BS ISO 16000-25:2011

BSI Standards Publication

Indoor air

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

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National foreword

This British Standard is the UK implementation of ISO 16000-25:2011.

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.

ISBN 978 0 580 63474 1

ICS 13.040.20

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 August 2011.

Amendments issued since publication

Date	Text affected

INTERNATIONAL STANDARD

ISO 16000-25

First edition
2011-07-01

Indoor air —

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

Air intérieur —

Partie 25: Dosage de l'émission de composés organiques semi-volatils des produits de construction — Méthode de la micro-chambre

Reference number
ISO 16000-25:2011(E)

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Published in Switzerland

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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 16000-25 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 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)*

- *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*

Introduction

The determination of semi-volatile organic compounds (SVOCs) emitted from building products using micro-chambers in conjunction with standardized sampling, storage of samples and preparation of test specimens aims, for example, to:

- provide manufacturers, builders, and end-users with emission data useful for the evaluation of the impact of building products on the indoor air quality;
- promote the development of improved products.

The measurement method specified in this part of ISO 16000 is applicable to products used in construction such as board materials, wallpapers, flooring materials, insulation materials, adhesives, paints, and their combinations.

SVOCs such as phthalic esters are found in many construction materials. If emitted into the indoor environment they adhere to many surfaces and can become a persistent indoor air contaminant.

This part of ISO 16000 specifies a test procedure for measuring SVOC emissions from construction products and materials. This method can, in principle, be used for most building products used indoors.

ISO 16017[6][7] and ISO 12219[1]–[5] also focus on volatile organic compound (VOC) measurements.

Indoor air —

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

1 Scope

This part of ISO 16000 specifies a test method for determination of the area-specific emission rate of semi-volatile organic compounds (SVOCs) from newly produced building products or furnishings under defined climate conditions using a micro-chamber. The method can in principle also be applied to aged products. This measurement method is applicable to products and materials, such as board materials, wallpapers, flooring materials, insulation materials, adhesives, paints, and their combinations.

Sampling, transport and storage of materials to be tested, and preparation of test specimens are specified in ISO 16000-11. Air sampling and analytical methods for the determination of SVOCs are specified in ISO 16000-6 and ISO 16017-1.

An example of a micro-chamber is described in Annex B.

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 554, *Standard atmospheres for conditioning and/or testing — Specifications*

ISO 16000-6, *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*

ISO 16000-11, *Indoor air — Part 11: Determination of the emission of volatile organic compounds from building products and furnishing — Sampling, storage of samples and preparation of test specimens*

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*

3 Terms and definitions

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

3.1
air change rate for micro-chamber
n
ratio of the volume of clean air brought into the micro-chamber per hour and the free micro-chamber volume measured in identical units

3.2
air flow rate for micro-chamber
$q_{V,\mathrm{c}}$
air volume entering into the micro-chamber per time

3.3
air velocity
air speed over the surface of the test specimen

[ISO 16000-9:2006, 3.3]

3.4
area specific air flow rate
q_{VA}
ratio between the supply air flow rate and the area of the test specimen

[ISO 16000-9:2006, 3.4]

3.5
area specific emission rate for SVOC
q_{mA}
building product specific rate describing the mass of a substance emitted from an exposed area per time at a given time from the start of the test

NOTE 1 For the purposes of this part of ISO 16000, the emission consists of SVOC.

NOTE 2 The term “area specific emission rate” is sometimes used in parallel with the term “emission factor”.

3.6
building product
product produced for incorporation in a permanent manner in construction works

[ISO 16000-9:2006, 3.5]

3.7
field blank
m_{t0}
⟨SVOC emission from building products⟩ mass of SVOC in the sorbent tube when all operations except air sampling are performed

NOTE Used in order to consider contamination originating from the sorbent tube itself and contamination originating in opening, closing, and transportation.

3.8
inert gas
gas without active chemical or other properties

NOTE Normally, helium (He) gas or nitrogen (N_2) is used as the gas for thermal desorption (TD) of SVOC adsorbed in the micro-chamber (see Reference [8]).

3.9
mass collected in control test
m_0
sum of mass during first and second steps of test without introducing specimen

3.10
mass collected in first step
m_1
mass of SVOC sampled and measured at the micro-chamber outlet, which are emitted and not absorbed in the micro-chamber

3.11
mass collected in second step

m_2

mass of SVOC sampled and measured at the micro-chamber outlet when thermal desorption is performed

3.12
micro-chamber

container enabling control of conditions for measurement of SVOC emissions from building materials

NOTE Micro-chambers typically range in volume as described in B.1.

