

SLOVENSKI STANDARD

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Indoor, ambient and workplace air - Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography - Part 2: Diffusive sampling (ISO 16017-2:2003)

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Innenraumluf, Außenluft und Luft am Arbeitsplatz - Probenahme und Analyse flüchtiger organischer Verbindungen durch Sorptionsröhrchen/thermische Desorption/Kapillar-Gaschromatographie - Teil 2: Probenahme mit Passivsammeln (ISO 16017-2:2003)

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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 2: Echantillonnage par diffusion (ISO 16017-2:2003)

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ICS:

13.040.01 Kakovost zraka na splošno Air quality in general

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en

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 16017-2

May 2003

ICS 13.040.01

English version

Indoor, ambient and workplace air - Sampling and analysis of
volatile organic compounds by sorbent tube/thermal
desorption/capillary gas chromatography - Part 2: Diffusive
sampling (ISO 16017-2:2003)

Air intérieur, air ambiant et air des lieux de travail -
Echantillonnage et analyse des composés organiques
volatils par tube à adsorption/désorption
thermique/chromatographie en phase gazeuse sur
capillaire - Partie 2: Echantillonnage par diffusion (ISO
16017-2:2003)

Innenraumluft, Außenluft und Luft am Arbeitsplatz -
Probenahme und Analyse flüchtiger organischer
Verbindungen durch Sorptionsröhrchen/thermische
Desorption/Kapillar- Gaschromatographie - Teil 2:
Probenahme mit Passivsammlern (ISO 16017-2:2003)

This European Standard was approved by CEN on 21 March 2003.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

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.

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

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EN ISO 16017-2:2003 (E)

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Foreword

This document (EN ISO 16017-2:2003) 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 November 2003, and conflicting national standards shall be withdrawn at the latest by November 2003.

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, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of ISO 16017-2:2003 has been approved by CEN as EN ISO 16017-2:2003 without any modifications.

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**Indoor, ambient and workplace air —
Sampling and analysis of volatile organic
compounds by sorbent tube/thermal
desorption/capillary gas
chromatography —****Part 2:
Diffusive sampling**
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*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 2: Échantillonnage par diffusion

Reference number
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ISO 16017-2:2003(E)

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 16017-2 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*

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

Part 2: Diffusive 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 indoor, ambient and workplace air.

This part of ISO 16017 is applicable to a wide range of VOCs, including hydrocarbons, halogenated hydrocarbons, esters, glycol ethers, ketones and alcohols. A number of sorbents¹⁾ are recommended for the sampling of these VOCs, each sorbent having a different range of applicability. Very polar compounds generally require derivatisation; very low-boiling compounds are only partially retained by the sorbents and can only be estimated qualitatively. Semi-volatile compounds are fully retained by the sorbents, but may only be partially recovered.

This part of ISO 16017 is applicable to the measurement of airborne vapours of VOCs in a mass concentration range of approximately 0,002 mg/m³ to 100 mg/m³ individual organic for an exposure time of 8 h, or 0,3 µg/m³ to 300 µg/m³ individual organic for an exposure time of four weeks.

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 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 such as Spherocharb and pure charcoals. Artefacts are typically at low nanogram levels for Tenax TA and at 5 ng to 50 ng levels for other porous polymers such as Chromosorb and Porapak.

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*

1) The sorbents listed in Annex B and elsewhere in this part of ISO 16017 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.

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3 Principle

The diffusive sampler (or samplers) is exposed to air for a measured time period. The rate of sampling is determined by prior calibration in a standard atmosphere (see 8.6). The organic vapour migrates down the tube by diffusion and is collected on the sorbent (see Annex A). 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. The analysis is calibrated by means of liquid or vapour spiking onto a sorbent tube.

Information on possible saturation of the sorbent bed, the effect of transients and the effect of face velocity is given in Annex A. Annex A also explains the dependence of effective uptake rates on the concentration level of pollutants and the time of diffusive sampling, for non-ideal sorbents, which results in different values being given in Tables 1 and 2. Further detailed information on the theory of performance of diffusive samplers is given in prEN 13528-3 [1].

