INTERNATIONAL STANDARD

ISO 16000-6

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Indoor air —

Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA® sorbent, thermal desorption and gas chromatography using MS/FID

Air intérieur —

Partie 6: Dosage des composés organiques volatils dans l'air intérieur des locaux et enceintes d'essai par échantillonnage actif sur le sorbant Tenax TA®, désorption thermique et chromatographie en phase gazeuse utilisant MS/FID

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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 16000-6 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 6, *Indoor air*.

ISO 16000 consists of the following parts, under the general title *Indoor air*:

— *Part 1: General aspects of sampling strategy*

— *Part 2: Sampling strategy for formaldehyde*

— *Part 3: Determination of formaldeyhde and other carbonyl compounds — Active sampling method*

— *Part 4: Determination of formaldehyde – Diffusive sampling method*

— *Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA® sorbent, thermal desorption and gas chromatography using MS/FID*

The following parts are under preparation:

— *Part 5: Measurement strategy for volatile organic compounds (VOCs)*

— *Part 7: Sampling strategy for determination of airborne asbestos fibre concentrations*

— *Part 8: Ventilation rate measurement*

— *Part 9: Determination of the emission of volatile organic compounds — Emission test chamber method*

— *Part 10: Determination of the emission of volatile organic compounds — Emission test cell method*

— *Part 11: Determination of the emission of volatile organic compounds — Sampling, storage of samples and preparation of test specimens*

Introduction

ISO 16000-1 describes general requirements relating to the measurement of indoor air pollutants and the important conditions to be observed before or during the sampling of individual pollutants or groups of pollutants. Aspects of the determination (sampling and analysis) and the sampling strategy of specific pollutants or groups of pollutants are described in the subsequent parts of ISO 16000 (see Foreword).

ISO 16000-5, dealing with the sampling strategy for VOCs in indoor air, is in preparation. It is a link between ISO 16000-1 and the analytical procedures described in this part of ISO 16000.

Furthermore, the two International Standards ISO 16017-1 on pumped sampling and ISO 16017-2 on diffusive sampling focus on VOC measurements.

Indoor air —

Part 6:
Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA® sorbent, thermal desorption and gas chromatography using MS/FID

1 Scope

This part of ISO 16000 specifies a method for determination of volatile organic compounds (VOCs) in indoor air and in air sampled for the determination of the emission of VOCs from building materials using test chambers and cells. The method is based on use of Tenax TA®[1)] sorbent with subsequent thermal desorption and gas chromatographic analysis[1].

The method is applicable to the measurement of non-polar and slightly polar VOCs in a concentration range of sub-microgram per cubic metre to up to several milligrams per cubic metre. Using the principles described in this method, some very volatile compounds (VVOC) and semivolatile organic compounds (SVOC) can also be analysed.

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 16000-1:—[2)] , *Indoor air — Part 1: General aspects of sampling strategy*

ISO 16017-1, *Indoor, ambient and workplace air — Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography — Part 1: Pumped sampling*

ENV 13419-1:1999, *Building products — Determination of the emission of volatile organic compounds — Part 1: Emission test chamber method*

ENV 13419-2:1999, *Building products — Determination of the emission of volatile organic compounds — Part 2: Emission test cell method*

1) Tenax TA® is the trade name of a product manufactured by Supelco, Inc. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

2) To be published.

3 Terms and definitions

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

3.1
semi-volatile organic compound
SVOC
organic compound whose boiling point is in the range from (240 °C to 260 °C) to (380 °C to 400 °C)

NOTE 1 This classification has been defined by the World Health Organization.

NOTE 2 Boiling points of some compounds are difficult or impossible to determine because they decompose before they boil at atmospheric pressure. Vapour pressure is another criterion for classification of compound volatility that may be used for classification of organic chemicals[3]. SVOCs have vapour pressures between 10^{-2} kPa and 10^{-8} kPa.

3.2
volatile organic compound
VOC
organic compound whose boiling point is in the range from (50 °C to 100 °C) to (240 °C to 260 °C)

NOTE 1 This classification has been defined by the World Health Organization.

NOTE 2 Boiling points of some compounds are difficult or impossible to determine because they decompose before they boil at atmospheric pressure. Vapour pressure is another criterion for classification of compound volatility that may be used for classification of organic chemicals[3]. VOCs generally have saturation vapour pressures at 25 °C greater than 10^2 kPa.

3.3
very volatile organic compound
VVOC
organic compound whose boiling point is in the range from < 0 °C to (50 °C to 100 °C)

NOTE 1 This classification has been defined by the World Health Organization.

NOTE 2 Boiling points of some compounds are difficult or impossible to determine because they decompose before they boil at atmospheric pressure. Vapour pressure is another criterion for classification of compound volatility that may be used for classification of organic chemicals[3]. VVOCs typically have vapour pressures greater than 15 kPa[3].