3.13
recovery

measured mass of a target semi-volatile organic compound in the air leaving the micro-chamber during thermal desorption (second step) divided by the mass of target semi-volatile organic compound added to the micro-chamber

NOTE 1 Recovery is expressed as a percentage.

NOTE 2 The recovery provides information about the performance of the entire method.

3.14
sample

part or piece of a building product that is representative of the production

[ISO 16000-9:2006, 3.10]

3.15
sampling period

duration over which a sample is taken

NOTE The sampling period is the time during which air is sampled from the outlet of the micro-chamber using sorbent tubes or other devices.

3.16
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 (Reference [9]).

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 can be used for classification of organic chemicals. SVOCs have vapour pressures between 10^{-2} mPa and 10 Pa.

3.17
sorbent tube blank

value of SVOC in the sorbent tube itself before air sampling

3.18
target semi-volatile organic compound

product specific semi-volatile organic compound

3.19
test specimen

⟨SVOC emission from building products⟩ part of the sample specially prepared for emission testing in a micro-chamber cell in order to simulate the emission behaviour of the material or product that is tested

3.20
test start
time of placing the test specimen in the micro-chamber

3.21
total mass collected in first and second step
sum of mass collected during first and second steps of test

4 Symbols

Symbol	Meaning	Unit
A	surface area of test specimen	square metres
A_c	inner surface area of the micro-chamber	square metres
S_L	surface area ratio (equal to A_c/A)	square metres per square metre
m_0	mass collected in control test	micrograms
m_1	mass collected in first step	micrograms
m_2	mass collected in second step	micrograms
m_{1+2}	mass collected in first and second steps	micrograms
$m_{\mathrm{t}0}$	field blank	micrograms
n	air change rate for micro-chamber	changes per hour
q_{mA}	area specific emission rate	micrograms per square metre hour
q_{VA}	area specific air flow rate (equal to $q_{V,\mathrm{c}}/A$)	cubic metres per square metre hour
$q_{V,\mathrm{c}}$	air flow rate for micro-chamber	cubic metres per hour
t	duration of first phase	hours
V	air volume of micro-chamber	cubic metres

5 Principle

The principle of the test is to determine the area specific emission rates of SVOCs emitted from the surface of a product test specimen. Although SVOCs are emitted in the micro-chamber, the greater part of these emissions are adsorbed in the chamber at temperatures of 40 °C or below. Therefore, in this test, the area specific emission rate of SVOC for a building material which is the object of a test is determined from the mass collected in the first and second steps. The outcome of the test is the mean rate of emission of SVOCs from the product over a 24 h period. For specific purposes, the emission rate over a different period of time could be determined using the same procedure, but varying the duration of the first step.

6 Micro-chamber system

6.1 General

A micro-chamber system designed and operated to determine area specific emission rates of SVOCs from building products shall contain the following: micro-chamber, clean air generation and humidification system, and monitoring and control systems to ensure that the test is carried out according to specified conditions.

The design of the micro-chamber may allow solid products with a smooth surface to be placed in or on (or under) the micro-chamber such that the sample itself forms a wall of the micro-chamber. This is analogous to the approach used in ISO 16000-9 and ISO 16000-10. In this case, it is important that the sample surface be sealed against the micro-chamber so that emissions from the edges and rear of the test specimen are excluded. To secure airtightness, other products shall be placed in specially constructed test specimen holders.

General specifications and requirements that apply to all types of micro-chambers in this part of ISO 16000 are given in 6.2 to 6.7.

