SLOVENSKI STANDARD
SIST EN ISO 16017-1:2002

01-maj-2002

Notranji in zunanji zrak ter zrak na delovnem mestu - Vzorļ enje in analiza hlapnih organskih spojin z absorpcijskimi cevkami in kapilarno plinsko kromatografijo s toplotno desorpcijo - 1. del: Vzorļ enje s ļ rpanjem (ISO 16017ï 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 (ISO 16017-1:2000)

Innenraumluft, Außenluft und Luft am Arbeitsplatz - Probenahme und Analyse flüchtiger organischer Verbindungen durch Sorptionsröhrchen/thermische Desorption/Kapillar-Gaschromatographie - Teil 1: Probenahme mit einer Pumpe
(ISO 16017-1:2000)

Air intérieur, air ambiant et air des lieux de travail -Echantillonnage et analyse des composés organiques volatils par tube a adsorption/désorption thermique/chromatographie en
phase gazeuse sur capillaire - Partie 1: Echantillonnage par pompage (ISO 16017-1:2000)

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English version

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

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This European Standard was approved by CEN on 30 September 2000.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN ISO 16017-1:2000 E

Foreword

The text of the International Standard ISO 16017-1:2000 has been prepared by Technical Committee ISO/TC 146 "Air quality" in collaboration with Technical Committee CEN/TC 264 "Air quality", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2001, and conflicting national standards shall be withdrawn at the latest by May 2001.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

NOTE FROM CMC: The foreword is susceptible to be amended on reception of the German language version. The confirmed or amended foreword, and when appropriate, the normative annex ZA for the references to international publications with their relevant European publications will be circulated with the German version.

Endorsement notice

The text of the International Standard ISO 16017-1:2000 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 16017-1

First edition
2000-11-15

Indoor, ambient and workplace air — Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography —

Part 1: Pumped sampling

Air intérieur, air ambiant et air des lieux de travail — Échantillonnage et analyse des composés organiques volatils par tube à adsorption/désorption thermique/chromatographie en phase gazeuse sur capillaire —

Partie 1: Échantillonnage par pompage

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Foreword

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

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

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

Attention is drawn to the possibility that some of the elements of this part of ISO 16017 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 16017-1 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 6, *Indoor air*.

ISO 16017 consists of the following parts, under the general title *Indoor, ambient and workplace air — Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography*:

— *Part 1: Pumped sampling*

— *Part 2: Diffusive sampling*

Annexes A and B form a normative part of this part of ISO 16017. Annexes C through F are for information only.

Indoor, ambient and workplace air — Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography —

Part 1:
Pumped sampling

1 Scope

This part of ISO 16017 gives general guidance for the sampling and analysis of volatile organic compounds (VOCs) in air. It is applicable to ambient, indoor and workplace atmospheres and the assessment of emissions from materials in small- or full-scale test chambers.

This part of ISO 16017 is appropriate for a wide range of VOCs, including hydrocarbons, halogenated hydrocarbons, esters, glycol ethers, ketones and alcohols. A number of sorbents [1)] are recommended for the sampling of these VOCs, each sorbent having a different range of applicability. Very polar compounds will generally require derivatization, very low boiling compounds will only be partially retained by the sorbents, depending on ambient temperature, and can only be estimated qualitatively. Semi-volatile compounds will be fully retained by the sorbents, but may only be partially recovered. Compounds for which this part of ISO 16017 has been tested are given in tables. This part of ISO 16017 may be applicable to compounds not listed, but in these cases it is advisable to use a back-up tube containing the same or stronger sorbent.

This part of ISO 16017 is applicable to the measurement of airborne vapours of VOCs in a concentration range of approximately 0,5 μg/ m^3 to 100 mg/m^3 individual compound.

The upper limit of the useful range is set by the sorptive capacity of the sorbent used and by the linear dynamic range of the gas chromatograph column and detector or by the sample-splitting capability of the analytical instrumentation used. The sorptive capacity is measured as a breakthrough volume of air, which determines the maximum air volume that shall not be exceeded when sampling.