3.4
total volatile organic compounds
TVOC
the sum of volatile organic compounds, sampled on Tenax TA®, which elute between and including n-hexane and n-hexadecane, are detected with a flame ionization detector (TVOC-FID) or mass spectrometric detector (TVOC-MS), and are quantified by converting the total area of the chromatogram in that analytical window to toluene equivalents

NOTE While this part of ISO 16000 describes the determination of individual VOCs, it is common in practice to generate a single concentration value to characterize the total amount of VOCs present in the air. This value is called the TVOC value (see 12.2 and Clause 14). It should be emphasized that the TVOC value so obtained depends on the sampling and analytical methods used, and therefore should be interpreted taking into account the full description of these methods.

4 Principle

A measured volume of sample air is collected from room air or from an emission test chamber (see ENV 13419-1) or an emission test cell (see ENV 13419-2) by drawing through one (or more) sorbent tube containing Tenax TA® sorbent. Volatile organic compounds are retained by the sorbent tube, and the compounds are subsequently analysed in the laboratory. The collected VOCs are desorbed by heat and transferred under inert carrier gas via cold trap/sorbent trap into a gas chromatograph equipped with a

capillary column or columns and a flame ionization detector and/or a mass spectrometric detector. The principle is described in ISO 16017-1.

5 Reagents and materials

5.1 Volatile organic compounds for calibration, of chromatographic quality.

5.2 Dilution solvent, for preparing calibration blend solution for liquid spiking.

The solvent should be of chromatographic quality. It shall be free from compounds co-eluting with the compound(s) of interest (5.1).

NOTE It is in most cases beneficial to use dilution solvent that is considerably more volatile than the VOCs to be analysed. Methanol most commonly fulfills this criteria. Health and safety data for organic compounds is given in e.g. International Chemical Safety Cards (ICSCS).

5.3 Tenax TA®, particle size 0,18 mm to 0,25 mm (60 mesh to 80 mesh).

Tenax TA® is a porous polymer based on 2,6-diphenylene oxide. Manufactured Tenax TA® contains quantities of impurities, which shall be removed before using it for VOC sampling. Perform cleaning by thermal conditioning the Tenax TA® under a flow of pure carrier gas. Select cleaning conditions so that no degradation of the polymer occurs, e.g. at temperature of 330 °C for at least 18 h using a carrier gas flowrate of 100 ml/min for packed sampling tubes. Pack precleaned Tenax TA® into sampling tubes that are tightly sealed and store in a closed, emission-free container. Check the success of the cleaning procedure by performing an analysis of the cleaned sorbent.

5.4 Standard atmospheres.

Prepare standard atmospheres of known concentrations of the compound(s) of interest by a recognized procedure. Methods described in ISO 6141 and the appropriate part of ISO 6145 are suitable. Prepare standard atmospheres equivalent to 100 µg/m^3. If the procedure is not applied under conditions that allow the establishment of full traceability of the generated concentrations to primary standards of mass and/or volume, or if the chemical inertness of the generation system cannot be guaranteed, the concentrations shall be confirmed using an independent procedure.

5.5 Standard sorbent tubes, loaded by spiking from standard atmospheres (5.4).

Prepare loaded sorbent tubes by passing an accurately known volume of the standard atmosphere through the sorbent tube, e.g. by means of a pump. The volume of atmosphere sampled shall not exceed the breakthrough volume of the analyte-sorbent combination. After loading, disconnect and seal the tube. Prepare fresh standards with each batch of samples. For indoor air and test chamber air, load sorbent tubes with e.g. 100 ml, 200 ml, 400 ml, 1 l, 2 l, 4 l or 10 l of the 100 µg/m^3 standard atmosphere selected.

5.6 Calibration blend solutions for liquid spiking.

The stability and safe storage times of calibration blend solutions prepared in 5.6.1 to 5.6.5 shall be determined. Fresh standard solutions shall be prepared accordingly or if evidence is noted of deterioration, e.g. reactions between alcohols and ketones.

5.6.1 Solution containing approximately 10 mg/ml of each liquid component.

Introduce 50 ml of dilution solvent into a 100 ml volumetric flask. Accurately weigh approximately 1 g of substance or substances of interest into a 100 ml volumetric flask, starting with the least volatile substance. Make up to 100 ml with dilution solvent, stopper and shake to mix.

5.6.2 Solution containing approximately 1 000 µg/ml of each liquid component.

Introduce 50 ml of dilution solvent into a 100 ml volumetric flask. Add 10 ml of solution 5.6.1. Make up to 100 ml with dilution solvent, stopper and shake to mix.

5.6.3 Solution containing approximately 100 µg/ml of each liquid component.

Introduce 50 ml of dilution solvent into a 100 ml volumetric flask. Add 10 ml of solution 5.6.2. Make up to 100 ml with dilution solvent, stopper and shake to mix.

5.6.4 Solution containing approximately 10 µg/ml of each liquid component.

Introduce 50 ml of dilution solvent into a 100 ml volumetric flask. Add 10 ml of solution 5.6.3. Make up to 100 ml with dilution solvent, stopper and shake to mix.

5.6.5 Solution containing approximately 1 µg/ml of each liquid component.

Introduce 50 ml of dilution solvent into a 100 ml volumetric flask. Add 10 ml of solution 5.6.4. Make up to 100 ml with dilution solvent, stopper and shake to mix.

