BS ISO 16000-6:2011

BSI Standards Publication

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 or MS-FID

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National foreword

This British Standard is the UK implementation of ISO 16000-6:2011.

The UK participation in its preparation was entrusted to Technical Committee EH/2/3, Ambient atmospheres.

A list of organizations represented on this committee can be obtained on request to its secretary.

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INTERNATIONAL STANDARD

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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 or MS-FID

Air intérieur —

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

This second edition cancels and replaces the first edition (ISO 16000-6:2004), which has been technically revised.

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 formaldehyde and other carbonyl compounds in indoor air and test chamber air — Active sampling method*

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

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

— *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 or MS-FID*

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

— *Part 8: Determination of local mean ages of air in buildings for characterizing ventilation conditions*

— *Part 9: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test chamber method*

— *Part 10: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test cell method*

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

— *Part 12: Sampling strategy for polychlorinated biphenyls (PCBs), polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polycyclic aromatic hydrocarbons (PAHs)*

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— *Part 13: Determination of total (gas and particle-phase) polychlorinated dioxin-like biphenyls (PCBs) and polychlorinated dibenzo-*p*-dioxins/dibenzofurans (PCDDs/PCDFs) — Collection on sorbent-backed filters*

— *Part 14: Determination of total (gas and particle-phase) polychlorinated dioxin-like biphenyls (PCBs) and polychlorinated dibenzo-*p*-dioxins/dibenzofurans (PCDDs/PCDFs) — Extraction, clean-up and analysis by high-resolution gas chromatography and mass spectrometry*

— *Part 15: Sampling strategy for nitrogen dioxide (NO_2)*

— *Part 16: Detection and enumeration of moulds — Sampling by filtration*

— *Part 17: Detection and enumeration of moulds — Culture-based method*

— *Part 18: Detection and enumeration of moulds — Sampling by impaction*

— *Part 19: Sampling strategy for moulds*

— *Part 23: Performance test for evaluating the reduction of formaldehyde concentrations by sorptive building materials*

— *Part 24: Performance test for evaluating the reduction of volatile organic compound (except formaldehyde) concentrations by sorptive building materials*

— *Part 25: Determination of the emission of semi-volatile organic compounds by building products — Micro-chamber method*

— *Part 26: Sampling strategy for carbon dioxide (CO_2)*

— *Part 28: Determination of odour emissions from building products using test chambers*

The following parts are under preparation:

— *Part 21: Detection and enumeration of moulds — Sampling from materials*

— *Part 27: Determination of settled fibrous dust on surfaces by SEM (scanning electron microscopy) (direct method)*

— *Part 29: Test methods for VOC detectors*

— *Part 30: Sensory testing of indoor air*

— *Part 31: Measurement of flame retardants and plasticizers based on organophosphorus compounds — Phosphoric acid ester*

— *Part 32: Investigation of constructions on pollutants and other injurious factors — Inspections*

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Introduction

ISO 16000-1 establishes 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 specified in the subsequent parts of ISO 16000 (see Foreword).

ISO 16000-5 (dealing with VOC sampling strategy) is a link between ISO 16000-1 (a generic standard establishing the principles) and this part of ISO 16000, which deals with sampling and analytical measurements.

ISO 16017 (see Clause 2 and Reference [8]) and ISO 12219[3]–[7] also focus on volatile organic compound (VOC) measurements.

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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 or 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 products or materials and other products used in indoor environments using test chambers and test cells. The method uses Tenax TA®[1)] sorbent with subsequent thermal desorption (TD) and gas chromatographic (GC) analysis[13] employing a capillary column or columns and a flame ionization detector (FID) and/or a mass spectrometric (MS) detector.

The method is applicable to the measurement of non-polar and slightly polar VOCs at concentrations ranging from sub-micrograms per cubic metre to several milligrams per cubic metre. Using the principles specified in this method, some very volatile compounds (VVOC) and semi-volatile organic compounds (SVOC) can also be analysed (see Annex D).

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, *Indoor air — Part 1: General aspects of sampling strategy*

ISO 16017-1:2000, *Indoor, ambient and workplace air — Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography — Part 1: Pumped sampling*

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[14].

1) Tenax TA® is the trade name of a product supplied by Buchem. This information is given for the convenience of users of this document 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.

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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[15].

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[14].

