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Environmental tobacco smoke — Estimation of its contribution to respirable suspended particles — Method based on solanesol

Fumée de tabac ambiante — Estimation de sa contribution aux particules en suspension respirables — Méthode basée sur le solanésol

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18144 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

Introduction

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

NOTE The Bibliography gives full references to the literature cited.

Solanesol, a C_{45} isoprenoid alcohol, meets this requirement, since it remains in a constant ratio to respirable suspended particles (RSP) contributed by tobacco smoke over a variety of ventilation conditions and sampling durations (see [2]). Ultraviolet particulate matter (UVPM) and fluorescent particulate matter (FPM), determined in accordance with ISO 15593[3], are tracers or markers which also fulfil 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 mass 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 mass of the RSP of ETS (see [4] to [6]), 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 (ETS-PM) to RSP (see [7] to [15]).

It is important to be able to quantify the contribution of ETS to RSP with a tobacco-specific marker because RSP is not specific to tobacco smoke. RSP is a necessary indicator of overall air quality; in the United States, the Occupational Safety and Health Administration (OSHA) has previously set a PEL (permissible exposure level) for respirable dust in the workplace of 5 000 $\mu g/m^3$. However, RSP emanates from numerous sources (see [16]) and has been shown to be an inappropriate tracer of ETS (see [4], [17] to [19]). 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 non-tobacco combustion sources. Although UVPM and FPM are useful in investigations of indoor air quality, solanesol is a better indicator of the tobacco smoke contribution to RSP. The test method described in this International Standard has been used to apportion RSP into ETS and non-ETS components by determining the mass ratio of solanesol to total RSP (see [4], [6], [10], [11], [14], [15], [20], [21]).

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 possible source of interference, the potential for interference is negligible. However, if there were an interference of this type, the mass 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. Solanesol concentrations typically range from not detected to 2 $\mu g/m^3$ in various indoor environments, with most levels at the lower end of this range.

Environmental tobacco smoke — Estimation of its contribution to respirable suspended particles — Method based on solanesol

1 Scope

This International Standard specifies a method for the sampling and determination of respirable suspended particles (RSP) and for the estimation of the RSP fraction attributable to environmental tobacco smoke (ETS). This method is applicable to personal and area sampling. This method is compatible with the determinations of gravimetric RSP, ultraviolet particulate matter (UVPM) and fluorescent particulate matter (FPM), which are also used to estimate the contribution of ETS to RSP.

NOTE See ISO 15593[3] for details.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

3 Terms and definitions

For the purposes of this document, 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[22].

3.3
environmental tobacco smoke particulate matter
ETS-PM
particulate phase of ETS

3.4
solanesol particulate matter
Sol-PM
estimation of the contribution of ETS-PM to RSP, based on the determination of a tobacco-specific compound: solanesol

4 Principle

A known volume of air is drawn through an inertial impactor or cyclone separating at 4,0 μm, thus separating RSP from the total suspended particulate matter. It is then drawn through a filter cassette containing a polytetrafluoroethylene (PTFE) membrane filter. Solanesol is collected on the filter as a component of RSP. The solanesol is extracted from the filter with methanol, then the extract is injected into a high-performance liquid chromatography (HPLC) system equipped with an ultraviolet (UV) detector (205 nm absorbance). The area of the resulting solanesol peak is compared to the areas obtained from the injection of standard solutions of solanesol, and the mass of solanesol is determined. The concentration of solanesol ($\mu g/m^3$) is calculated from the mass of solanesol and the volume of air sampled. The concentration of RSP attributable to ETS, referred to as Sol-PM, is calculated from the airborne concentration of solanesol and the experimentally determined mass ratio of solanesol to RSP in ETS (see [6], [24], [25]). If desired, the total concentration of RSP (see ISO 15593[3]) may be calculated for apportionment of RSP into ETS and non-ETS fractions.

5 Limits and detection

The method specified in this International Standard allows the estimation of solanesol content within the following limits. At a sampling rate of 2 l/min, this test method shows limits of detection (LOD) and quantification (LOQ) (see [13]) of 0,042 $\mu g/m^3$ and 0,139 $\mu g/m^3$, respectively, for a 1 h sampling period, and 0,005 $\mu g/m^3$ and 0,017 $\mu g/m^3$, respectively, for an 8 h sampling period.

6 Reagents

All reagents shall be of recognized analytical grade.