5.7 Standard sorbent tubes, loaded by spiking.

Standard sorbent tubes are prepared by injecting aliquots of standard solutions onto clean sorbent tubes as follows. A sorbent tube is fitted to the injection unit of the gas chromatograph (GC) (6.10) through which inert purge gas is passed at 100 ml/min, and a 1 µl to 5 µl aliquot of an appropriate standard solution is injected through the septum. After 5 min, the tube is then disconnected and sealed. Prepare fresh standard tubes with each batch of samples.

Introducing liquid standards onto sorbent tubes via a GC injector is considered the optimum approach to liquid standard introduction, as components reach the sorbent bed in the vapor phase. Alternatively, liquid standards may be introduced directly onto the sorbent bed using a syringe (6.3).

NOTE Calibration mixtures should be prepared in controlled environmental temperature conditions. Before use the solutions are tempered accordingly.

5.8 Commercial, preloaded standard tubes.

Certified, preloaded commercial standard tubes are available. They can be used for establishing analytical quality control and for routine calibration.

5.9 Inert carrier gas, e.g. He, Ar, N_2.

The purity of the carrier gas should permit the detection of an injection of 0,5 ng of toluene.

NOTE The quality of the carrier gas is of great importance, as contaminants possibly contained in the gases are enriched in the cold trap together with the substances to be analysed.

6 Apparatus

Ordinary laboratory apparatus and the following.

6.1 Sorbent tubes, of stainless steel or glass, containing at least 200 mg of Tenax TA® sorbent (5.3), with metal screw caps and polytetrafluoroethene (PTFE) ferrules.

Tubes with outside diameter of 6 mm, inside diameter of 5 mm, and of length 90 mm fulfil the requirement and are used in many commercial thermal desorbers. Use deactivated glass wool or other suitable mechanism, e.g. stainless steel frit, to retain the sorbent in the tube.

Precleaned sorbent tubes containing Tenax TA® are available commercially; or the sorbent tubes can be filled in the laboratory as follows.

Weigh the appropriate amount of adsorbent, using no less than 200 mg of sorbent per tube to maintain the sorption capacity. To pack the tube, insert a plug of deactivated glass wool or a stainless steel gauze into one end of the tube. Transfer the adsorbent into the tube, assisted by suction if desired. Place an additional plug or gauze after the sorbent to retain it in the tube.

NOTE A description of the determination of breakthrough volume is given in ISO 16017-1:2000, Annex B. Breakthrough volumes are proportional to the dimensions of the sampling tube and quantity of sorbent. As an approximate measure, doubling the bed-length while tube diameter is kept constant doubles the SSV (safe sampling volume).

6.2 Sorbent tube unions.

For sampling, two sorbent tubes may be connected in series using metal screw-cap couplings with PTFE ferrules.

6.3 Precision syringes, readable to at least 0,1 µl.

6.4 Sampling pump.

The pump should fulfil the requirements of EN 1232 or ASTM D 3686.

6.5 Tubing.

PE (polyethene) or PTFE tubing of appropriate diameter is used to ensure a leak-proof fit to both pump and sample tube. Sampling tubes shall not be used with plastic tubing upstream of the sorbent. Interferences from the tubing can introduce contaminants.

6.6 Flow meter calibrator.

Bubble meter or other suitable device for gas flow calibration.

6.7 Gas chromatographic (GC) system, fitted with a flame ionization detector and/or mass spectrometric detector capable of detecting an injection of at least 1 ng of toluene with a signal-to-noise ratio of at least 5 to 1.

6.8 Capillary column.

A suitable GC capillary column is selected for separation of analytes in the sample. Bonded 100 % dimethylpolysiloxane columns of 30 m to 60 m, internal diameter 0,25 mm to 0,32 mm and phase thickness 0,25 µm to 0,33 µm are examples of columns proven to be suitable for indoor air and emission test chamber air VOC analysis.

NOTE A dimethylpolysiloxane column, e.g. an HP-1[3)] column, does not separate *d*-3-carene from 2-ethyl-1-hexanol with certain oven programmes, nor does it separate *m*- and *p*-xylenes.

6.9 Thermal desorption apparatus, for the two-stage thermal desorption of the sorbent tubes and transfer of desorbed vapours via an inert gas flow into a GC.

A typical apparatus contains a mechanism for holding the tubes to be desorbed whilst they are heated and purged simultaneously with inert carrier gas. The desorption temperature and time are adjustable, as is the carrier gas flowrate. The apparatus may also incorporate additional features, such as automatic sample-tube loading, leak testing, and a cold trap or other suitable device to concentrate the desorbed sample. The

3) HP-1 is the trade name of a product manufactured by Agilent, Inc. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

desorbed sample, contained in the purge gas, is routed to the gas chromatograph and capillary column via a heated transfer line.

6.10 Injection facility for preparing standards by liquid spiking (optional).

A conventional gas chromatographic injection unit may be used for preparing calibration standards. This can be used *in situ*, or it can be mounted separately. The carrier gas line to the injector should be retained. The back of the injection unit should be adapted if necessary to fit the sample tube. This can be done conveniently by means of compression coupling with an O-ring seal.

