,4'-tetrahydroxybenzophenone (THBP) for UVPM, scopoletin for FPM.

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4. PRINCIPLE

Drawing a known volume of air through an inertial impactor or cyclone separating at $4.0\,\mu m$, thus separating RSP from total suspended particulate matter, and then through a filter cassette containing a polytetrafluoroethylene (PTFE) membrane filter. Collection of RSP on the filter, followed by gravimetric determination of the mass of RSP so collected. Extraction of the RSP from the filter for the determination of UVPM and FPM by absorbance and fluorescence measurement, respectively, using high performance liquid chromatography (HPLC) equipment.

Note: If HPLC apparatus is not available, absorbance and fluorescence may be measured by spectrophotometer with the addition of a note in the expression of results.

5. APPARATUS

- 5.1. Polytetrafluoroethylene (PTFE) membrane filter, of pore size 1.0 µm and diameter 37 mm. The PTFE membrane is bonded to a high density polyethylene support net, referred to as the filter backing, to improve durability and handling ease.
- **5.2.** Filter cassette, of black, opaque, conductive polypropylene in a three-piece configuration consisting of a 12.7 mm spacer ring inserted between the top (inlet) and bottom (outlet) pieces. The filter cassette holds the PTFE membrane filter during sampling. All connections to the filter cassette are made with flexible (e.g., plastic) tubing.
- **5.3.** Barometer and thermometer, for taking pressure and temperature readings at the sampling site (optional).
- **5.4.** Bubble flowmeter or mass flowmeter for calibration of the sampling pump.
- **5.5.** Personal sampling pump, consistent-flow air sampling pump, calibrated for a flow rate dependent upon the separating characteristics of the impactor or cyclone in use.
- 5.6. Inertial impactor or cyclone, with nominal cut point of 4.0 µm at specified flow rate. If alternate definition of RSP is used, ensure that the impactor or cyclone is compatible with this definition.
- **5.7.** Stopcock grease, for coating impactor plates.
- **5.8.** High performance liquid chromatography (HPLC) system, consisting of solvent delivery system, autosampler, ultraviolet detector, fluorescence detector, peak integration system, and 3.0 m stainless steel tubing with 0.23 mm inside diameter.
- **5.9.** Sampling containers, consisting of low-actinic borosilicate glass autosampler vials, 4 ml capacity, with screw caps and PTFE-lined septa.
- **5.10.** Desiccator cabinet, for preparing a humidity-controlled chamber where filters are stored prior to weighing.
- **5.11.** Static inhibitor, for removing static charge from filters.
- **5.12.** Wrist-action shaking device, for solvent extraction.
- **5.13.** Microgram balance.
- **5.14.** The necessary general laboratory equipment (balances, forceps, one mark pipettes, one mark volumetric flasks, etc.)

6. REAGENTS

- **6.1.** Methanol (HPLC grade)
- **6.2.** 2,2',4,4'-Tetrahydroxybenzophenone (THBP) (99%)
- **6.3.** Scopoletin (95%)
- **6.4.** Glycerol (99.5%)
- **6.5.** Helium (99.995%)
- **6.6.** Glycerol Solution with a mass fraction of 80.0%. Mix 800 g of glycerol with 200 g distilled, deionized water. (Prepare a fresh solution at least every 12 months)

7. STANDARDS

- 7.1. UVPM surrogate standard solutions: (1) *Primary standard of THBP (1000 μg/ml)* weigh 100 mg of THBP directly into a 100 ml volumetric flask, dilute to the mark with methanol, and shake to mix. (2) *Secondary standard of THBP (16 μg/ml)* transfer 4.00 ml of the primary standard to a 250 ml volumetric flask, dilute to the mark with methanol, and shake to mix.
- 7.2. Prepare five working standards of THBP covering the expected concentration range of the samples by transferring defined volumes of the secondary standard to 100 ml volumetric flasks, dilute to the mark with methanol, and shake to mix. Typical volumes are 1, 2, 5, 10, 20, and 40 ml that yield UVPM standards of THBP content of 0.16, 0.32, 0.80, 1.60, 3.20, and 6.40 μg/ml, respectively. Of these, select either the five lowest or the five highest in concentration to cover the expected concentration range of the samples.
- 7.3. FPM surrogate standard solutions: (1) *Primary standard of scopoletin (350 μg/ml)* weigh 35 mg of scopoletin directly into a 100 ml volumetric flask, dilute to the mark with methanol, and shake to mix. (2) *Secondary standard of scopoletin (3.50 μg/ml)* transfer 1.00 ml of the primary standard to a 100 ml volumetric flask, dilute to the mark with methanol, and shake to mix. This secondary standard is also the highest level working standard. (3) *Tertiary standard of scopoletin (0.350 μg/ml)* transfer 10.00 ml of the secondary standard to a 100 ml volumetric flask, dilute to the mark with methanol and shake to mix. This tertiary standard is also one of the working standards.
- 7.4. Prepare five working standards of scopoletin covering the expected concentration range of the samples by transferring defined volumes of the secondary standard and the tertiary standard to 100 ml volumetric flasks, dilute to the mark with methanol, and shake to mix. Typical volumes used are 1, and 3 ml of the tertiary standard, and 1, 3, and 30 ml of the secondary standard that yield FPM standards of scopoletin content of 0.0035, 0.0105, 0.035, 0.105, 0.350 (the tertiary standard), 1.05, and 3.50 μg/ml (the secondary standard). From this range of working standards, select either the five lowest or the five highest levels to cover the expected concentration range of the samples.