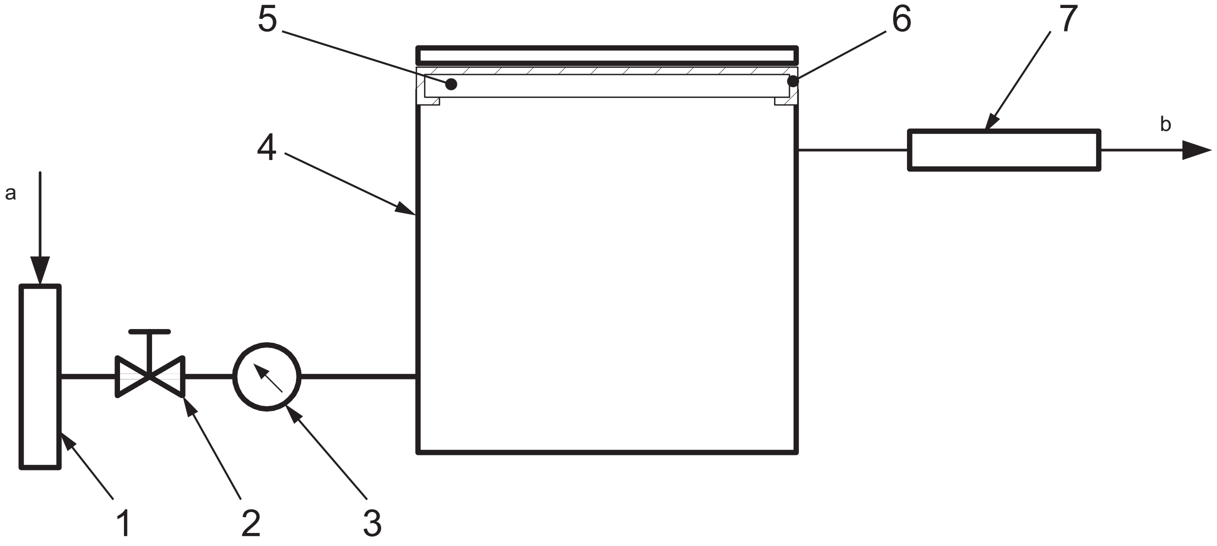
Quality assurance and quality control activities shall be carried out as specified in Annex A.

6.2 Micro-chamber

An appropriate volume size and an appropriate size ratio of the dimension has been tested and is given in Table B.1. The micro-chamber and the parts of the sampling system coming into contact with the emitted SVOCs (all tubings and couplings) are normally made of glass or inert non-outgassing materials, such as inert-coated stainless steel and polished stainless steel. However, in all cases, the requirements specified in 6.3 and 6.7 shall be fulfilled. Depending on the material of micro-chamber construction (e.g. some types of glass), surface treatment may be required to aid thermal desorption.

NOTE Polished stainless steel can catalyse degradation of some SVOCs.

The sealing material housing the test specimen shall be low emitting and low adsorbing, and shall not contribute to the micro-chamber background concentration. Schematic diagrams of the micro-chamber apparatus are shown in Figure 1 and Figure 2.



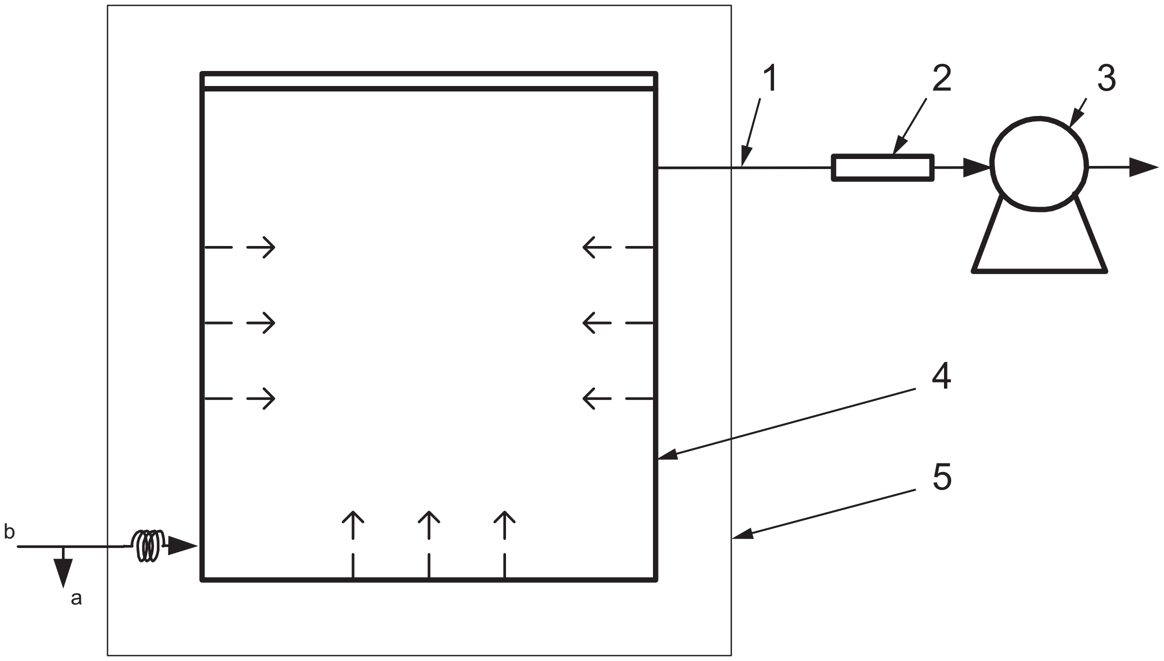
Key

1	clean air supply
2	air flow regulator
3	air flow meter
4	micro-chamber
5	test specimen
6	sealing material
7	manifold air sampling (sorbent tube)

[a] Air inlet.