7 Conditioning and storage of sorbent tubes

7.1 Conditioning

Prior to each sampling use, condition the precleaned sorbent tubes at 300 °C for 10 min under inert carrier gas at a flowrate of 50 ml/min to 100 ml/min, to remove trace organic volatiles possibly trapped on the tube. Analyse a representative number of conditioned tubes for blank value, using routine analytical parameters, to ensure that the thermal desorption blank is sufficiently small. The sorbent tube blank level is acceptable if artifact peaks are no greater than 10 % of the typical areas of the analytes of interest. If the blank is unacceptable, recondition the tubes by repeating the conditioning procedure. If after repeated conditioning the blank is still unacceptable, the tubes shall be refilled (see procedure in 6.1).

7.2 Storage of conditioned sorbent tubes before sampling

Seal conditioned sorbent tubes with metal screw-cap fittings with PTFE ferrules and store in an emission-free container at room temperature. Use conditioned sampling tubes within four weeks. Recondition tubes stored for more than four weeks before sampling.

8 Calibration of pump

Calibrate the pump with the sorbent tube assembly in line, using an appropriate external calibrated meter.

9 Sampling

9.1 Indoor air sampling

Assemble the sampling line. If more than one tube is used in order to ensure that the breakthrough volume for one tube and the analyte of interest is not exceeded, prepare a tube assembly by joining the tubes in series with a union (6.2). Attach the pump to the sorbent tube or tube assembly with PE or PTFE tubing. Start the pump and note and record the sampling flowrate or register reading, note starting time, temperature and, if necessary for calculation, also barometric pressure. An appropriate sampling flowrate is in the range of 50 ml/min to 200 ml/min. At the end of the sampling period, note and record the flowrate or register reading, turn the pump off, and note and record the time, temperature and, if necessary, barometric pressure. Disconnect the sampling tube from the sampling line and seal both ends using screw-cap fittings with PTFE ferrules.

If sampling flowrate is determined using an integrated flow-measuring device, e.g. a mass flow meter, connect the sampling tube to the sampling line, start the pump, note and record the time and flowrate or register reading. Note and record temperature and, if necessary, barometric pressure. An appropriate sampling flowrate is in the range of 50 ml/min to 200 ml/min. At the end of the sampling period, note and record the flowrate or register reading, turn off the pump, note and register the time the pump was turned off. Disconnect the sampling tube from the sampling line and seal both ends using screw-cap fittings with PTFE ferrules.

Sampling from indoor air shall be performed taking into account the general aspects of sampling strategy as described in ISO 16000-1.

Sampling flowrates lower than 50 ml/min may be used if the operator finds it necessary, e.g. to enable longer sampling times.

9.2 Test chamber air sampling

Assemble the sampling line. If the sampling flowrate is determined with a calibrator, start the pump, note and record the sampling flowrate. Appropriate sampling flowrate is in the range of 50 ml/min to 200 ml/min. When sampling from an emission chamber, the sampling flow shall not exceed 80 % of the air flowrate of the chamber. Connect the sampling tube to the test chamber outlet or other sampling port of the emission test chamber, note and record the time the tube was connected. Note and record temperature and if necessary barometric pressure. At the end of the sampling period disconnect the sampling tube from the chamber sampling port, note and record the time of disconnection, repeat the sampling flow determination, and turn off the pump. Disconnect the sampling tube from the sampling line and seal both ends using screw cap fittings with PTFE ferrules.

If sampling flowrate is determined by using an integrated flow-measuring device, e.g. a mass flow meter, connect the sampling tube to the sampling line and further to the chamber sampling port, start the pump, note and record the time and flowrate or register reading. Note and record temperature and, if necessary, barometric pressure. An appropriate sampling flowrate is in the range of 50 ml/min to 200 ml/min. At the end of the sampling period, note and record the flowrate or register reading, turn off the pump, note and register the time the pump was turned off. Disconnect the sampling tube from the sampling line and seal both ends using screw cap fittings with PTFE ferrules.

9.3 Sampling volumes

Safe sampling volumes (ssv), i.e. the amount of gas that can be sampled without breakthrough of VOCs, are listed in Annex B. In general, the suitable sampling volumes when sampling VOCs from non-industrial indoor air is 1 l to 5 l for sampling tubes with 200 mg of Tenax TA®. In material emission measurements, the material type and age, loading factor and air exchange rate in the chamber determine suitable sampling volumes. The recommended sampling volume, in general, is $\leqslant$ 5 l.

Sampling volume has to be adjusted to the expected concentrations. When sampling unknown concentrations, it is recommended to take at least three parallel samples with different sampling volumes. If the analytical result is not dependent on the sampling volume, no breakthrough of the analytes has occurred.

9.4 Storage of loaded samples

Loaded sampling tubes shall be tightly sealed and stored in an emission-free container at ambient room temperature. The effect of storage on loaded VOC from indoor or chamber air is not known, although certain experiences (see Annex C) suggest that they may be stable over several months at room temperature. To avoid possible changes, the samples should be analysed as soon as possible and preferably within four weeks after collection.

9.5 Field blanks

Field blanks shall be Tenax TA® sampling tubes identical to those used for VOC sampling. These tubes are subjected to the same handling procedure in the field as the sample tubes, except for the actual period of sampling. Field blanks shall be marked, stored and analysed in sequence with the actual samples. In a measurement campaign, about 10 % of the samples analysed shall be field blanks. If only a few measurements are performed, at least one field blank shall be prepared and analysed.