8. PROCEDURES

8.1. *Filter and Filter Cassette Preparation*

Prepare a humidity-controlled chamber ($50 \pm 2\%$ relative humidity) by placing an aqueous solution of glycerol in a tray in the bottom of a desiccator cabinet (see Ref. 3.15). Remove the top covers of individual boxes of membrane filters and place the boxes in the humidity-controlled chamber for at least 12 h prior to weighing. Calibrate and zero the microgram balance according to manufacturer's instructions. Prior to weighing, place the filter on a dust-and lint-free surface under an antistatic device for about 0.2 min. Weigh the filter to the nearest microgram on a microgram balance containing another antistatic device attached to the wall inside the weighing chamber. Repeat the last two steps until three masses are obtained for each filter, ensuring that the balance is zeroed between each individual weighing. Record the mean of the three replicate weighings as the tare mass (m_{IS}) .

Place the weighed filter inside the three-place 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). Tightly seal the prepared filter cassette containing the weighed filter and, if desired, seal the cassette with a cassette sealing band as a precaution against leaks and/or tampering, and allow the band to dry thoroughly. If the prepared filter cassette is to be used immediately, proceed to the next

step for calibration. Otherwise, plug the inlet and outlet pores of the cassette with plastic plugs provided.

8.2. Air Pumping System Calibration

Adjust the potentiometer on the air sampling pump to obtain the flow rate specified for the particular type of inertial impactor or cyclone being used. Calibrate the air sampling pump prior to and immediately after sampling. For calibration, connect the flowmeter to the inlet of the impactor or cyclone. Measure the flow with the prepared filter cassette in place between the pump and the impactor or cyclone.

Note: The flow rate through the prepared filter cassette cannot be measured with some types of cyclone in place without using specialized equipment (see Ref. 3.13). For calibration of sampling systems using these types of cyclones without the necessary specialized equipment, connect the flowmeter directly to the prepared filter cassette, and measure the flow (with the filter cassette in place between the pump and the flowmeter) prior to attaching the cyclone to the prepared filter cassette.

Record the barometric pressure and ambient temperature. If using a mass flowmeter, record the volumetric flow rate (q_V) of the air sampling pump. If using a bubble flowmeter, generate several soap-film bubbles in the flowmeter, and allow them to 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_V , expressed in liters per minute (l/min), from the following equation:

$$q_V = \frac{V_s}{t_s} \tag{1}$$

where

 $V_{\rm s}$ is the volume measured with flowmeter, expressed in liters (1);

 $t_{\rm s}$ is the average time for a soap-film bubble to travel a known volume in the bubble flowmeter, expressed in minutes (min).

8.3. *Sample Collection*

With the prepared filter cassette containing the weighed filter correctly inserted and positioned between the air sampling pump and the impactor or cyclone, turn on the pump power switch to begin sampling, and record the start time. Record the temperature and barometric pressure of the atmosphere being sampled (optional). Collect samples at the flow rate required for the impactor or cyclone in use, for a minimum time period of 1 h. Turn off the pump at the end of the desired sampling period, and record the time elapsed during sample collection (t).

Recheck the flow rate of the pump again after sample collection, and use the average flow rate, \bar{q}_v (mean of before and after sample collection), in later calculations.

Immediately remove the filter cassette containing the sample collected on the weighed filter from the sampling system, and plug the inlet and outlet ports of the cassette with plastic plugs. Treat a minimum of six prepared filter cassettes containing weighed filters in the same manner as the samples (remove plugs, measure flow, replace plugs, and

transport). Label and process these filters as field blanks. If the collected samples are not to be prepared and analyzed immediately, then store the filter cassettes containing the samples in a freezer (0 °C or less) or under dry ice, transport them frozen to the laboratory, and store frozen until analysis.

