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Environmental tobacco smoke — Estimation of its contribution to respirable suspended particles — Determination of particulate matter by ultraviolet absorbance and by fluorescence

Fumée de tabac ambiante — Estimation de sa contribution aux particules respirables suspendues dans l'air — Détermination de la matière particulaire par absorption dans l'ultraviolet et par fluorescence

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15593 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

Annex A of this International Standard is for information only.

Introduction

Environmental tobacco smoke (ETS) is an aerosol consisting of vapour 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 reference [1]).

NOTE The bibliography gives full references to the literature cited. References to the literature are given in the text for information for the user of this International Standard.

Ultraviolet particulate matter (UVPM) and fluorescent particulate matter (FPM) fulfil this requirement, staying in a constant 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 vapour phase) does not remain in a consistent ratio to ETS particulate matter (ETS-PM) (see reference [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, bacteria, etc. (see reference [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 references [4] to [7]). The test methods described in this International Standard have been used effectively to reduce the uncontrollable bias inherent in the use of RSP as a tracer of ETS (see references [4] to [6], and [8] to [13]).

Because the measured spectral properties are not unique to 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 reference [14]). FPM is considered to be less prone to, but not free from, interferences. As a result, these methods provide only an indication, and not the absolute level, of the contribution of ETS to indoor RSP due to the potential presence of unquantifiable interferences.

Environmental tobacco smoke — Estimation of its contribution to respirable suspended particles — Determination of particulate matter by ultraviolet absorbance and by fluorescence

1 Scope

This International Standard specifies methods for the sampling and determination of respirable suspended particles (RSP) for the estimation of the RSP fraction attributable to environmental tobacco smoke (ETS).

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 648, *Laboratory glassware — One-mark pipettes*.

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3696, *Water for analytical use — Specification and test methods*.

3 Terms, definitions and abbreviated terms

For the purposes of this International Standard, the following terms and definitions apply.

3.1
environmental tobacco smoke
ETS
mixture of aged and diluted exhaled mainstream smoke and aged and diluted sidestream smoke

3.2
respirable suspended particles
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 µm

NOTE See ISO 7708 [15].

3.3
ultraviolet particulate matter
UVPM
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

3.4
fluorescent particulate matter
FPM
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

3.5
environmental tobacco smoke particulate matter
ETS-PM
particulate phase of ETS

3.6
surrogate standard
chemical whose concentration has been related quantitatively to a known concentration in the solution of ETS-PM

EXAMPLES 2,2′,4,4′-Tetrahydroxybenzophenone (THBP) for UVPM; scopoletin for FPM.

4 Principle

A known volume of air is drawn through an inertial impactor or cyclone separating at 4,0 µm, thus separating the respirable suspended particles (RSP) from the total suspended particulate matter. It is then drawn through a filter cassette containing a polytetrafluoroethylene (PTFE) membrane filter. The RSP are collected on the filter, followed by gravimetric determination of the mass of RSP so collected. The RSP are extracted from the filter for the determination of UVPM and FPM by absorbance and fluorescence measurements, respectively, using high-performance liquid chromatography (HPLC) apparatus.

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

5 Limits and detection

The methods specified in this International Standard 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 µg/m^3 and 8,3 µg/m^3, respectively. Under the same conditions, the FPM method shows an LOD and LOQ of 1,4 µg/m^3 and 4,7 µg/m^3, respectively.

6 Reagents

All reagents shall be of recognized analytical grade. Water shall be in accordance with at least grade 3 of ISO 3696.

6.1 Methanol, HPLC grade.

6.2 2,2′,4,4′-Tetrahydroxybenzophenone (THBP), of minimum purity 99 %.

6.3 Scopoletin, of minimum purity 95 %.

6.4 Glycerol, of minimum purity 99,5 %.

6.5 Helium, of minimum purity 99,995 %.

6.6 UVPM surrogate standard solutions

Store all standard solutions in low-actinic borosilicate glass screw-cap jars in a refrigerator (at about 4 °C) when not in use. Prepare fresh standards from THBP at least every 12 months.