10 Analysis

10.1 General

For analysis, VOCs are thermally desorbed from the sampling tubes. Separate the individual VOCs using capillary columns in a gas chromatograph and detect with a flame ionization detector (FID) and mass spectrometric detector (MS) or with MS only. MS can be used for both identification and quantification of compounds, while FID signals alone are used only for compound quantification.

When a flame ionization detector and a mass spectrometric detector are used together for the analysis, the detectors can either be fitted to the same gas chromatograph or to different gas chromatographs. In the latter case, identical sample injection and separation parameters shall be used in both instruments to produce comparable chromatograms.

When the quantification is made using FID, calibration standard mixtures of different concentrations or at least a single level calibrant shall be analysed with each set of samples as a check on system performance.

When using MS for quantification, calibration standard mixtures of at least three — or better, five or seven — different concentrations shall be analysed with each set of samples to update the calibration.

Internal standards, e.g. isotopically labelled compounds, may be used to control the performance of sampling and analysis.

10.2 Thermal desorption

Select desorption time and desorption gas flowrate so that the desorption efficiency for octadecane is better than 95 %. Determination of desorption efficiency is described in ISO 16017-1.

Typical desorption conditions for VOC analysis using a secondary cold trap and sampling tube containing 200 mg to 250 mg Tenax TA® are

Desorption temperature	260 °C to 280 °C
Desorption time	5 min to 15 min
Desorption gas flowrate	30 ml/min to 50 ml/min
Cold-trap high temperature	280 °C
Cold-trap low temperature	– 30 °C
Cold-trap sorbent	Tenax TA®
Transfer-line temperature	220 °C
Split ratios	Split ratios between the sample tube and secondary trap and between the secondary trap and analytical column (if applicable) should be selected dependent on expected atmospheric concentration. (See guidance from respective manufacturers of the thermal desorption apparatus.)

NOTE The more volatile VVOCs can break through the cold trap under these conditions and will not be quantitatively determined by the analysis.

10.3 Temperature programme

Temperature programming of the analytical column is needed when analysing mixtures of substances showing large differences in boiling points and polarities in order to achieve a good resolution in minimal time.

10.4 Analysis of the samples

Analyse VOC samples preferably within four weeks from sampling. Analyse field blanks and appropriate standards in sequence with the samples. Identify VOCs with MS and quantify them from the FID or MS chromatogram.

11 Identification of single VOCs

For identification of single, non-target VOCs, analyse the samples with MS operating in the scan mode. Identify single VOCs detected in the sample using the mass spectrometer total ion chromatogram and the retention time of the compound. Compare the total ion chromatogram with either the mass spectra of pure compounds or commercially available compilations (libraries) of mass spectra. User-generated libraries may also be used. Correspondence of retention time with a retention time of a compound used for calibration on a single column should not exclusively be regarded as proof of identity.

Identify as many compounds as possible, particularly those representing the 10 highest peaks and those present at concentrations above 2 µg/m^3. A list of VOCs which, according to current experience, are frequently encountered in indoor air and emitted from materials is given in Annex A. A satisfactory level of identification has been achieved if the area of identified VOCs when summed correspond to two-thirds of the total area of all the peaks in the chromatogram eluting between and including C_6 to C_{16}.

The SIM (Selected Ion Monitoring) mode of MS operation may also be used. The choice is left to the operator, who must be aware of the differences between SIM and scan modes.

12 Concentration of analytes in the sampled air

12.1 General

Identified compounds are quantified using their individual response factors when the reference compound is available. In other cases, quantification is reported as a toluene equivalent. Unidentified compounds are quantified using the toluene response factor.

12.2 Volatile organic compounds (VOCs)

Compound-specific response factors and the linearity of FID and MS for compounds of interest are determined by calibrating the analytical system with standard solutions (5.5, 5.6.2, 5.6.3, 5.6.4, 5.6.5 or 5.9). Prepare a calibration curve using at least three different concentrations over the linear range (better using five or seven different concentrations). The lowest concentration used for calibration shall be at or below the lowest sample concentration.

The peak areas of a single VOC chromatogram are proportional to the mass of compound injected. For each compound, the relationship between the mass of analyte injected and the corresponding peak area is determined. The slope of the calibration curve over the linear range is the response factor of the VOC studied:

$$A_{St} = b_{St} m_{St} + c_{St} \quad (1)$$

where

A_{St} is the analyte peak area in the chromatogram of the standard, in area units;

b_{St} is the slope of the calibration curve;

m_{St} is the mass of analyte in the standard, in nanograms;

c_{St} is the intersect of ordinates and calibration curve. If the calibration curve crosses the origin, b_{St} is considered zero.

The mass of analyte present in the sample is calculated from the detector peak area using the response factor of the analyte:

$$m_A = \frac{A_A}{b_{St}} - c_A \quad (2)$$

where

m_A is the mass of analyte in the sample, in nanograms;

A_A is the peak area of analyte in the chromatogram of the sample, in area units;

b_{St} is the slope of the calibration curve;

c_A is the intersect of ordinates and the calibration curve. If the calibration curve crosses the origin, c_A is considered zero.