[b] Exhaust outlet.

Figure 1 — Schematic diagram of a type of micro-chamber apparatus as used in step 1 of the test



Key

1 transfer line
2 sorbent tube
3 sampling pump
4 micro-chamber
5 chamber heating device

[a] Vent line.
[b] Inert gas inlet.

Figure 2 — Schematic diagram of a type of micro-chamber apparatus as used in step 2 of the test

6.3 Surface area ratio

The ratio of the surface area of the test specimen to the inner surface area of the micro-chamber shall be 0,15 ± 0,007 5.

NOTE A surface area ratio outside this range can give different measurement results.

6.4 Airtightness

The micro-chamber shall have an airtight condition, in which exchange of air with uncontrolled external air is minimal.

The emission test chamber shall be operated slightly above atmospheric pressure to avoid influence from the laboratory atmosphere.

NOTE One method of minimizing ingress of laboratory air during the test is to ensure a slightly positive pressure within the micro-chamber. One way this can be achieved is to supply air to the micro-chamber at a rate that is ~50 % faster than the pumped sampling rate leaving the micro-chamber. If this approach is followed, a vent line can be conveniently installed immediately before the air inlet to the micro-chamber, allowing excess air to be discharged away from the immediate testing location.

6.5 Air supply devices

The micro-chamber shall be provided with a device, such as a flow control device, which makes it possible to control the ventilation rate continuously to a set numerical value.

6.6 Sealing of test specimen

The edges and rear surface of the test specimen shall be sealed. A low emission, low adsorption material shall be used as the seal.

6.7 Recovery and sink effects

A standard solution of target SVOC within an accuracy of ±10 % shall be prepared and a known mass of the solution shall be injected into the micro-chamber. The micro-chamber shall be heated to 200 °C to 220 °C and the desorbed compounds shall be sampled in a sorbent tube in a procedure analogous to step 2 of this test. The same mass of the standard solution shall be directly injected into another sorbent tube. The target compounds adsorbed into the sorbent tubes shall be determined by thermal desorption and gas chromatography-mass spectrometry (TD-GC/MS). The ratio of the result obtained from the standard addition into the micro-chamber to that from the direct injection shall be treated as the recovery. The recovery ratio shall be greater than 80 %. The results of this recovery test shall be reported in the test report as expected concentration versus measured concentration.

NOTE Sink effects, leaks or poor calibration can cause difficulties in meeting the minimum requirements. Sink and adsorption characteristics are very much dependent on the type of compound emitted. Additional recovery tests using target SVOCs with different molecular masses and polarity can be used to increase understanding of these effects.

6.8 Air purification device

Clean air shall be provided using an air purification device or from a clean air cylinder, so that the air supplied to the micro-chamber is as pure as possible. The contaminant level is specified in 8.3.

6.9 Temperature and humidity control device

For temperature control, the micro-chamber shall be placed in a test location, such as a thermostatic oven, which can maintain the micro-chamber at the necessary temperature.

Relative humidity should be controlled by mixing dry air and air at the required humidity during the first step of the test. Temperature and relative humidity shall be monitored independently from the temperature and humidity control system, and dew condensation shall be prevented in the micro-chamber. Water for humidification should be pure without SVOCs. The contaminant level is specified in 8.3.

6.10 Flow meter

The correct ventilation rate in the micro-chamber shall be measured by using a flow meter installed at the outlet of the micro-chamber.

6.11 Thermostatic oven

The temperature range should be between 23 °C and 250 °C.

The temperature of the thermostatic oven should be controlled within an accuracy of ±0,5 °C and a temperature distribution accuracy of ±2 °C.

6.12 Sampling pump

The pump should be controlled within an accuracy of ±10 %. When a transfer line is used, the length shall be as short as possible, in order to maintain the same temperature as in the micro-chamber. A low adsorptive material shall be used for the transfer line (outlet-line).

6.13 Oven to heat micro-chamber

In order to prevent oxidization of SVOC, inert gas should be used. The heating device shall be capable of maintaining the micro-chamber temperature at approximately 250 °C.

The gas supply line shall also be maintained at the specified temperature.

NOTE If it can be demonstrated that the SVOC compound of interest does not oxidize during thermal desorption in air, pure dry air can be used instead of inert gas.

7 Apparatus and materials

The equipment necessary for carrying out an emission test are listed below.