6.1 Acetonitrile, HPLC grade.

6.2 Methanol, HPLC grade.

6.3 Solanesol, minimum purity 90 %.

6.4 Helium, minimum purity 99,995 % grade.

6.5 Solanesol standard solutions

Store all standard solutions in low-actinic borosilicate glass screw-cap jars in a freezer (0 °C or less) when not in use. Prepare fresh standards from solanesol at least every 12 months.

6.5.1 Primary standard of solanesol

Prepare a primary standard of solanesol (300 μg/ml) by weighing 30 mg of solanesol (assuming 100 % solanesol purity; 6.3) directly into a 100 ml volumetric flask. Dilute to the mark with methanol (6.2) and shake to mix.

Actual concentration of standard solutions will depend on the exact mass and purity of the solanesol reagent used (6.3 and 6.5.1). Obtain the solanesol purity from the vendor for the specific lot of solanesol used. The actual purity of the solanesol reagent used shall be taken into account when calculating the exact concentration of the standard solutions prepared.

6.5.2 Secondary standard of solanesol

Prepare a secondary standard of solanesol (15 μg/ml) by transferring 5,00 ml of the primary standard to a 100 volumetric flask. Dilute to the mark with methanol (6.2) and shake to mix.

6.5.3 Tertiary standard of solanesol

Prepare a tertiary standard of solanesol (6 μg/ml) by transferring 2,00 ml of the primary standard to a 100 ml volumetric flask. Dilute to the mark with methanol (6.2) and shake to mix.

6.5.4 Working standards of solanesol

Prepare five working standards covering the expected concentration range of the samples by transferring defined volumes of the tertiary, secondary, and primary standards to 100 ml volumetric flasks. Dilute to the mark with methanol (6.2) and shake to mix. Typical volumes used are 1 ml of the tertiary standard (6.5.3); 1 ml, 3 ml, and 7 ml of the secondary standard (6.5.2); and 1 ml of the primary standard (6.5.1). These volumes yield a calibration range of solanesol standards with the following concentrations of solanesol: 0,060 μg/ml, 0,150 μg/ml, 0,450 μg/ml, 1,05 μg/ml and 3,00 μg/ml.

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 plastic tubing.

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

7.1.3 Bubble flowmeter or **mass flowmeter**, for calibration of the sampling pump.

7.1.4 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 (7.1.5).

7.1.5 Inertial impactor or **cyclone**, with nominal cut point of 4,0 μm at the specified flow rate. If an alternative definition of RSP is used (3.2), ensure that the impactor or cyclone is compatible with this definition.

7.1.6 Stopcock grease, for coating impactor plates.

7.2 Analytical system

7.2.1 High-performance liquid chromatography (HPLC) system, consisting of an HPLC pump, a 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 inside diameter, reversed-phase C_{18} column (of pore size 30 nm and particle size 5 μm). C_{18} packing material with low carbon loading has been found to be preferable.

7.2.3 Guard cartridge column, with packing material and dimensions compatible with the HPLC column (7.2.2), placed in front of the analytical column for protecting and prolonging the life of the column.

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

7.3 Dispensing pipettes, 3,00 ml.

7.4 Filter forceps, for handling filters.

7.5 Wrist-action shaking device, for solvent extraction.

7.6 One-mark pipettes, complying with class A of ISO 648:1977.

7.7 One-mark volumetric flasks, complying with class A of ISO 1042:1998.

8 Sampling procedure

8.1 Calibration of air pumping system

If a gravimetric determination of RSP is to be performed, then weigh the filters in accordance with ISO 15593[3], prior to calibrating the air pumping system.

Adjust the potentiometer on the air sampling pump (7.1.4) to obtain the flow rate specified for the particular type of inertial impactor or cyclone (7.1.5) being used.

Calibrate the air sampling pump prior to and immediately after sampling. For calibration, connect the flowmeter (7.1.3) to the inlet of the inertial 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 cyclones in place without using specialized equipment (see [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.

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 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_V, expressed in litres per minute (l/min), from the following equation:

$$q_V = \frac{V}{t} \tag{1}$$

where

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

t is the average time for a soap-film bubble to travel V litres in the bubble flowmeter, expressed in minutes (min).

8.2 Sample collection

With the prepared filter cassette (7.1.2) 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 Some pumps have microprocessing capabilities and/or built-in elapsed time meters for preset sampling periods.

Collect samples at the flow rate required for the impactor or cyclone (7.1.5) 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.

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 membrane filter (7.1.1) 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 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 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 frozen until analysis.

Analyse 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 (see [23]).