6.6.1 Primary standard of THBP (1 000 µg/ml), prepared by weighing 100 mg of THBP (6.2) directly into a 100 ml volumetric flask, diluting to the mark with methanol, and shaking to mix.

6.6.2 Secondary standard of THBP (16 µg/ml), prepared by transferring 4,00 ml of the primary standard (6.6.1) to a 250 ml volumetric flask, diluting to the mark with methanol, and shaking to mix.

6.6.3 Working standards of THBP

Prepare five working standards covering the expected concentration range of the samples by transferring defined volumes of the secondary standard (6.6.2) to 100 ml volumetric flasks, diluting to the mark with methanol, and shaking to mix.

Typical volumes used are 1 ml, 2 ml, 5 ml, 10 ml, 20 ml and 40 ml that yield UVPM standards of THBP content of 0,16 µg/ml, 0,32 µg/ml, 0,80 µg/ml, 1,60 µg/ml, 3,20 µg/ml and 6,40 µg/ml, respectively. Of these, select either the five lowest or the five highest in concentration to cover the expected range of samples.

6.7 FPM surrogate standard solutions

Store all standard solutions in low-actinic borosilicate glass screw-cap jars in a refrigerator (at about 4 °C) when not in use. Prepare fresh standards from scopoletin at least every 6 months.

6.7.1 Primary standard of scopoletin (350 µg/ml), prepared by weighing 35 mg of scopoletin [assuming 100 % purity scopoletin (6.3)] directly into a 100 ml volumetric flask, diluting to the mark with methanol, and shaking to mix.

6.7.2 Secondary standard of scopoletin (3,50 µg/ml), prepared by transferring 1,00 ml of the primary standard (6.7.1) to a 100 ml volumetric flask, diluting to the mark with methanol, and shaking to mix. This secondary standard is also the highest level working standard.

6.7.3 Tertiary standard of scopoletin (0,350 µg/ml), prepared by transferring 10,00 ml of the secondary standard (6.7.2) to a 100 ml volumetric flask, diluting to the mark with methanol, and shaking to mix. This tertiary standard is also one of the working standards.

6.7.4 Working standards of scopoletin

Prepare five working standards covering the expected concentration range of the samples by transferring defined volumes of the secondary standard (6.7.2) and the tertiary standard (6.7.3) to 100 ml volumetric flasks, diluting to the mark with methanol, and shaking to mix.

Typical volumes used are 1 ml and 3 ml of the tertiary standard and 1 ml, 3 ml and 30 ml of the secondary standard, that yield FPM standards of scopoletin content of 0,003 5 µg/ml, 0,0105 µg/ml, 0,035 µg/ml, 0,105 µg/ml, 0,350 µg/ml (the tertiary standard), 1,05 µg/ml 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.

6.8 Glycerol solution

Prepare an aqueous solution of glycerol with a mass fraction of 80,0 % by mixing 800 g of glycerol (6.4) with 200 g distilled, deionized water. Prepare a fresh solution at least every 12 months.

7 Apparatus

Usual laboratory apparatus and, in particular, the following items.

7.1 Sample collection system

7.1.1 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.

7.1.2 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.

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, constant-flow air sampling pump, calibrated for a flow rate dependent upon the separating characteristics of the impactor or cyclone in use (7.1.6).

7.1.6 Inertial impactor or **cyclone**, with nominal cut point of 4,0 µm at the specified flow rate.

If the alternative definition of RSP is used (see 3.2), 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 High-performance liquid chromatography (HPLC) system, consisting of a solvent-delivery system, autosampler, ultraviolet detector, fluorescence detector, peak integration system, and 3,0 m stainless-steel tubing with 0,23 mm inside diameter.