7.1 Micro-chamber (6.2).

7.2 Air supply device (6.5).

7.3 Material for sealing (6.6) rear and edges of test specimen.

7.4 Air purification device (6.8).

7.5 Flow meter (6.10).

7.6 Temperature and humidity device (6.9).

7.7 Means of heating micro-chamber (6.13).

7.8 Sorbent and sample tube.

7.9 Analytical instrument. SVOC retained on the sorbent tube shall be analysed using TD and gas chromatography (GC) (flame ionization or mass spectrometry detection) as specified in ISO 16000-6 and ISO 16017-1.

8 Test conditions

8.1 Temperature and relative humidity in first step test

Products for use in Europe and North America shall be tested at temperature and relative air humidity (23 ± 2) °C, (50 ± 5) % RH during the emission test (ISO 554).

For products with applications under other climatic conditions, alternative temperature and air humidity conditions may be used, preferably as specified in ISO 554.

8.2 Temperature conditions in second step test

After completely replacing the air in the micro-chamber with inert gas ventilation under room temperature conditions, the micro-chamber temperature shall be increased from room temperature to between 200 °C and 220 °C, and then kept at 200 °C to 220 °C for around 40 min.

The test specimen should be removed from the micro-chamber before this part of the test. It is necessary to take the maximum heating temperature in the second step test into account according to both the physicochemical properties of the target SVOC and recovery rate.

8.3 Supply air quality and background concentration

Supply air shall not contain any SVOCs at levels greater than the micro-chamber background requirements.

Background concentrations shall be low enough not to interfere with the emission determinations beyond quality assurance limits.

The background concentration of any single target SVOC shall be lower than 50 ng/m^3.

The water used for humidification shall not contain SVOCs at levels which could interfere with the analysis or compromise background concentrations.

8.4 Area specific air flow rate

Tests on different types of micro-chamber (see Annexes B and C) have shown that area specific air flow rate is not critical. However, this parameter should not be set below 0,15 m/h in order to maintain relevance to real world situations.

9 Verification of test conditions

9.1 General

All control measures shall be traceable to a certified standard according to the quality assurance and quality control schemes (see Annex A).

9.2 Temperature and relative humidity control systems

Control of temperature can be made by placing the micro-chamber within a location controlled to the required temperature.

Control of relative air humidity and temperature can be made by various systems with, for example, built-in humidity control of the supply air.

9.3 Test conditions in the micro-chamber

Temperature, relative humidity, and air flow rate shall be measured with instruments meeting the following accuracy:

— temperature	±0,5 °C
— relative air humidity	±5 %
— air flow rate	±3 %

9.4 Air flow rate in the micro-chamber for the first step test

The air flow rate shall be checked and readjusted prior to air sampling using a calibrated gas flow meter. The air flow rates shall not vary by more than ±3 % of the set value. The air velocity in the micro-chamber shall be constant.

CAUTION — If the test is carried out with a gas volume meter or flow meter which is not permanently installed, be aware that the back pressure introduced by the meter can lower the flow rate through the micro-chamber.

9.5 Micro-chamber airtightness

The micro-chamber airtightness shall be checked at the beginning of an emission test, by comparison of air flow rates at the inlet and the outlet ports. The flows at the two locations should not differ by more than 5 %.

10 Test specimens

Studies of the emission of SVOCs from building products placed in micro-chambers show that the product requires proper handling prior to testing.

After completing preparations for the emission test, the sample should be removed from the packaging used in transportation and the test specimen should be prepared and mounted in the micro-chamber. The start of the test (time, $t = 0$) should be the point when the test specimen is mounted in the micro-chamber. The edge and back of the test specimen should be sealed (6.6) with aluminum foil or similar materials (see References [8] and [11]).

Sample the product to be tested, store the sample before testing, and prepare test specimens in accordance with the procedures specified in ISO 16000-11.

Treatment of the surface of the test specimen, e.g. wiping off, is likely to influence SVOC emission properties. Initial emission of SVOC can be strongly influenced by contaminants adsorbed on to the surface. Therefore, appropriate packing and storage of samples are particularly important considerations. The initial influence of surface-adsorbed contaminants can be investigated by repeating the emission test after an extended period.

11 Micro-chamber preparation

The micro-chamber shall be cleaned in order to fulfil the requirements of 8.3. Before starting a test, the micro-chamber should be dismantled and washed with distilled water and acetone. In order to volatilize any remaining chemical substances, heat the micro-chamber to 250 °C or above. After heating, the micro-chamber shall be cooled to the room air temperature.