9 Analytical procedure

9.1 Preparation of samples and blanks

If a gravimetric determination of RSP is to be performed, then reweigh the filters in accordance with ISO 15593[3], prior to preparing the samples and blanks.

Place each filter in a clean sample vial (7.2.4), label the vial and add 3,00 ml of methanol (V_m). Prepare field blanks in exactly the same manner as the samples. In addition, prepare and analyse two unweighed filters as laboratory blanks.

If samples and field blanks were stored frozen, allow them to reach room temperature before adding the methanol.

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 (7.5), and extract under agitation for 60 min.

9.2 Determination of solanesol

9.2.1 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 is equipped with a UV detector. 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.

Use the following operating conditions:

— purge gas: helium;

— mobile phase: 95:5 (by volume) of acetonitrile:methanol;

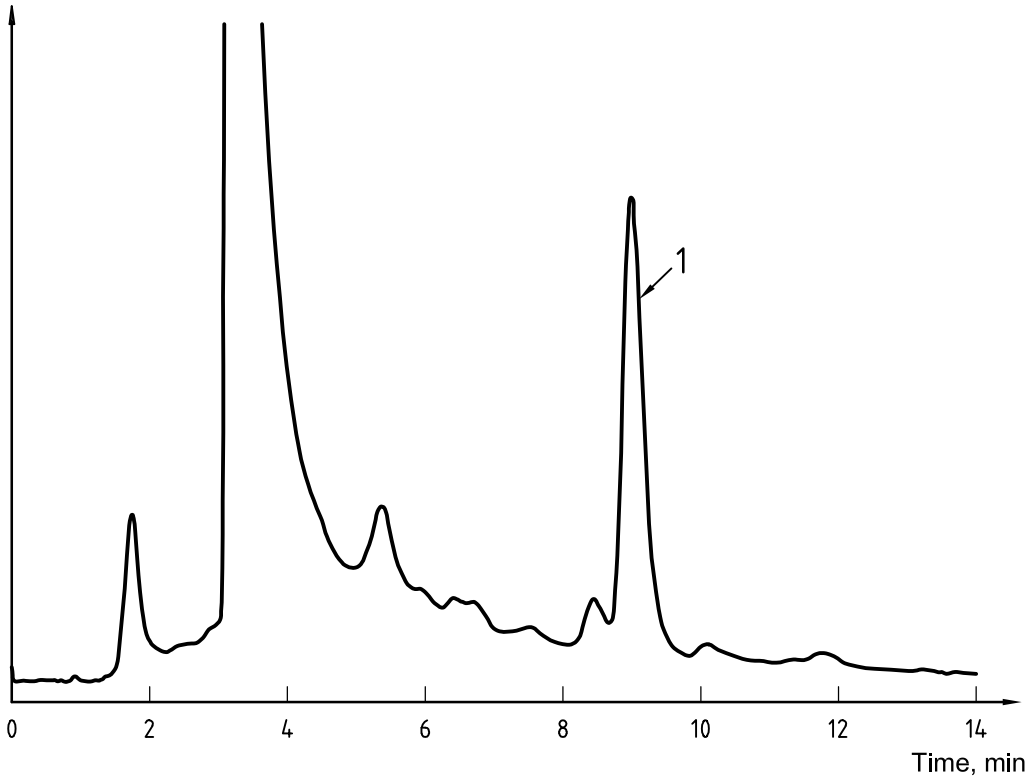
— HPLC pump flow: 0,5 ml/min;

— injection volume: 100 µl;

— run time: 15 min;

— detector wavelength setting: 205 nm.

Under these conditions using the HPLC column and guard column specified (7.2.2 and 7.2.3), the retention time for solanesol is about 9 min. Figure 1 shows a typical chromatogram from an ETS sample.



Peak identification

1 solanesol

Figure 1 — Example of a chromatogram of an environmental tobacco smoke (ETS) sample

9.2.2 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 vial (7.2.4) each day for instrument calibration. Cap and tightly seal the vials.

Load one set of solanesol working standards (6.5.4) at the beginning of the autosampler queue. Next, load all samples, field blanks and laboratory blanks (9.1) in queue following the working standards. Load a second set of working standards at the end of the autosampler queue.

Make injections of each solution and obtain integrated peak area counts for all standards, samples and blanks using the data acquisition system and peak integration system. Compare the peak areas of the samples and standards, and use the corresponding calibration curve to calculate the concentrations of solanesol in the samples.