The mass concentration of identified VOCs in the sampled air is calculated by means of the following equation:

$$\rho_A = \frac{m_A - m_{A0}}{V} \quad (3)$$

where

ρ_A is the mass concentration of analyte in the air sampled, in micrograms per cubic metre;

m_A is the mass of analyte present in the sampling tube, in nanograms;

m_{A0} is the mass of analyte present in the blank tube, in nanograms;

V is the sampling volume, in litres.

If necessary, the concentrations are adjusted to 20 °C and 101,3 kPa:

$$\rho_{A;101,3;293} = \rho_A \cdot \frac{101{,}3\ \text{kPa}}{p} \cdot \frac{(t + 273\ \text{K})}{293\ \text{K}} \quad (4)$$

where

p is the actual pressure of the air sampled, in kilopascals;

t is the actual temperature of the air sampled, in degrees Celsius.

Unidentified compounds in the sample are quantified using the calibration response factor for toluene

12.3 Total volatile organic compounds (TVOCs)

The TVOC concentration is determined as follows.

Consider the entire area of the chromatogram between n-hexane and n-hexadecane. Using the toluene response factor, convert the area into mass units of toluene. Using Equation (3), calculate the TVOC mass concentration in the sampled air.

The parameters of a “Standard Spectra Tune”, or equivalent MS parameters, shall be set when using MS for this purpose. Otherwise, the use of an FID is preferred.

NOTE 1 These recommendations are given to improve the comparability of TVOC results.

NOTE 2 TVOC determined in toluene equivalents is semiquantitative, since individual compounds in the mixture may have response factors varying widely from the toluene response factor.

12.4 VVOC and SVOC compounds observed outside the TVOC range

To obtain information on additional organic compounds present in indoor air or emitted from products into test chamber air, it is appropriate not only to determine VOC but also to have some information on VVOC and SVOC, i.e. organic compounds eluted before C_6 and after C_{16}. For this, the total area of compounds detected outside the C_6 to C_{16} range is converted into toluene equivalents in accordance with Equations (2) and (3), using the toluene response factor. Individual compounds eluting before and after hexane and hexadecane should be identified to the extent possible, and the summed concentrations reported for VVOC and SVOC. The quality of the results so obtained will be lower than that of the VOC analysis, because the sampling and analytical procedures used may not be fully appropriate for VVOC and SVOC determination.

NOTE For quantitative determination of VOCs outside the TVOC range, alternative sorbents and analytical conditions can be used (see ISO 16017-1).

13 Performance characteristics

Before this method is used, its performance characteristics should be determined in accordance with the *Guide to the Expression of Uncertainty in Measurement* (GUM) [14]. This determination should include, as a minimum, the estimation of uncertainty components from the following sources:

Sampling:
- Flow
- Time
- Temperature
- Pressure
- Sampling efficiency

Sampling integrity:
- Measure and stability
- Blank stability

Desorption efficiency

Calibration:
- Standards
- Lack-of-fit of calibration function

Analysis:
- Repeatability
- Blank level

Environmental influences:
- Temperature at sampling
- Humidity at sampling
- Interferents

Field repeatability

Chamber techniques:
Air change
Test specimen preparation

The accuracy and repeatability of the measuring method are important factors, which shall be determined in order to evaluate the results and the suitability of the method for the intended purposes. The accuracy of the VOC measurement method can be determined if atmospheres of known level (micrograms per cubic metre) can be reliably produced. This is relatively difficult and therefore most researchers only determine the repeatability of their measuring method by repeated sampling from a constant atmosphere.

In a study of chlorinated butadienes in indoor air, the uncertainty of the measurement results was assessed based on the principles of the GUM. The combined relative uncertainty for the measurement of hexachlorobutadiene at the volume fraction level of $0{,}6 \times 10^{-9}$ was $\pm$ 12 % and the expanded relative uncertainty (at the 95 % confidence level) was $\pm$ 23 %[4]

The repeatability of sampling of non-polar hydrocarbons from cylinder atmospheres containing six VOCs is reported[5]. For 2-litre samples, the repeatability for Tenax TA® was less than 10 %, and for 0,5-litre samples it was 12 %.

NOTE In material emission testing, interlaboratory comparisons have been organized to assess the agreement among laboratories undertaking tests to characterize the emission of VOCs from indoor materials and products. The results of these intercomparisons are published in reports[6], [7].

14 Test report

The test report shall contain at least the following information:

a) purpose of the measurements;

b) description of the sampling location;

c) time and date of the sampling;

d) sampling conditions (temperature, relative humidity);

e) reference to this International Standard;

f) full description of the sampling procedure;

g) full description of the analytical procedure;

h) detection limit of the analytical method;

i) concentrations of identified compounds, provided with CAS-numbers, including calculation and calibration principles used;

j) uncertainty of the reported results.

The results should be complemented by the following:

— $TVOC_{FID}$ or $TVOC_{MS}$ mass concentration in toluene equivalents;

— mass concentration of total $VVOC_{FID}$ or $VVOC_{MS}$ and total $SVOC_{FID}$ or $SVOC_{MS}$ detected before C_6 and after C_{16}, in toluene equivalents.