12 Test method

12.1 Mass collected in control test (field blank)

Mass collected in the control test (field blank) with a clean micro-chamber shall be less than 10 % compared with the total mass of target SVOC. The confirmation of recovery shall be proven.

Analyte recovery from the micro-chamber shall be measured in accordance with 6.7.

12.2 Position of test specimen in the micro-chamber for the first step

There shall be no direct contact between the test specimen and the inner walls of the micro-chamber as shown in Figure 1 and Figure 2.

12.3 Emission test (first step test)

The concentration measurements shall be carried out at predefined sampling times, typically 24 h. Depending on the objective of the test, it can be appropriate to sample the air at additional times. Air-sampling duration for concentration measurements depends on the analytical methods to be used and they shall be documented.

Humidified clean air (50 % RH) shall be supplied during the air sampling in the emission test.

NOTE For specific purposes, the emission rate over a different period of time can be determined using the same procedure.

12.4 Desorption test (second step test)

After completing step 1 of the emission test, remove the test specimen from the micro-chamber, and heat the micro-chamber. Another conditioned sorbent tube shall be used for this part of the test. Inert gas is supplied and then heated to 200 °C or above.

The sampling and ventilation of the micro-chamber should be started simultaneously. The sampling time should be 40 min until desorption is complete.

13 Calculation of area specific emission rates and expression of results

Calculate the area specific emission rate of the test specimen using Equation (1):

$$q_{mA} = \left(\frac{m_1}{q_{V,\mathrm{c}}\, t}\, q_{VA} \right) + \frac{m_2}{A\, t} = \frac{m_1 + m_2}{A\, t} \tag{1}$$

where the variables are as defined in Clause 4.

14 Performance characteristics

Performance characteristics for the air sampling and analysis aspects of this test method, when used in conjunction with ISO 16000-6, are discussed in ISO 16000-6 and ISO 16017-1.

15 Test report

The test report shall contain at least the following information:

a) testing organization:
   1) name and address,
   2) person responsible for testing;

b) type of product:
   1) when applicable, trade name,
   2) sample selection process (sampling method, etc.),
   3) product history (date of manufacture, lot number, date of delivery to testing organization, date and time of removal from packaging, date and time of preparation of test specimen, etc.);

c) results of determination of area specific emission rate of target SVOC, expressed in micrograms per square metre hour;

d) data analysis, quoting the method used when calculating the designated specific emission rate from the measured total mass collected (mathematical model and formulae);

e) test conditions:
   1) micro-chamber conditions (temperature, relative humidity, air flow rate),
   2) area of test specimen, surface area ratio, and air flow rate,

   3) whether sealing process was used,
   4) information on sampling method for target SVOC (sorbent tube used, sampling mass, length of sampling period from introduction into micro-chamber and number of cycles of sampling, etc.);

f) measuring equipment, including information on devices and methods used (micro-chamber, sealing material, air purification device, temperature and humidity control device, flow meter, chamber heating device, sampling pump, analytical instrument, etc.);

g) quality control and quality assurance:
   1) mass collected in control test and field blank for target SVOC,
   2) recovery data for target SVOC,
   3) number of times measurements were performed,
   4) if multiple sampling was performed, individual analytical values for each case,
   5) accuracy of temperature, relative humidity, and air flow rate reports on quality assurance;

h) reference to this part of ISO 16000 (ISO 16000-25:2011);

i) all operating details not specified in this part of ISO 16000, or regarded as optional, together with details of any incidents which may have influenced the test result(s).

Annex A
(normative)

System for quality assurance and quality control (QA/QC)

A.1 General

Micro-chamber testing of organic emissions from indoor materials or products shall be conducted within the framework of a quality assurance project plan (QAPP). The QAPP shall contain a project description, data quality objectives and acceptance criteria, QA/QC approaches and activities, and QA/QC audits.

A.2 Project description

A brief description shall include which materials are to be tested; how the testing is to be conducted; and who is responsible for various project activities. The project experimental design shall contain the necessary information for this portion of the QAPP.

A.3 Data quality objectives and acceptance criteria

This section of the QAPP defines the precision, accuracy, and completeness desired for each parameter being measured.

A.4 QA/QC approaches and activities

The types of QA/QC activities that can be specified in the QAPP include establishment of a system of records or notebooks to ensure proper operation of equipment and recording of data (e.g. see Reference [14]):

a) sample log to record receipt, storage, and disposition of materials;

b) GC standards preparation log to document preparation of all organic compound substances;

c) calibration logs to contain environmental systems calibration data;

d) instrument maintenance logs to document maintenance and repairs of all equipment;

e) materials testing logs in which to record all pertinent information for each test, including sample details, sample ID number (identity number), and GC run ID number;

f) sorbent tube cleanup and desorption log detailing thermal cleanup and QC validation of sorbent cartridges;

g) separate electronic log to document location and content of electronically stored data;

h) manuals governing operation or all equipment used by the project.