9.2.3 Constructing the solanesol calibration curve

Construct the solanesol calibration curve by plotting the mean peak area of solanesol (y-axis) versus the concentration of solanesol (in micrograms per millilitre on the x-axis) in the working standards (6.5.4). Using a linear regression model, obtain the slope and y-intercept.

NOTE If detector non-linearity is significant, a weighted regression (for example, $1/x$ weighting) and/or a second-order polynomial regression may be more appropriate.

10 Expression of results

10.1 Calculation of solanesol contents

10.1.1 Solanesol content in the test solution

Convert the area counts obtained from injections of samples and blanks to solanesol contents (μg/ml) using the calibration curve obtained in 9.2.3.

The solanesol content, ρ_S, expressed in micrograms per millilitre of test solution (μg/ml) (see 9.1), is given by the equation

$$\rho_S = \rho_{Ss} - \rho_{Sb} \quad (2)$$

where

ρ_{Ss} is the solanesol content of sample, obtained from the calibration curve given in 9.2.3, expressed in micrograms per millilitre of test solution (μg/ml);

ρ_{Sb} is the average solanesol content of all blanks (see 8.2 or 9.1), obtained from the calibration curve given in 9.2.3, expressed in micrograms per millilitre of blank solution (μg/ml).

Either the field blanks (see 8.2) or the laboratory blanks (see 9.1) may be used, whichever are deemed more appropriate.

The mass of solanesol, m_S, extracted from the filter, expressed in micrograms (μg), is given by the equation

$$m_S = \rho_S \times V_m \quad (3)$$

where

ρ_S is the solanesol content, calculated by Equation (2);

V_m is the volume of methanol used for extraction of filter (see 9.1), expressed in millilitres (ml).

10.1.2 Solanesol content in the air

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

$$\rho_{Sa} = \frac{m_S \times 1\,000}{t \times \overline{q}_V} \quad (4)$$

where

m_S is the mass of solanesol, calculated by Equation (3);

t is the time elapsed during sample collection (see 8.2), expressed in minutes (min);

$\overline{q}_V$ is the average volumetric flow rate (see 8.1 and 8.2) of the air sampling pump, expressed in litres per minute (l/min).

1 000 is the conversion factor for the conversion of litres to cubic metres, in the sampled air, expressed in litres per cubic metre (l/m^3);

10.2 ETS-PM contribution to RSP as estimated by solanesol

10.2.1 Calculation of Sol-PM

The RSP content, ρ_{SP}, attributable to ETS-PM content in the sampled air, based on determining solanesol content in the sampled air and on the fact that solanesol makes up 3,03 % by mass of RSP in ETS, expressed in micrograms per cubic metre ($\mu g/m^3$), is given by the equation

$$\rho_{SP} = \frac{\rho_{Sa}}{0{,}030\ 3} \tag{5}$$

where

ρ_{Sa} is the solanesol content in sampled air, calculated by Equation (4);

0,030 3 is the empirically determined mass ratio of solanesol to RSP in ETS.

NOTE This ratio is the sales-weighted average mass ratio of solanesol to RSP in ETS, having been determined experimentally in an environmental test chamber, where the only RSP present was that generated from the normal smoking of selected cigarettes. Individual ratios include: 0,030 3 determined for the leading 50 cigarette brand styles in the United States (see [6]), 0,023 0 for the leading six cigarette brand styles in each of 10 countries in Europe and Asia (see [24]), and 0,025 8 for six leading cigarette brand styles in each of eight countries in other regions of the world (see [25]). It should also be noted that, if the ETS-PM being measured is from a specific tobacco product with a known mass ratio of solanesol to RSP, then this ratio should be substituted. The applicability of this ratio has not been determined for tobacco smoke not meeting the definition of ETS given in 3.1 (e.g. machine-generated sidestream smoke).

10.2.2 Apportionment of RSP as estimated by Sol-PM

If desired, apportion the total RSP into the fraction attributable to ETS as estimated by the tobacco-selective marker Sol-PM, by calculating Sol-PM as a percentage of RSP in the sampled air. Of the total RSP content in the sampled air, the portion attributable to the ETS particulate phase, ω_{ES}, expressed as mass fraction in percent (%), is given by the equation

$$\omega_{ES} = \frac{\rho_{SP}}{\rho_{Ra}} \times 100 \tag{6}$$

where

ρ_{SP} is the RSP content attributable to ETS-PM, calculated by Equation (5);

ρ_{Ra} is the total RSP content in the sampled air (see ISO 15593[3]), expressed in micrograms per cubic metre ($\mu g/m^3$).