The lower limit of the useful range depends on the noise level of the detector and on blank levels of analyte and/or interfering artefacts on the sorbent tubes. Artefacts are typically sub-nanogram for well-conditioned Tenax GR and carbonaceous sorbents such as Carbopack/Carbotrap type materials, carbonized molecular sieves and molecular sieves such as Spherocarb, or pure charcoal; at low nanogram levels for Tenax TA and at 5 ng to 50 ng levels for other porous polymers such as Chromosorbs and Porapaks. Sensitivity is typically limited to 0,5 μg/m^3 for 10-litre air samples with this latter group of sorbents because of their inherent high background.

The procedure specified in this part of ISO 16017 is applicable to low flowrate personal sampling pumps and gives a time-weighted average result. It is not applicable to the measurement of instantaneous or short-term fluctuations in concentration.

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1) The sorbents listed in annex C and elsewhere in this International Standard are those known to perform as specified under this part of ISO 16017. Each sorbent or product that is identified by a trademarked name is unique and has a sole manufacturer; however, they are widely available from many different suppliers. This information is given for the convenience of users of this part of ISO 16017 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 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 16017. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 16017 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ISO 6141:2000, *Gas analysis — Requirements for certificates for calibration gases and gas mixtures.*

ISO 6145-1:1986, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 1: Methods of calibration.*

ISO 6145-3:1986, *Gas analysis — Preparation of calibration gas mixtures — Dynamic volumetric methods — Part 3: Periodic injections into a flowing gas stream.*

ISO 6145-4:1986, *Gas analysis — Preparation of calibration gas mixtures — Dynamic volumetric methods — Part 4: Continuous injection method.*

ISO 6145-5:—[2)], *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 5: Capillary calibration devices.*

ISO 6145-6:—[2)], *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 6: Critical orifices.*

ISO 6349:1979, *Gas analysis — Preparation of calibration gas mixtures — Permeation method.*

EN 1076:1997, *Workplace atmospheres — Pumped sorbent tubes for the determination of gases and vapours — Requirements and test methods.*

3 Terms and definitions

For the purposes of this part of ISO 16017, the following terms and definitions apply.

3.1
breakthrough volume
volume of test atmosphere that can be passed through a sorbent tube before the concentration of eluting vapour reaches 5 % of the applied test concentration

NOTE 1 The breakthrough volume varies with the vapour and the sorbent type.

NOTE 2 See reference [4]. 3.2

retention volume
elution volume at peak maximum of a small aliquot of an organic vapour eluted from a sorbent tube by air or chromatographic carrier gas

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2) To be published.

4 Principle

A measured volume of sample air is drawn through one (or more) sorbent tubes in series; an appropriate sorbent (or sorbents) being selected for the compound or mixture to be sampled. Provided suitable sorbents are chosen, volatile organic components are retained by the sorbent tube and thus are removed from the flowing air stream. The collected vapour (on each tube) is desorbed by heat and is transferred under inert carrier gas into a gas chromatograph equipped with a capillary column and a flame ionization detector or other suitable detector, where it is analysed. Analytical calibration is achieved by means of liquid or vapour spiking onto a sorbent tube.

5 Reagents and materials

During the analysis, use only reagents of recognized analytical reagent grade.

Fresh standard calibration blend solutions should be prepared weekly, or more frequently if evidence is noted of deterioration, e.g. condensation reactions between alcohols and ketones.

5.1 Volatile organic compounds, for calibration purposes, using either liquid spiking (5.7 and 5.8) or vapour spiking (5.4 to 5.6) onto sorbent tubes.

5.2 Dilution solvent, for preparing calibration blend solution for liquid spiking (5.7). This should be of chromatographic quality. It shall be free from compounds co-eluting with the compound or compounds of interest (5.1).

NOTE Methanol is frequently used. Alternative dilution solvents e.g. ethyl acetate or cyclohexane, can be used, particularly if there is no possibility of reaction or chromatographic co-elution.