No HPLC analytical column is used. If this analysis is attempted using an ultraviolet spectrometer, a cell with path length of at least 40 mm is recommended.

7.2.2 Sample containers, consisting of low-actinic borosilicate glass autosampler vials, of 4 ml capacity, with screw caps and PTFE-lined septa.

7.3 Microgram balance, for weighing filters, accurate to 1 µg.

7.4 Desiccator cabinet, for use as a humidity-controlled chamber where filters are stored prior to weighing.

7.5 Static inhibitor, for removing static charge from filters.

7.6 Filter forceps, for handling filters.

7.7 Shaking device, with wrist-action for solvent extraction.

7.8 One-mark pipettes, complying with class A of ISO 648.

7.9 One-mark volumetric flasks, complying with class A of ISO 1042.

8 Sampling procedure

8.1 Filter and filter cassette preparation

Prepare a humidity-controlled chamber [(50 ± 2) % relative humidity] by placing an aqueous solution of glycerol (6.8) in a tray in the bottom of the desiccator cabinet (7.4) (see reference [16]). Remove the top covers of individual boxes of membrane filters (7.1.1), and place the boxes in the humidity-controlled chamber for at least 12 h prior to weighing.

Calibrate and zero the microgram balance (7.3) according to the manufacturer's instructions. Prior to weighing, place the filter on a dust- and lint-free surface under an antistatic device (7.5) for about 0,2 min.

Weigh the filter to the nearest microgram on a microgram balance (7.3) containing another antistatic device attached to the wall inside the weighing chamber.

Handle the filter with clean forceps only.

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_{1S}).

Place the weighed filter inside the three-piece filter cassette (7.1.2), with the filter backing (7.1.1) facing the cassette outlet (bottom piece), and with the spacer ring (centre 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. Allow the band to dry thoroughly. If the prepared filter cassette is to be used immediately, proceed to the next step for calibration (see 8.2). Otherwise, plug the inlet and outlet ports of the cassette with the plastic plugs provided.

NOTE The three-piece filter cassette (with a spacer ring in the centre) is not always needed.

8.2 Calibration of air pumping system

Adjust the potentiometer on the air sampling pump (7.1.5) to obtain the flow rate specified for the particular type of inertial impactor or cyclone (7.1.6) being used.

Calibrate the air sampling pump prior to and immediately after sampling. For calibration, connect the flowmeter (7.1.4) 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.

The flow rate through the prepared filter cassette cannot be measured with some types of cyclone in place without using specialized equipment (see reference [13]). For calibration of sampling systems using these types of cyclone 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 with a stopwatch the time for a soap-film bubble to travel a known volume. Obtain five replicate measurements and compute the mean time.

Calculate the volumetric flow rate, q_V, expressed in litres per minute (l/min), from the following equation:

$$q_V = \frac{V_S}{t_S} \qquad (1)$$

where

V_S is the volume measured with flowmeter, expressed in litres (l);

t_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 (7.1.2) 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.

NOTE 1 Some pumps have microprocessing capabilities and/or built-in elapsed time meters for preset sampling periods.

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

Collect samples at the flow rate required for the impactor or cyclone (7.1.6) 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).

NOTE 2 This test method is limited in sampling period only by the capacity of the membrane filter for total collected mass (about 2 000 µg). This test method has been evaluated up to a 24 h sampling period. A minimum sampling period of at least 1 h is recommended.

Recheck the flow rate of the pump again after sample collection, and use the average flow rate, $\overline{q_V}$ (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 then transport). Label and process these filters as field blanks.

If the collected samples are not to be prepared and analysed immediately, then store the filter cassettes containing the samples in a freezer (at 0 °C or less) or under dry ice, transport them frozen to the laboratory, and store them frozen until analysis.

Analyse all the filters within 6 weeks after sample collection. It has been established that samples are stable for at least 6 weeks at –10 °C storage conditions.

9 Analytical procedure

9.1 Determination of sample mass

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

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

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 described in 8.1. Record the mean of the three replicate weighings as the final mass (m_{2S}).