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[26] in 1998 involving 11 laboratories for solanesol and 6 levels. Data from 2 laboratories contained outliers. The outliers were not included in the calculation of the repeatability standard deviations and the

reproducibility standard deviations. Precision data were determined to vary linearly with mean level over the range 2,2 μg to 7,4 μg per sample for solanesol. These relationships are the following:

— repeatability standard deviation, $s_r = \text{a} \times m$;

— reproducibility standard deviation, $s_R = \text{A} \times m$;

where m is the mean sample content, in micrograms per sample.

The values of the constants a and A are listed in Table 1.

Table 1 — Values of a and A

Analyte	a	A
Solanesol	0,032	0,168

13 Test report

The test report shall give the ambient solanesol concentration, 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. The mass ratio applied in Equation (5) 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:

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

— preparation, storage, shipment and handling of samples;

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

— 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 Solanesol is typically not detected in sample blanks. Detectable 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 (that is, it should never turn past horizontal) during sampling.

A.1.5 In the event that 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 conclusion of each sampling period.

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

A.3 Method sensitivity, precision and linearity

A.3.1 The sensitivity of this method is demonstrated by the detection limit of 0,042 $\mu g/m^3$ for solanesol determination with a 1 h sampling period.

A.3.2 Determining repeatability and reproducibility ensures method precision.

A.3.3 Non-linearity in the calibration curve may occur at concentrations near the upper useable range of the UV detector in use.

A.4 Method modification

A.4.1 The sampling period described in this method may be extended beyond 24 h provided that the capacity of the filter is not exceeded.

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 UVPM and FPM (see ISO 15593[3]), which are also used as tracers of the particulate phase of ETS.

A.5 Safety

A.5.1 If spilling of solvent or any of the reagents 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.

Bibliography

[1] National Research Council, *Environmental Tobacco Smoke — Measuring Exposures and Assessing Health Effects*. National Academy Press, Washington, DC, 1986, p. 70

[2] NELSON, P.R., HEAVNER, D.L., COLLIE, B.B., MAIOLO, K.C. and OGDEN, M.W. Effect of Ventilation and Sampling Time on Environmental Tobacco Smoke Component Ratios. *Environmental Science and Technology*, **26** (10), 1992, pp. 1909-1915

[3] ISO 15593:2001, *Environmental tobacco smoke — Estimation of its contribution to respirable suspended particles — Determination of particulate matter by ultraviolet absorbance and by fluorescence*

[4] OGDEN, M.W., MAIOLO, K.C., OLDAKER, G.B. III and CONRAD, F.W. Jr. Evaluation of Methods for Estimating the Contribution of ETS to Respirable Suspended Particles. *INDOOR AIR '90, Proceedings of the 5th International Conference on Indoor Air Quality and Climate,* International Conference on Indoor Air Quality and Climate, Ottawa, Vol. 2, 1990, pp. 415-420

[5] TANG, H., RICHARDS, G., BENNER, C.L., TUOMINEN, J.P., LEE, M.L., LEWIS, E.A., HANSEN, L.D. and EATOUGH, D.J. Solanesol — A Tracer for Environmental Tobacco Smoke Particles. *Environmental Science and Technology* **24**, (6), 1990, pp. 848-852

[6] HEAVNER, D.L., MORGAN, W T. and OGDEN, M.W. Determination of Volatile Organic Compounds and Respirable Suspended Particulate Matter in New Jersey and Pennsylvania Homes and Workplaces. *Environment International*, **22** (2), 1996, pp. 159-183

[7] OGDEN, M.W. and MAIOLO, K.C. Comparison of GC and LC for Determining Solanesol in Environmental Tobacco Smoke. *LC GC Magazine*, **10** (6), 1992, pp. 459-462

[8] GUERIN, M.R., JENKINS, R.A. and TOMKINS, B.A. *The Chemistry of Environmental Tobacco Smoke: Composition and Measurement*, Lewis Publishers, Chelsea, MI, 1992

[9] EATOUGH, D.J. Assessing Exposure to Environmental Tobacco Smoke. *Modeling of Indoor Air Quality and Exposure, ASTM STP 1205*, N. L. Nagda (ed.), ASTM, 1993, pp. 42-63

[10] PHILLIPS, K., HOWARD, D.A., BROWNE, D. and LEWSLEY, J.M. Assessment of Personal Exposures to Environmental Tobacco Smoke in British Nonsmokers. *Environment International*, **20** (6), 1994, pp. 693-712