8.4. *Sample Preparation*

After sample collection, return the filter cassette containing the sample collected on the weighed filter to the weighing area. Remove the plugs, and place the filter cassette in the humidity-controlled chamber for at least 12 h prior to reweighing. Reweigh the filter following the procedure as previously described. Record the mean of the three replicate weighings as the final mass (m_{2S}). Transfer the filter to a clean sample container, and then seal and label it. Begin the UVPM and/or FPM determination immediately, or store the sealed vial in a freezer (below 0 °C) until analysis.

Add 3.00 ml of methanol (V_m) to each sample vial. Prepare field blanks in exactly the same manner as the samples. In addition, prepare and analyze two unweighed filters as laboratory blanks. Seal the vial tightly with the septum/cap assembly, and place in a holding tray. After all samples have been prepared, transfer the vials to a wrist-action shaking device, and extract under agitation for 60 min.

8.5. *High Performance Liquid Chromatography (HPLC)*

Set up the apparatus and operate the HPLC system in accordance with the manufacturer's instructions. For this method the HPLC operating conditions are as follows:

Purge gas: helium Mobile phase: methanol HPLC pump flow: 0.4 ml/min Injection volume: 50 µl

Run time: 2 min

The detector wavelength settings are as follows:

Ultraviolet detector: 325 nm

Fluorescence detector: 300 nm excitation and 420 nm emission

Under these conditions, the retention time for UVPM is about 0.5 min and for FPM (with the fluorescence detector connected in the series downstream from the UV detector) is about 0.7 min.

Allow working standards stored below room temperature to reach ambient temperature before transfer and use, observing a minimum equilibration time of 1 h. Transfer a sufficient volume (2 to 3 ml) of each working standard to a clean sample container each day for instrument calibration. Cap and tightly seal the vials. Prepare a methanol blank by transferring methanol to a sample container. Analyze this blank as a "zero" standard. Prepare the "zero" standard for each run from the methanol used for extracting samples; i.e., do not prepare it in advance and store with the other standard solutions.

Load THBP working standards at the beginning of the autosampler queue, followed by scopoletin working standards (if performing UVPM and FPM determinations simultaneously; otherwise, omit standards for the analysis not being conducted). Load the "zero" standard, samples, field blanks, and laboratory blanks in queue following the working standards. Make duplicate injections of each solution, and obtain integrated area counts for each. Compare the peak areas of samples and standards, and use the

corresponding calibration curve to calculate the concentrations of UVPM and FPM, or both, in the samples.

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

8.6. Determination

For UVPM, calculate the mean area counts obtained from duplicate injections of each working standard (y-axis, including the "zero" standard) and, together with THBP working standard concentrations (x-axis, in μ g/ml, including the "zero" standard), construct a linear regression model, and obtain the slope and y-intercept.

Note: If detector nonlinearly is significant, a weighted regression (e.g., 1/x weighting) or a second-order polynomial regression may be more appropriate.

For FPM, calculate the mean area counts obtained from duplicate injections of each working standard (y-axis, including the "zero" standard) and, together with scopoletin working standard concentrations (x-axis, in $\mu g/ml$, including the "zero" standard), construct a linear regression model, and obtain the slope and y-intercept.

Note: If detector nonlinearly is significant, a weighted regression (e.g., 1/x weighting) or a second-order polynomial regression may be more appropriate. Also, especially for FPM, ensure that detector response for all standards is within the operating range of the instrument. If not, alter detector sensitivity settings accordingly, or delete higher-level standards as necessary.

8.7. *Calculations*

The mass of RSP, m_R , expressed in micrograms (μg), is given by the equation:

$$m_R = (m_{2S} - m_{1S}) - \overline{m}_B \tag{2}$$

where

 m_{IS} is the tare mass of the filter used for sampling, expressed in micrograms (µg);

 m_{2S} is the final mass of the filter used for sampling, expressed in micrograms (µg);

is the average mass of RSP (mean of the difference between the final mass of filter used as field blank, m_{2B} , and the tare mass of filter used as field blank, m_{IB}) found in all field blanks, expressed in micrograms (µg).

The RSP content, ρ_{RA} , in the sampled air, expressed in micrograms per cubic meter $(\mu g/m^3)$, is given by the equation:

$$\rho_{RA} = \frac{m_R \times 1000}{t \times \bar{q}_V} \tag{3}$$

where

 m_R is the RSP mass, calculated by equation (2);

t is the time elapsed during sample collection, expressed in minutes (min);

 \overline{q}_V is the average volumetric flow rate of the air sampling pump, expressed in liters per minute (l/min).