4 Reagents and materials

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

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

4.1 Volatile organic compounds.

A wide range of VOCs are required as reagents for calibration purposes, using either liquid spiking (4.7 and 4.8) or vapour spiking (4.4 to 4.6) onto sorbent tubes.

4.2 Dilution solvent, for preparing calibration blend solutions for liquid spiking (4.7).

The solvent should be of chromatographic quality. It shall be free from compounds co-eluting with the compound(s) of interest (4.1).

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.

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

Each sorbent should be preconditioned under a flow of inert gas by heating it overnight at a temperature at least 25 °C below the published maximum for that sorbent before packing the tubes. 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 prepaced by the manufacturer are also available for most sorbents and as such only require conditioning. Care should be taken with the disposal of the sorbents, using normal laboratory practice.

NOTE A guide for sorbent selection is given in Annex C. Equivalent sorbents may be used. A guide to sorbent conditioning and analytical desorption parameters is given in Annex D. In most cases the sorbents can be used for indoor air measurements as well as for ambient air and workplace atmosphere measurements.

4.4 Calibration standards.

Calibration standards are preferably prepared by loading required amounts of the compounds of interest on sorbent tubes from standard atmospheres (see 4.5 and 4.6), 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 4.7 and 4.8) provided that the accuracy of the spiking technique is established by one of the following methods:

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

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

4.5 Standard atmospheres, of known concentrations of the compound(s) of interest.

Prepare standard atmospheres by an independent method. Methods described in ISO 6141 and several parts of ISO 6145 are suitable (see Bibliography). 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.

4.6 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 of mass concentrations equivalent to 10 mg/m³ and 100 µg/m³. For workplace air, load sorbent tubes with 100 ml, 200 ml, 400 ml, 1 l, 2 l, or 4 l of the 10 mg/m³ 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/m³ atmosphere.

4.7 Solutions for liquid spiking.

4.7.1 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 (4.2), stopper and shake to mix.

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4.7.2 Solutions 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 4.7.1. Make up to 100 ml with dilution solvent, stopper and shake to mix.

4.7.3 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 (4.2), stopper and shake to mix.

4.7.4 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 4.7.3. Make up to 100 ml with dilution solvent, stopper and shake to mix.

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

For gases, e.g. ethylene oxide, a high-level calibration solution can 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.

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4.7.6 Solution containing approximately 10 µg/ml of gas components

For gases, e.g. ethylene oxide, a low level calibration solution may be prepared as follows. Obtain pure gas at atmospheric pressure by filling a small plastic gas bag from a gas cylinder. Fill a 10 µl gas-tight syringe with 10 µl 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 occupies 22,4 litres.

4.8 Standard sorbent tubes loaded by liquid spiking

Loaded sorbent tubes are prepared by injecting aliquots of standard solutions onto clean sorbent tubes as follows. A sorbent tube is fitted into the injection unit (5.7) through which inert purge gas is passed at 100 ml/min and a 1 µl to 4 µl aliquot of an appropriate standard solution injected through the septum. After 5 min, the tube is then disconnected and sealed. Prepare fresh standards with each batch of samples. For workplace air, load sorbent tubes with 1 µl to 5 µl of solution 4.7.1, 4.7.2 or 4.7.5. For ambient and indoor air, load sorbent tubes with 1 µl to 5 µl of solution 4.7.3, 4.7.4 or 4.7.6.

5 Apparatus

Use ordinary laboratory apparatus and the following devices.

5.1 Sorbent tubes.

These tubes shall be compatible with the thermal desorption apparatus to be used (5.6). Typically, but not exclusively, they are constructed of stainless steel tubing of dimensions 6,3 mm (1/4 in) OD, 5 mm ID and 90 mm long. Tubes of other dimensions may be used, but the uptake rates given in Table 1 are based on these tube dimensions. For labile analytes, such as sulfur-containing compounds, glass-lined or glass tubes (typically 4 mm ID) should be used. Mark one end of the tube, for example by a scored ring, about 10 mm from the (diffusive) sampling end. Pack the tubes with preconditioned sorbents so that the sorbent bed will be within the desorber heated zone and a consistent gap of about 14 mm is retained at the marked (diffusive) end of the tube.