[11] PHILLIPS, K., BENTLEY, M.C., HOWARD, D.A. and ALVÁN, G. Assessment of Air Quality in Stockholm by Personal Monitoring of Nonsmokers for Respirable Suspended Particles and Environmental Tobacco Smoke. *Scandinavian Journal of Work, Environment and Health*, **22**, Supplement 1, 1996, pp. 1-24

[12] STERLING, E.M., COLLETT, C.W. and ROSS, J.A. Assessment of Non-Smokers' Exposure to Environmental Tobacco Smoke Using Personal-Exposure and Fixed-Location Monitoring. *Indoor Built Environment*, **5**, 1996, pp. 112-125

[13] OGDEN, M.W., HEAVNER, D.L., FOSTER, T.L., MAIOLO, K.C., CASH, S.L., RICHARDSON, J.D., MARTIN, P., SIMMONS, P.S., CONRAD, F.W. and NELSON, P.R. Personal Monitoring System for Measuring Environmental Tobacco Smoke Exposure. *Environmental Technology*, **17**, 1996, pp. 239-250

[14] PHILLIPS, K., BENTLEY, M.C., ABRAR, M., HOWARD, D.A. and COOK, J. Low Level Saliva Cotinine Determination and Its Application as a Biomarker for Environmental Tobacco Smoke Exposure. *Human and Experimental Toxicology*, **18** (4), 1999, pp. 291-296

[15] JENKINS, R.A., GUERIN, M.R. and TOMKINS, B.A. *The Chemistry of Environmental Tobacco Smoke: Composition and Measurement. Indoor Air Research Series*, 2nd Edition, Lewis Publishers, Boca Raton, FL, 2000

[16] OWEN, M.K., ENSOR, D.S. and SPARKS, L.E. Airborne Particle Sizes and Sources Found in Indoor Air. *Atmospheric Environment*, **26A** (12), 1992, pp. 2149-2162

[17] SPENGLER, J.D., TREITMAN, R.D., TOSTESON, T.D., MAGE, D.T. and SOCZEK, M.L. Personal Exposures to Respirable Suspended Particulates and Implications for Air Pollution Epidemiology. *Environmental Science and Technology*, **19**, 1985, pp. 700-707

[18] CONNER, J.M., OLDAKER, G.B. III and MURPHY, J.J. Method for Assessing the Contribution of Environmental Tobacco Smoke to Respirable Suspended Particles in Indoor Environments. *Environmental Technology*, **11**, 1990, pp. 189-196

[19] PROCTOR, C.J. A Multi-Analyte Approach to the Measurement of Environmental Tobacco Smoke. *Indoor Air Quality and Ventilation*, Selper, London, 1990, pp. 427-436

[20] OGDEN, M.W. and MAIOLO, K.C. Collection and Determination of Solanesol as a Tracer of Environmental Tobacco Smoke in Indoor Air. *Environmental Science and Technology*, **23** (9), 1989, pp. 1148-1154

[21] JENKINS, R.A., PALAUSKY, A., COUNTS, R.W., BAYNE, C.K., DINDAL, A B. and GUERIN, M.R. Exposure to Environmental Tobacco Smoke in Sixteen Cities in the United States as Determined by Personal Breathing Zone Air Sampling. *Journal of Exposure Analysis and Environmental Epidemiology*, **6** (4), 1996, pp. 473-502

[22] ISO 7708:1995, *Air quality — Particle size fraction definitions for health-related sampling*

[23] OGDEN, M.W. and RICHARDSON, J.D. Effect of Lighting and Storage Conditions on the Stability of Ultraviolet Particulate Matter, Fluorescent Particulate Matter, and Solanesol. *Tobacco Science*, **42**, 1998, pp. 10-15

[24] NELSON, P.R., CONRAD, F.W., KELLY, S.P., MAIOLO, K.C., RICHARDSON, J.D. and OGDEN, M.W. Composition of Environmental Tobacco Smoke (ETS) from International Cigarettes and Determination of ETS-RSP: Particulate Marker Ratios. *Environment International*, **23** (1), 1997, pp. 47-52

[25] NELSON, P.R., CONRAD, F.W., KELLY, S.P., MAIOLO, K.C., RICHARDSON, J.D. and OGDEN, M.W. Composition of Environmental Tobacco Smoke (ETS) from International Cigarettes, Part II: Nine Country Follow-up. *Environment International*, **24** (3), 1998, pp. 251-257

[26] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

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