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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:

$$q_V = \frac{V_S}{t_S} \tag{1}$$

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).