15 Quality control

An appropriate level of quality control shall be employed, including verification that

— field blanks are prepared according to 9.5,

— the sorbent tube blank level is acceptable if artifact peaks are no greater than 10 % of the typical areas of the analytes of interest,

— desorption efficiency of VOCs can be controlled by using internal standards (see ISO 16017-1). To monitor the response factors, a standard mixture of selected, representative compounds is analysed in sequence with the actual samples,

— the collection efficiency can be controlled by using back-up tubes or taking samples of different sampling volumes less than the safe sampling volume,

— repeatability of the measuring method has been determined, e.g. using collection and analysis of duplicate samples. A relative standard deviation of $\leqslant$ 15 % should be reached,

— the recovery of C_6 to C_{16} hydrocarbons is 95 %.

Annex A
(informative)

Examples of compounds detected in indoor air and from building products in test chambers

Table A.1 — Examples of compounds detected in indoor air and emitted from building products in test chambers[8], [9]

Chemical compound	CAS No.	Boiling point °C
Aromatic hydrocarbons		
1,2,3-Trimethylbenzene	526-73-8	176
1,2,4,5-Tetramethylbenzene	95-93-2	197
1,2,4-Trimethylbenzene	95-63-6	169
1,3,5-Trimethylbenzene	108-67-8	165
1,3-Diisopropyl benzene	99-62-7	203
1,4-Diisopropyl benzene	100-18-5	203
1-Methyl-2-propylbenzene	1074-17-5	
1-Methyl-3-propylbenzene	1074-43-7	175
1-Propenylbenzene	637-50-3	175
2-Ethyltoluene	611-14-3	165
3-Ethyltoluene/4-Ethyltoluene	620-14-4/622-96-8	162
2-Phenyloctane	777-22-0	123
4-Phenylcyclohexene	4994-16-5	251 [a]
5-Phenyldecane	4537-11-5	
5-Phenylundecane	4537-15-9	
α-Methylstyrene	98-83-9	165
Benzene	71-43-2	80
Ethylbenzene	100-41-4	136
Ethylylbenzene/Ethynylbenzene	536-74-3	144
Isopropylbenzene	98-82-8	152
m/*p*-Methylstyrene	100-80-1/622-97-9	168/169
m/*p*-Xylene	108-38-3/106-42-3	139/138
Naphthalene	91-20-3	218
n-Butylbenzene	104-51-8	183
n-Propylbenzene	103-65-1	159
o-Methylstyrene	611-15-4	171
o-Xylene	95-47-6	144

Table A.1 (*continued*)

Chemical compound	CAS No.	Boiling point °C
Styrene	100-42-5	145
Toluene	108-88-3	111
Aliphatic hydrocarbons n-C_6 to n-C_{16}		
1-Decene	872-05-9	171
1-Octene	111-66-0	121
2,2,4,6,6-Pentamethylheptane	13475-82-6	178
2,4,6-Trimethyloctane	62016-37-9	
2-Methylhexane	591-76-4	90
2-Methylnonane	871-83-0	167
2-Methyloctane	3221-61-2	143
2-Methylpentane	107-83-5	60 [b]
3,5-Dimethyloctane	15869-93-9	159
3-Methylhexane	589-34-4	92
3-Methyloctane	2216-33-3	143
3-Methylpentane	96-14-0	63 [b]
4-Methyldecane	2847-72-5	189
Isododecane	31807-55-3	216
n-Decane	124-18-5	174
n-Dodecane	112-40-3	216
n-Heptane	142-82-5	98
n-Hexadecane	544-76-3	287
n-Hexane	110-54-3	69
n-Nonane	111-84-2	151
n-Octane	111-65-9	125
n-Pentadecane	629-62-9	271
n-Tetradecane	629-59-4	254
n-Tridecane	629-50-5	235
n-Undecane	1120-21-4	196
Cycloalkanes		
1,4-Dimethylcyclohexane	589-90-2	124
1-Methyl-4-methylethylcyclohexane (*cis*/*trans*)	6069-98-3 /1678-82-6	167
Cyclohexane	110-82-7	81
Methylcyclohexane	108-87-2	101
Methylcyclopentane	96-37-7	72

Chemical compound	CAS No.	Boiling point °C
Terpenes		
β-Caryophyllene	87-44-5	129
α-Pinene	80-56-8	156
β-Pinene	18172-67-3	164
3-Carene	13466-78-9	167
a-Cedrene	469-61-4	262
Camphene	79-92-5	158
Limonene	138-86-3	176
Longifolene	475-20-7	254
Turpentine	8006-64-2	150 to 180
Alcohols		
1-Butanol	71-36-3	118
1-Hexanol	111-27-3	158
1-Octanol	111-87-5	194
1-Pentanol	71-41-0	137
1-Propanol	71-23-8	97
2-Ethyl-1-hexanol	104-76-7	182
2-Methyl-1-propanol (isobutanol)	78-83-1	108
2-Methyl-2-propanol	75-65-0	82
2-Propanol	67-63-0	82
BHT (2,6-di-tert-butyl-4-methylphenol)	128-37-0	265
Cyclohexanol	108-93-0	161
Phenol	108-95-2	182
2,2,4-trimethyl-1,3-pentanediol isobutyrate	25265-77-4	244
Glycols/Glycol ethers		
1-Methoxy-2-propanol	107-98-2	118
2-Butoxyethanol	111-76-2	171
2-Butoxyethoxyethanol	112-34-5	231
2-Ethoxyethanol	110-80-5	136
2-Methoxyethanol	109-86-4	125
2-Phenoxyethanol	122-99-6	245
3-Phenyl-1-propanol	6180-61-6	235
2-(2-Butoxyethoxy)ethanol	112-34-5	230
Dimethoxyethane	110-71-4	85
Dimethoxymethane	109-87-5	42 [b]
Propylene glycol	57-55-6	189