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[15].

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[14].

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[15].

3.4
total volatile organic compounds
TVOCs
sum of volatile organic compounds, sampled on Tenax TA®, which elute between and including *n*-hexane and *n*-hexadecane on a non-polar capillary column, detected with a flame ionization detector (TVOC-FID) or mass spectrometric detector (TVOC-MS), and quantified by converting the total area of the chromatogram in that analytical window to a nominal mass using the chromatographic response factor for toluene (toluene equivalents)

NOTE While this part of ISO 16000 specifies 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 11.3 and Clause 13). 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, an emission test chamber (see ISO 16000-9) or an emission test cell (see ISO 16000-10) by drawing through one (or more) sorbent tube containing Tenax TA® sorbent. Volatile organic compounds (VOCs) 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 a cold trap or sorbent trap into a gas chromatograph equipped with a capillary column or columns and a flame ionization detector and/or a mass spectrometric detector.

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, of chromatographic quality, 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 fulfils this criterion. Health and safety data for organic compounds are given, for example, in International Chemical Safety Cards (ICSCs)[24].

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5.3 Tenax TA®, particle size 0,18 mm to 0,60 mm (30 mesh to 80 mesh).

Tenax TA® is a porous polymer based on 2,6-diphenyleneoxide. 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 a temperature of 300 °C for 10 h using a carrier gas flow rate of 50 ml/min to 100 ml/min for packed sampling tubes. Pack pre-cleaned 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.

NOTE Pre-packed, conditioned (cleaned) and capped sorbent tubes are available commercially.

5.4 Standard atmospheres, of known concentrations of the compound(s) of interest, prepared by a recognized procedure. Methods specified in ISO 6141[1] and the appropriate part of ISO 6145[2] are suitable.

Prepare standard atmospheres equivalent to about 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), prepared 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.

5.6.1 General. The stability and safe storage times of calibration blend solutions shall be determined. Fresh standard solutions shall be prepared accordingly or if there is evidence of deterioration, e.g. reactions between alcohols and ketones. Appropriate calibration solution concentrations vary depending upon expected target analyte levels in each batch of samples. Examples of solution preparation for a range of applications are given in 5.6.2 to 5.6.6.

5.6.2 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.

5.6.3 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 5.6.2. Make up to 100 ml with dilution solvent, stopper and shake to mix.

5.6.4 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 5.6.3. Make up to 100 ml with dilution solvent, stopper and shake to mix.

5.6.5 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 5.6.4. Make up to 100 ml with dilution solvent, stopper and shake to mix.

5.6.6 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 5.6.5. Make up to 100 ml with dilution solvent, stopper and shake to mix.

5.7 Standard sorbent tubes, loaded by spiking, prepared by injecting aliquots of standard solutions on to clean sorbent tubes.

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The sampling end of a sorbent tube is fitted to the unheated 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 disconnected and sealed. Prepare fresh standard tubes with each batch of samples.

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

Calibration mixtures should be prepared in controlled ambient temperature conditions. Before use, temper the solutions accordingly.

NOTE 1 When preparing standard tubes containing SVOC analytes, efficient transfer is enhanced if the configuration of injector allows the tip of the syringe to make gentle contact with the sorbent retaining mechanism (e.g. gauze or frit) within the tube.

NOTE 2 Standard tubes containing VVOCs are more typically prepared either from standard atmospheres (see 5.4 and 5.5) or from concentrated gas standards sourced commercially. It is appropriate for concentrated gas standards to be introduced to the sampling end of sorbent tubes in a stream of carrier gas via an unheated GC injector.

NOTE 3 If standard tubes are being prepared by introducing aliquots from more than one standard solution or gas, it is appropriate first to introduce the standard containing higher boiling components and to introduce the lightest components last. This minimizes risk of analyte breakthrough during the standard tube loading process.

5.8 Commercial, preloaded standard tubes, certified, are available and 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.

CAUTION — 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 in particular 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 6,4 mm (0.25 inch), inside diameter of 5 mm, and of length 89 mm (3.5 inch) 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.

NOTE 1 The unit inch is not allowed in ISO documents; inch equivalents are given for information only.

Pre-cleaned sorbent tubes containing Tenax TA® are available commercially. Alternatively, 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 2 The determination of breakthrough volume is specified 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 safe sampling volume (SSV).

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