If required, the RSP content found in the sampled air, adjusted to standard conditions of temperature and pressure, ρ_{RS} , expressed in micrograms per cubic meter ($\mu g/m^3$), is given by the equation:

$$\rho_{RS} = \rho_{RA} \times \frac{101.325}{p} \times \frac{T + 273}{298} \tag{4}$$

where

 ρ_{RA} is the RSP content in the sampled air, calculated by equation (3);

 ρ is the barometric pressure of the sampled air, expressed in kilopascals (kPa);

T is the temperature of the sampled air, expressed in degrees Celsius ($^{\circ}$ C);

101.325 is the standard pressure, expressed in kilopascals (kPa);

is the standard temperature, expressed in Kelvin (K).

Convert the mean area counts obtained from duplicate injections of samples and blanks to UVPM contents using the calibration curve obtained previously (UVPM is expressed as surrogate standard equivalents in micrograms per milliliter (µg/ml)).

The UVPM content, ρ_U , expressed as environmental tobacco smoke (ETS) equivalents in micrograms per milliliter (μ g/ml) of test solution, is given by the equation:

$$\rho_U = \left[\rho_{US} - \overline{\rho}_{UB} \right] \times 8.0 \tag{5}$$

where

 ρ_{US} is the UVPM content of sample, obtained from the calibration curve given previously (see 8.5), expressed in micrograms per milliliter ($\mu g/ml$) of test solution;

 ρ_{UB} is the average UVPM content of all blanks, obtained from the calibration curve given previously (see 8.5), expressed in micrograms per milliliter (μ g/ml) of blank solution; either the field blanks or the laboratory blanks may be used, whichever are deemed more appropriate;

is the conversion factor from surrogate standard to ETS equivalents (i.e., 8.0 µg of environmental tobacco smoke particulate matter (ETS-PM) has an absorbance equivalent to 1.0 µg of 2,2',4,4'-tetrahydroxybenzophenone (THBP)).

Note: 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 (see Ref. 3.5), 7.5 for the leading 50 cigarette brand styles in the United States (see Ref. 3.16), 8.2 for the leading six cigarette brand styles in each of 10 non-U.S. countries (see Ref. 3.17), and 7.2 for the leading six cigarette brand styles in each of 8 non-U.S. countries (see Ref. 3.18). 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 mass of UVPM, m_U , extracted from the filter, expressed in micrograms (µg), is given by the equation:

$$m_U = \rho_U \times V_m \tag{6}$$

where

 ρ_U is the UVPM content, calculated by equation (5);

 V_m is the volume of methanol used for extraction of filter, expressed in milliliters (ml).

The UVPM content in the sampled air, ρ_{UA} , expressed as ETS equivalents in micrograms per cubic meter ($\mu g/m^3$), is given by the equation:

$$\rho_{UA} = \frac{m_U \times 1000}{t \times \overline{q}_V} \tag{7}$$

where

 m_U is the UVPM mass, calculated by equation (6);

is the time elapsed during sample collection, expressed in minutes (min);

 q_V is the average volumetric flow rate of the air sampling pump, expressed in liters per minute (1/min).

If required, adjust the UVPM content found in the sampled air to standard conditions of temperature and pressure by the relationship given in equation (4).

If desired, divide the RSP into the fraction attributable to ETS-PM, based on ultraviolet absorbance, by calculating UVPM as a percentage of RSP in the sampled air. Of the RSP content found in the sampled air, the portion attributable to ETS (as estimated by the UVPM content found), ω_{EU} , expressed as mass fraction in percent (%), is given by the equation:

$$\omega_{EU} = \frac{\rho_{UA}}{\rho_{RA}} \times 100 \tag{8}$$

where

 ρ_{UA} is the UVPM content, calculated by equation (7);

 ρ_{RA} is the RSP content, calculated by equation (3).

Convert the mean area counts obtained from duplicate injections of samples and blanks to FPM contents using the calibration curve obtained previously (FPM is expressed as surrogate standard equivalents in micrograms per milliliter ($\mu g/ml$)).