Transfer the filter to a clean sample container (see 7.2.2), 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.

9.2 Preparation of samples and blanks

If samples and field blanks stored in the sealed vials were stored in a freezer, allow them to reach room temperature before adding the methanol.

Add 3,00 ml of methanol (V_m) to each sample vial (see 9.1). Prepare field blanks in exactly the same manner as the samples. In addition, prepare and analyse two unweighed filters as laboratory blanks.

If high concentration samples are being analysed, 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.

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 the shaking device (7.7), and extract under agitation for 60 min.

9.3 Determination of UVPM and FPM

9.3.1 Setting up the apparatus

Set up the apparatus and operate the high-performance liquid chromatography system in accordance with the manufacturer's instructions.

The HPLC operating conditions are

— purge gas: helium,

— mobile phase: methanol,

— HPLC pump flow rate: 0,4 ml/min,

— injection volume: 50 µl,

— run time: 2 min.

The detector wavelength settings are

— 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 series downstream from the UV detector) it is about 0,7 min.

9.3.2 Analysis of samples and blanks

Allow working standards stored below room temperature to reach room temperature before transfer and use, observing a minimum equilibration time of 1 h. Transfer a sufficient volume (2 ml to 3 ml) of each working standard to a clean sample container (7.2.2) each day for instrument calibration. Cap and tightly seal the vials.

Prepare a methanol blank by transferring methanol (6.1) to a sample container. Analyse 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 the THBP working standards (see 6.6.3) at the beginning of the autosampler queue, followed by scopoletin working standards (see 6.7.4) (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 (see 9.2) 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 or FPM, or both, in the samples.

It is acceptable to either use 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.

9.3.3 Constructing the calibration curves

9.3.3.1 UVPM calibration curve

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 (6.6.3) (x-axis, in micrograms per millilitre, including the "zero" standard), construct a linear regression model, and obtain the slope and y-intercept.

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

9.3.3.2 FPM calibration curve

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 (6.7.4) (x-axis, in micrograms per millilitre, including the "zero" standard), construct a linear regression model, and obtain the slope and y-intercept.

If detector nonlinearity is significant, a weighted regression 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 the detector sensitivity settings accordingly, or delete higher-level standards as necessary.

10 Expression of results

10.1 Calculation of RSP mass in the sample

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

$$m_R = (m_{2S} - m_{1S}) - \overline{m_B} \quad (2)$$

where

m_{1S} is the tare mass (see 8.1) of the filter used for sampling, expressed in micrograms (µg);

m_{2S} is the final mass (see 9.1) of the filter used for sampling, expressed in micrograms (µg);

$\overline{m_B}$ 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_{1B}) found in all field blanks (see 8.3), expressed in micrograms (µg).

10.2 Calculation of RSP content in the air

The RSP content, ρ_{RA}, in the sampled air, expressed in micrograms per cubic metre (µg/m^3), is given by the equation:

$$\rho_{RA} = \frac{m_R \times 1\,000}{t \times \overline{q_V}} \quad (3)$$

where

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

t is the time elapsed during sample collection (see 8.3), expressed in minutes (min);

$\overline{q_V}$ is the average volumetric flow rate (see 8.2 and 8.3) of the air sampling pump, expressed in litres per minute (l/min).

10.3 Calculation of RSP content in the air in relation to standard conditions of temperature and pressure

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 metre (µg/m^3), is given by the equation:

$$\rho_{RS} = \rho_{RA} \times \frac{101{,}325}{p} \times \frac{T + 273}{298} \quad (4)$$

where

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

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

T is the temperature of the sampled air, expressed in degrees Celsius (°C);

101,325 is the standard pressure, expressed in kilopascals (kPa);

298 is the standard temperature, expressed in kelvin (K).