QC activities are carried out by project staff in a routine, consistent manner to provide necessary feedback in operation of all measurement systems. Such activities can include:

- routine maintenance and calibration of systems;
- daily recording of GC calibration accuracy and precision (i.e. control charting);

- timely monitoring of percentage recovery of the internal standard that was added to all samples;
- collection and analysis of duplicate samples;
- QC checking of organic collection sorbent tubes;
- periodic analysis of audit gases supplied by an independent source.

A.5 QA/QC audits

Finally, the QA/QC programme shall include periodic audits by QA personnel to evaluate compliance with QAPP protocols.

Annex B
(informative)

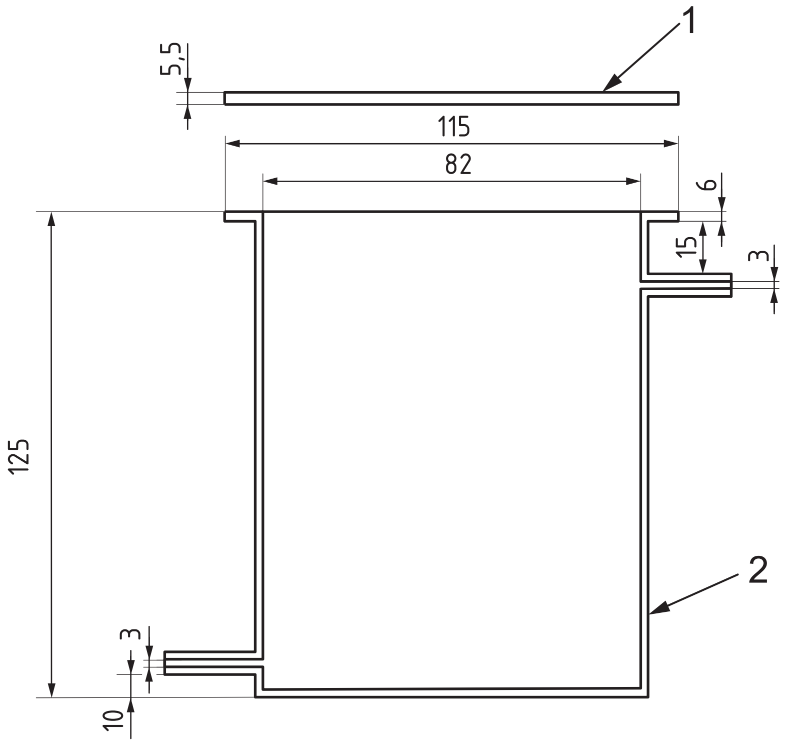
Examples of micro-chambers and procedure

B.1 Chamber construction and dimensions

Figure B.1 shows an example of a micro-chamber comprising a container and a cover. In this case, the parts of the micro-chamber which come into contact with the SVOC are made of glass and are surface treated to facilitate desorption. The volume of the chamber is 630 ml, with a relative tolerance of ±5 %. Dimensions are shown in Figure B.1.

The ratio of the inner size and the height of the micro-chamber may be changed according to the data given in B.4. Micro-chambers with these dimensions have been validated in comparative studies. Micro-chambers with other dimensions and other volumes can result in different measurement results.