The FPM content, ρ_F , expressed as environmental tobacco smoke (ETS) equivalents in micrograms per milliliter ($\mu g/ml$) of test solution, is given by the equation:

$$\rho_F = \left[\rho_{FS} - \overline{\rho}_{FB} \right] \times 33.6 \tag{9}$$

where

 ρ_{FS} is the FPM content of the sample, obtained from the calibration curve given previously, expressed in micrograms per milliliter (μ g/ml) of test solution;

- $\overline{\rho}_{FB}$ is the average FPM content of all blanks, obtained from the calibration curve given previously, expressed in micrograms per milliliter (μ g/ml) of blank solution; either the field blanks or the laboratory blanks may be used, whichever are deemed more appropriate;
- is the conversion factor from surrogate standard to ETS equivalents (i.e., 33.6 µg of environmental tobacco smoke particulate matter (ETS-PM) has fluorescence intensity equivalent to 1.0 µg scopoletin).

Note: 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 (see Ref. 3.5), 39.0 for the leading 50 cigarette brand styles in the United States (see Ref. 3.16), 44.2 for the leading six cigarette brand styles in each of 10 non-U.S. countries (see Ref. 3.17), and 41.8 for the leading six cigarette brand styles in each of 8 non-U.S. countries (see Ref. 3.18).. 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 mass of FPM, m_F , extracted from the filter, expressed in micrograms (μg), is given by the equation:

$$m_F = \rho_F \times V_m \tag{10}$$

where

 ρ_F is the FPM content, calculated by equation (9);

 V_m is the volume of methanol used for extraction of filter, expressed in milliliters (ml).

The FPM content in the sampled air, ρ_{FA} , expressed as ETS equivalents in micrograms per cubic meter ($\mu g/m^3$), is given by the equation:

$$\rho_{FA} = \frac{m_F \times 1000}{t \times \overline{q}_V} \tag{11}$$

where

 m_F is the FPM mass, calculated by equation (10);

is the time elapsed during sample collection, expressed in minutes (min);

 \overline{q}_V is the average volumetric flow rate of the air sampling pump, expressed in liters per minute (l/min).

If required, adjust the FPM content found in the sampled air to standard conditions of temperature and pressure by the relationship given in equation (4).

If desired, divide the RSP into the fraction attributable to ETS-PM, based on fluorescence, by calculating FPM as a percentage of RSP in the sampled air. Of the RSP content found in the sampled air, the portion attributable to ETS (as estimated by the FPM content found) ω_{EF} , expressed as a mass fraction in percent (%), is given by the equation:

$$\omega_{EF} = \frac{\rho_{FA}}{\rho_{RA}} \times 100 \tag{12}$$

where

 ρ_{FA} is the FPM content, calculated by equation (11);

 ρ_{RA} is the RSP content, calculated by equation (3).

9. POTENTIAL INTERFERENCE

Because the measured spectral properties are not unique for ETS-PM, these methods will always be a conservative measure (i.e., an overestimation) of the contribution of ETS to indoor RSP. Combustion sources are known to add significantly to the UVPM measure (see Ref. 3.19). FPM is considered to be less prone to, but not free from, interferences. As a result, these methods provide only an indication of, and not the absolute level of, the contribution of ETS to indoor RSP due to the potential presence of unquantifiable interferences.

10. REPEATABILITY AND REPRODUCIBILITY

The precision data were determined from an experiment organized and analyzed in accordance with ISO 5725-1 and ISO 5725-2 in 1998 involving 10 laboratories for RSP, 11 laboratories for UVPM and FPM, and 6 levels (see Ref. 3.20). 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 reproducibility standard deviations. Precision data were determined to vary linearly with the mean level over the range 71 μ g to 219 μ g/sample for RSP, 7.8 μ g to 28.1 μ g/sample (in surrogate standard equivalents) for UVPM, and 1.7 μ g to 8.7 μ g/sample (in surrogate standard equivalents) for FPM. These relationships are the following:

- repeatability standard deviation, $s_r = a \times m$
- reproducibility standard deviation, $s_R = A \times m$

where

m is the mean sample level, expressed in micrograms per sample (μ g/sample); a and A are as listed in Table 1.

Table 1 — Values of a and A

Analyte	а	A
RSP	0.072	0.089
UVPM	0.018	0.086
FPM	0.048	0.144

11. LIMITS AND DETECTION

The methods specified allow the estimation of RSP content to within the following limits. At a sampling rate of 2 l/min over 1 h, the UVPM test method shows limits of detection (LOD) and quantification (LOQ) of $2.5 \,\mu\text{g/m}^3$ and $8.3 \,\mu\text{g/m}^3$, respectively. Under the same conditions, the FPM method shows an LOD and LOQ of $1.4 \,\mu\text{g/m}^3$ and $4.7 \,\mu\text{g/m}^3$, respectively (See Ref. 3.13).