Chemical compound	CAS No.	Boiling point °C
Aldehydes		
2-Butenal	123-73-9	104
2-Decenal	2497-25-8	
2-Ethylhexanal	123-05-7	163
2-Furancarboxaldehyde	98-01-1	162
2-Heptenal (*cis*/*trans*)	57266-86-1/18829-55-5	90 to 91 at 50 mmHg
2-Nonenal	2463-53-8	100 to 102 at 16 mmHg
2-Pentenal	1576-87-0	115 to 125
2-Undecenal	1337-83-3	
Acetaldehyde	75-07-0	21 [b]
Benzaldehyde	100-52-7	179
Butanal	123-72-8	76
Decanal	112-31-2	208
Heptanal	111-71-7	153
Hexanal	66-25-1	129
Nonanal	124-19-6	190
Octanal	124-13-0	171
Pentanal	110-62-3	103
Propanal	123-38-6	49 [b]
Ketones		
2-Butanone	78-93-3	80
2-Methylcyclohexanone	583-60-8	163
2-Methylcyclopentanone	1120-72-5	139
3-Methyl-2-butanone	563-80-4	95
4-methyl-2-pentanone	108-10-1	117
3,5,5-Trimethylcyclohex-2-enone	78-59-1	214
Acetone	67-64-1	56 [b]
Acetophenone	98-86-2	202
Cyclohexanone	108-94-1	155
Cyclopentanone	120-92-3	130
Methyl ethyl ketone	78-93-3	80
Methyl isobutyl ketone	108-10-1	118
Halocarbons		
1,1,1,2-Tetrachloroethane	630-20-6	130
1,1,2,2-Tetrachloroethane	79-34-5	146
1,1,1-Trichloroethane	71-55-6	74
1,1,2-Trichloroethane	79-00-5	114
1,2-Dichloroethane	107-06-2	84

Chemical compound	CAS No.	Boiling point °C
1,4-Dichlorobenzene	106-46-7	173
Carbon tetrachloride	56-23-5	76
Chlorobenzene	108-90-7	131
Dichloromethane	75-09-2	40 [b]
Tetrachloroethene	127-18-4	121
Trichloroethene	79-01-6	87
Acids		
2,2-Dimethylpropanoic acid	75-98-9	164
Acetic acid	64-19-7	118
Butyric acid	107-92-6	163
Heptanoic acid	111-14-8	223
Hexadecanoic acid	57-10-3	350
Hexanoic acid	142-62-1	202
Isobutyric acid	79-31-2	153
Octanoic acid	124-07-2	240
Pentanoic acid	109-52-4	186
Propanoic acid	79-09-4	141
Esters		
2-Ethoxyethyl acetate	111-15-9	156
2-Ethylhexyl acetate	103-09-3	198
2-Methoxyethyl acetate	110-49-6	145
Butoxyethyl acetate	112-07-2	192
Butyl acetate	123-86-4	126
Butyl formate	592-84-7	107
Ethyl acetate	141-78-6	77
Ethyl acrylate	140-88-5	100
Isobutyl acetate	110-19-0	118
Isopropyl acetate	108-21-4	90
Linalool acetate	115-95-7	220
Methyl acrylate	96-33-3	81
Methyl methacrylate	80-62-6	100
Propyl acetate	109-60-4	102
2,2,4-Trimethylpentanediol diisobutyrate	6846-50-0	424
Vinyl acetate	108-05-4	72 [b]

Chemical compound	CAS No.	Boiling point °C
Phthalates		
Alkyl phthalates		
Dibutyl phthalate	84-74-2	340
Dimethyl phthalate	131-11-3	284
Other		
1,4-Dioxane	123-91-1	101
1-Methyl-2-pyrrolidinone	872-50-4	202
2-Pentylfuran	3777-69-3	> 120
Aniline	62-53-3	184
Caprolactam	105-60-2	267
Indene	95-13-6	182
Nitrobenzene	98-95-3	211
Pyridine	110-86-1	116
Tetrahydrofuran	109-99-9	67 [b]

NOTE 1 Safe sampling volumes for organic vapours are given in Annex B.

NOTE 2 When analysing VOCs eluting before n-hexane, the complementary sorbents given in ISO 16017-1 can be used.

[a] Value of 1-phenylcyclohexene.

[b] Compounds with boiling points below that of hexane are not retained quantitatively by Tenax TA® when using the sampling tube size and sampling volumes recommended in this part of ISO 16000.