10.4 Calculation of UVPM contents

10.4.1 UVPM content in the test solution

Convert the mean area counts obtained from duplicate injections of samples and blanks to UVPM contents using the calibration curve obtained in 9.3.3.1 (UVPM is expressed as surrogate standard equivalents in micrograms per millilitre).

The UVPM content, ρ_U, expressed as environmental tobacco smoke (ETS) equivalents in micrograms per millilitre of test solution (see 9.2), is given by the equation:

$$\rho_U = \left[\rho_{US} - \overline{\rho_{UB}} \right] \times 8{,}0 \quad (5)$$

where

ρ_{US} is the UVPM content of sample, obtained from the calibration curve given in 9.3.3.1, expressed in micrograms per millilitre of test solution;

$\overline{\rho_{UB}}$ is the average UVPM content of all blanks (see 8.3 or 9.2), obtained from the calibration curve given in 9.3.3.1, expressed in micrograms per millilitre of blank solution; either the field blanks (see 8.3) or the laboratory blanks (see 9.2) may be used, whichever are deemed more appropriate;

8,0 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. [5]), 7,5 for the leading 50 cigarette brand styles in the United States (see ref. [17]), and 8,2 for the leading six cigarette brand styles in each of 10 European countries (see ref. [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:

$$m_U = \rho_U \times V_m \quad (6)$$

where

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

V_m is the volume of methanol used for extraction of filter (see 9.2), expressed in millilitres (ml).

10.4.2 UVPM content in the air

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

$$\rho_{UA} = \frac{m_U \times 1000}{t \times \overline{q_V}} \quad (7)$$

where

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

t is the time elapsed during sample collection (see 8.3), expressed in minutes (min);

$\overline{q_V}$ is the average volumetric flow rate (see 8.2 and 8.3) of the air sampling pump, expressed in litres per minute (l/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).

10.4.3 ETS-PM contribution to RSP based on ultraviolet absorbance

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:

$$\omega_{EU} = \frac{\rho_{UA}}{\rho_{RA}} \times 100 \quad (8)$$

where

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

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

10.5 Calculation of FPM contents

10.5.1 FPM content in the test solution

Convert the mean area counts obtained from duplicate injections of samples and blanks to FPM contents using the calibration curve obtained in 9.3.3.2 (FPM is expressed as surrogate standard equivalents in micrograms per millilitre).

The FPM content, ρ_F, expressed as environmental tobacco smoke (ETS) equivalents in micrograms per millilitre of test solution (see 9.2), is given by the equation:

$$\rho_F = \left[\rho_{FS} - \overline{\rho_{FB}} \right] \times 33,6 \qquad (9)$$

where

ρ_{FS} is the FPM content of the sample, obtained from the calibration curve given in 9.3.3.2, expressed in micrograms per millilitre of test solution;

$\overline{\rho_{FB}}$ is the average FPM content of all blanks (see 8.3 or 9.2), obtained from the calibration curve given in 9.3.3.2, expressed in micrograms per millilitre of blank solution; either the field blanks (see 8.3) or the laboratory blanks (see 9.2) may be used, whichever are deemed more appropriate;

33,6 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. [5]), 39,0 for the leading 50 cigarette brand styles in the United States (see ref. [17]), and 44,2 for the leading six cigarette brand styles in each of 10 European countries (see ref. [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:

$$m_F = \rho_F \times V_m \qquad (10)$$

where

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

V_m is the volume of methanol used for extraction of filter (see 9.2), expressed in millilitres (ml).

10.5.2 FPM content in the air

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

$$\rho_{FA} = \frac{m_F \times 1000}{t \times \overline{q_V}} \qquad (11)$$

where

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

t is the time elapsed during sample collection (see 8.3), expressed in minutes (min);

$\overline{q_V}$ is the average volumetric flow rate (see 8.2 and 8.3) of the air sampling pump, expressed in litres 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).

