

---

---

**Indoor air —**

Part 6:

**Determination of volatile organic  
compounds in indoor and test chamber  
air by active sampling on Tenax TA<sup>®</sup>  
sorber, thermal desorption and gas  
chromatography using MS/FID**

iTeh STANDARD PREVIEW  
(standards.iteh.ai)

*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<sup>®</sup>; désorption thermique et chromatographie en phase  
gazeuse utilisant MS/FID*



**PDF disclaimer**

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

[ISO 16000-6:2004](#)

<https://standards.iteh.ai/catalog/standards/sist/cfa5039e-24e5-49c2-8029-cf64d52f7887/iso-16000-6-2004>

© ISO 2004

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.org](mailto:copyright@iso.org)  
Web [www.iso.org](http://www.iso.org)

Published in Switzerland

# Contents

Page

Foreword.....	iv
Introduction .....	v
1 Scope.....	1
2 Normative references .....	1
3 Terms and definitions.....	2
4 Principle .....	2
5 Reagents and materials.....	3
6 Apparatus.....	4
7 Conditioning and storage of sorbent tubes .....	6
7.1 Conditioning .....	6
7.2 Storage of conditioned sorbent tubes before sampling .....	6
8 Calibration of pump .....	6
9 Sampling .....	6
9.1 Indoor air sampling.....	6
9.2 Test chamber air sampling.....	7
9.3 Sampling volumes .....	7
9.4 Storage of loaded samples .....	7
9.5 Field blanks .....	7
10 Analysis.....	8
10.1 General .....	8
10.2 Thermal desorption.....	8
10.3 Temperature programme.....	8
10.4 Analysis of the samples .....	9
11 Identification of single VOCs .....	9
12 Concentration of analytes in the sampled air .....	9
12.1 General .....	9
12.2 Volatile organic compounds (VOCs).....	9
12.3 Total volatile organic compounds (TVOCs) .....	10
12.4 VVOC and SVOC compounds observed outside the TVOC range .....	11
13 Performance characteristics.....	11
14 Test report.....	12
15 Quality control.....	13
Annex A (informative) Examples of compounds detected in indoor air and from building products in test chambers .....	14
Annex B (informative) Safe sampling volumes (SSV) for selected organic vapours sampled on Tenax TA® .....	20
Annex C (informative) Storage recovery of solvents on Tenax TA® sorbent tubes .....	22
Bibliography .....	24

## 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 formaldehyde 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<sup>®</sup> 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.

## iTeh STANDARD PREVIEW (standards.iteh.ai)

[ISO 16000-6:2004](https://standards.iteh.ai/catalog/standards/sist/cfa5039e-24e5-49c2-8029-cf64d52f7887/iso-16000-6-2004)

<https://standards.iteh.ai/catalog/standards/sist/cfa5039e-24e5-49c2-8029-cf64d52f7887/iso-16000-6-2004>

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

ISO 16000-6:2004

<https://standards.iteh.ai/catalog/standards/sist/cfa5039e-24e5-49c2-8029-cf64d52f7887/iso-16000-6-2004>

## Indoor air —

### Part 6:

## Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA<sup>®</sup> 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<sup>®</sup> sorbent with subsequent thermal desorption and gas chromatographic analysis<sup>[1]</sup>.

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:—<sup>2)</sup>, *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<sup>®</sup> 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<sup>[3]</sup>. SVOCs have vapour pressures between 10<sup>-2</sup> kPa and 10<sup>-8</sup> 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<sup>[3]</sup>. VOCs generally have saturation vapour pressures at 25 °C greater than 10<sup>2</sup> kPa.

iTeh STANDARD PREVIEW

(standards.iteh.ai)

#### 3.3

##### **very volatile organic compound**

##### **VVOC**

organic compound whose boiling point is in the range from (0 °C to 50 °C) to (50 °C to 100 °C)

<https://standards.iteh.ai/catalog/standards/sist/cfa5039e-24e5-49c2-8029->

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<sup>[3]</sup>. VVOCs typically have vapour pressures greater than 15 kPa<sup>[3]</sup>.

#### 3.4

##### **total volatile organic compounds**

##### **TVOC**

the sum of volatile organic compounds, sampled on Tenax TA<sup>®</sup>, 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<sup>®</sup> 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<sup>®</sup>**, particle size 0,18 mm to 0,25 mm (60 mesh to 80 mesh).

Tenax TA<sup>®</sup> is a porous polymer based on 2,6-diphenylene oxide. Manufactured Tenax TA<sup>®</sup> contains quantities of impurities, which shall be removed before using it for VOC sampling. Perform cleaning by thermal conditioning the Tenax TA<sup>®</sup> 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<sup>®</sup> 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.** (standards.iteh.ai)

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<sup>3</sup>. 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<sup>3</sup> 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.

(standards.iteh.ai)

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<sub>2</sub>.**

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<sup>®</sup> 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<sup>®</sup> 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.

ISO 16000-6:2004

Bubble meter or other suitable device for gas flow calibration  
<https://standards.iteh.ai/catalog/standards/sist/cfa5039e-24e5-49c2-8029-c164d5217887/iso-16000-6-2004>

**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<sup>3)</sup> 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.

**iTeh STANDARD PREVIEW**  
(standards.iteh.ai)  
<https://standards.iteh.ai/catalog/standards/sist/cfa5039e-24e5-49c2-8029-cf64d52f7887/iso-16000-6-2004>

## 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.