Uptake rates in Table 1 are given for tubes with a nominal air gap (between sorbent bed and diffusive end cap) of at least 14 mm. In practice, packed tube dimensions vary^[2], and tubes should be rejected where the air gap (between stainless steel screen retaining the sorbent bed and the end of the tube) is outside the range 14,0 mm to 14,6 mm.

Tubes contain between 200 mg and 1 000 mg sorbent, depending on sorbent density, which is typically about 250 mg porous polymer, or 500 mg carbon molecular sieve or graphitized carbon. The sorbents are retained by a stainless steel gauze at the diffusion end and an unsilanized glass wool plug and/or a second stainless gauze at the other end.

5.2 Sorbent tube end caps.

The tubes shall be sealed, e.g. with metal screw cap fittings with PTFE seals.

5.3 Sorbent tube end caps for sampling.

The diffusive end cap is similar to 5.2, but allows the ingress of vapour through a metal gauze, the size of the opening being the same as the cross-section of the tube.

Some versions of the end cap incorporate a silicone membrane next to the gauze.

5.4 Syringes.

A precision 10 µl liquid syringe readable to 0,1 µl, a precision 10 µl gas-tight syringe readable to 0,1 µl and a precision 1 ml gas-tight syringe readable to 0,01 ml.

5.5 Gas chromatograph, fitted with a flame ionization detector, photoionization detector, mass spectrometric or other suitable detector capable of detecting an injection of 0,5 ng toluene with a signal-to-noise ratio of at least 5 to 1, and including a gas chromatograph capillary column capable of separating the analytes of interest from other components.

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

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 is adjustable, as is the carrier gas flow rate. The apparatus should also incorporate additional features, such as automatic sample tube loading, leak-testing, and a cold trap in the transfer line to concentrate the desorbed sample (8.2). The desorbed sample, contained in the purge gas, is routed to the gas chromatograph and capillary column via a heated transfer line.

5.7 Injection facility for preparing standards by liquid spiking.

A conventional gas chromatographic injection port may be used for preparing sample tube 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 port should be adapted if necessary to fit the sample tube. This can be done conveniently by means of a compression coupling with an O-ring seal.

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6 Sample tube conditioning

Prior to use, tubes should be reconditioned by desorbing them at a temperature at or just above the analytical desorption temperature (see Annex D) for 10 min with a carrier gas flow of at least 100 ml/min. The carrier gas flow should be towards the diffusive sampling end to prevent recontamination of the sorbents. Tubes should then be analysed, using routine analytical parameters, to ensure that the thermal desorption blank is sufficiently small. If the blank is unacceptable, tubes should be reconditioned by repeating this procedure. Once a sample has been analysed, the tube may be reused to collect a further sample immediately. However, it is advisable to check the thermal desorption blank if the tubes are left for an extended period before reuse, or if sampling for a different analyte is envisaged. Tubes should be sealed with metal screwcaps with combined PTFE ferrule fittings and stored in an airtight container when not sampling or being conditioned.

NOTE The sorbent tube blank level is acceptable if interfering peaks are no greater than 10 % of the typical areas of the analytes of interest.

7 Sampling

Select a sorbent tube (or tubes) appropriate for the compound or mixture to be sampled. Guidance on suitable sorbents is given in Tables 1 and 2 and Annex B.

Immediately before sampling, remove the storage end cap from the marked end of the sample tube and replace it with a diffusion end cap. Make sure the diffusion cap is properly seated and the other end cap is in place.

When used for personal sampling, the tube(s) should be mounted in the breathing zone. When used for fixed-location sampling, choose a suitable site; for indoor air in accordance with ISO 16000-1. For ambient air, recommendations for site selection and for the protection of samples from adverse environmental conditions are given in Annex A and prEN 13528-3 [1]. Attention shall be paid to three main considerations: air velocity, protection from precipitation, and security. More information is given in the next paragraph, in A.5 and reference [1].