10.5.3 ETS-PM contribution to RSP based on fluorescence

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:

$$\omega_{EF} = \frac{\rho_{FA}}{\rho_{RA}} \times 100 \qquad (12)$$

where

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

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

11 Laboratory performance criteria and quality assurance

Guidance concerning performance criteria and a summary of quality assurance measures that should be achieved within each laboratory are provided in annex A.

12 Repeatability and reproducibility

The precision data were determined from an experiment organized and analysed 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. 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 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:

— repeatability standard deviation, $s_r = a \cdot m$

— reproducibility standard deviation, $s_R = A \cdot m$

where

m is the mean sample level, expressed in micrograms per sample;

a and A are as listed in Table 1.

Table 1 — Values of a and A

Analyte	a	A
RSP	0,072	0,089
UVPM	0,018	0,086
FPM	0,048	0,114

13 Test report

The test report shall give the ambient UVPM, FPM and RSP concentrations in micrograms per cubic metre, and shall include all conditions which may affect the result (e.g. atmosphere, sampling time and sampling rate). It shall also give all details necessary for the identification of the atmosphere under test. For UVPM or FPM, or both, the conversion factor applied in equation (5) or equation (9), or both, shall also be specified.

Annex A
(informative)

Laboratory performance criteria — Quality assurance measures

A.1 Standard operating procedures (SOPs)

A.1.1 Users should generate SOPs describing and documenting the following activities in their laboratory:

a) assembly, calibration, leak-check and operation of the specific sampling system and equipment used;

b) preparation, storage, shipment and handling of samples;

c) assembly, leak-check, calibration and operation of the analytical system, addressing the specific equipment used;

d) all aspects of data recording and processing, including lists of computer hardware and software used.

A.1.2 The SOPs should provide specific, step-by-step instructions and should be readily available to, and understood by, the laboratory personnel conducting the work.

A.1.3 Sample blanks should contain less than the equivalent of 0,5 µg of ETS particulate matter (UVPM and/or FPM). Larger quantities would be evidence of contamination during sampling or analysis.

A.1.4 Periodically, wipe clean the surface of the inertial impactor and apply a thin coat of stopcock grease. If a cyclone is used, empty the grit pot prior to each use, and ensure that the cyclone remains upright (i.e. it should never turn past horizontal) during sampling.

A.1.5 In the event an initial sample result is above the calibration range, prepare and analyse additional standards, or dilute and re-analyse the sample.

A.2 Calibration of personal sampling pumps

A.2.1 Calibrate sampling pumps at the beginning and at the end of each sampling period.

A.2.2 Set the pump flow controller using a bubble flowmeter or mass flowmeter at the appropriate sampling rate (i.e. at a rate depending on the separating characteristics of the impactor or cyclone in use) at standard conditions (optional) with the prepared filter cassette in place.

A.2.3 For conversion of measured flows to standard flows, record the barometric pressure and ambient temperature during both pump calibration and sampling.

A.3 Method sensitivity, precision, and linearity

A.3.1 The sensitivity of the 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 sampling period.

A.3.2 Nonlinearity in the calibration curve can occur at concentrations near the upper useable 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 and thus to require additional dilution and re-analysis.

A.4 Method modification

A.4.1 The sampling period described in these methods may be extended beyond 24 h provided that the capacity of the filter is not exceeded. Also, a sampling period of less than 1 h may be used in areas of very high ETS-PM concentration (e.g. in an environmental test chamber).

A.4.2 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.

A.4.3 The test solution resulting from the procedure described herein is also compatible with the determination of solanesol, a high-molecular-weight isoprenoid alcohol which has also been used as a tracer of the particulate phase of ETS (see refs. [14], [21]).

A.5 Safety

A.5.1 If spilling of THBP reagent, scopoletin reagent, or solvent occurs, take quick and appropriate cleanup action. (See Material Safety Data Sheet that is provided by the seller of the chemical as prescribed by law.)

A.5.2 When preparing standards, as with handling any chemicals, avoid contact with skin and eyes.

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