5.3 Sorbents, of recommended particle size 0,18 mm to 0,25 mm (60 to 80 mesh).

Each sorbent should be preconditioned under a flow of inert gas by heating it overnight (= 16 h) at a temperature at least 25 °C below the published maximum for that sorbent before packing the tubes. To prevent recontamination of the sorbents, they shall be kept in a clean atmosphere during cooling to room temperature, storage, and loading into the tubes. Wherever possible, analytical desorption temperatures should be kept below those used for conditioning. Tubes prepacked by the manufacturer are also available for most sorbents and as such only require conditioning.

NOTE 1 Sorbent particle sizes larger than 0,18 mm to 0,25 mm may be used but the breakthrough characteristics given in Tables 1 to 6 may be affected. Smaller sorbent particle size ranges are not recommended because of back-pressure problems.

NOTE 2 A description of sorbents is given in annex C and a guide for sorbent selection is given in annex D. Equivalent sorbents may be used. A guide to sorbent conditioning and analytical desorption parameters is given in annex E.

5.4 Calibration standards, preferably prepared by loading required amounts of the compounds of interest on sorbent tubes from standard atmospheres (see 5.5 and 5.6), as this procedure most closely resembles the practical sampling situation.

If this way of preparation is not practicable, standards may be prepared by a liquid spiking procedure (see 5.7 and 5.8), provided that the accuracy of the spiking technique is either:

a) established by using procedures giving spiking levels fully traceable to primary standards of mass and/or volume, or,

b) confirmed by comparison with reference materials, if available, standards produced using standard atmospheres, or results of reference measurement procedures.

NOTE The loading ranges given in 5.6, 5.7 and 5.8 are not mandatory and approximate to the application range given in clause 1 for a 2-litre sample. For specific applications in which larger volumes are used to measure lower concentrations, other loading ranges may be more appropriate.

5.5 Standard atmospheres.

Prepare standard atmospheres of known concentrations of the compound(s) of interest by a recognized procedure. Methods described in ISO 6141, the appropriate part of ISO 6145 and ISO 6349 are suitable. 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.

5.6 Standard sorbent tubes, loaded by spiking from standard atmospheres.

Prepare loaded sorbent tubes by passing an accurately known volume of the calibration 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. Prepare standard atmospheres equivalent to 10 mg/m^3 and 100 μg/m^3. For workplace air, load sorbent tubes with 100 ml, 200 ml, 400 ml, 1 l, 2 l, or 4 l of the 10 mg/m^3 atmosphere. For ambient or indoor air, load sorbent tubes with 100 ml, 200 ml, 400 ml, 1 l, 2 l, 4 l or 10 l of the 100 μg/m^3 atmosphere.

5.7 Solutions for liquid spiking.

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

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 (5.2), stopper and shake to mix.

5.7.2 Solution containing approximately 1 mg/ml of liquid components.

Introduce 50 ml of dilution solvent into a 100 ml volumetric flask. Add 10 ml of solution 5.7.1 Make up to 100 ml with dilution solvent, stopper and shake to mix.

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

Accurately weigh approximately 10 mg 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 (5.2), stopper and shake to mix.

5.7.4 Solution containing approximately 10 μg/ml of liquid components.

Introduce 50 ml of dilution solvent into a 100 ml volumetric flask. Add 10 ml of solution described in 5.7.3. Make up to 100 ml with dilution solvent, stopper and shake to mix.

5.7.5 Solution containing approximately 1 mg/ml of gas components.

For gases, e.g. ethylene oxide, a high level calibration solution may be prepared as follows. Obtain gas at atmospheric pressure by filling a small plastic gas bag from a gas cylinder containing pure gas. Fill a 1-ml gas-tight syringe with 1 ml of the pure gas and close the valve of the syringe. Using a 2-ml septum vial, add 2 ml dilution solvent and close with the septum cap. Insert the tip of the syringe needle through the septum cap into the dilution solvent. Open the valve and withdraw the plunger slightly to allow the dilution solvent to enter the syringe. The action of the gas dissolving in the dilution solvent creates a vacuum, and the syringe fills with solvent. Return the solution to the flask. Flush the syringe twice with the solution and return the washings to the flask. Calculate the mass of gas added using the gas laws, i.e. 1 mole of gas at STP (standard temperature and pressure: 273,15 K and 1 013,25 hPa) occupies 22,4 litres, but correct for any non-ideality of the particular pure gas compound.