Dimensions in millimetres



Key

1 cover

2 container

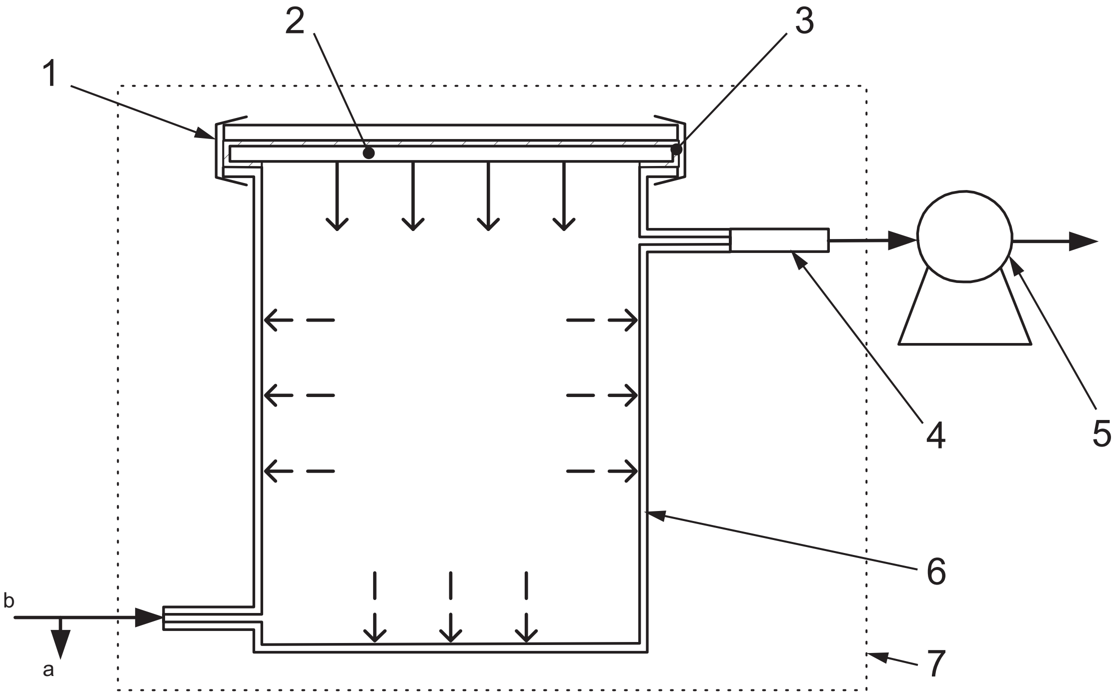
Figure B.1 — Dimensions of micro-chamber

B.2 Emission test (first step test)

Typical operating conditions are as follows:

- 24 h duration of first step;
- air flow rate 1,2 l/h (20 ml/min);
- pump flow rate 0,9 l/h (15 ml/min).

A typical test set-up is shown in Figure B.2.



Key

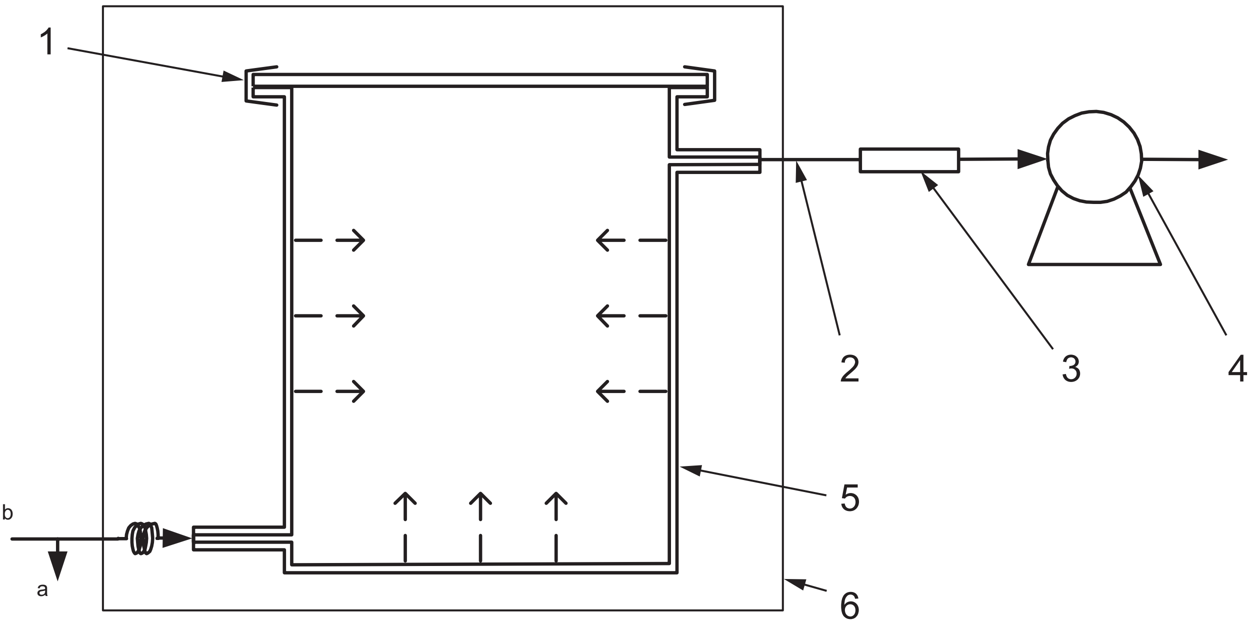
1 clamp
2 test specimen
3 sealing material
4 sorbent tube (sampling of air in chamber)
5 sampling pump
6 micro-chamber
7 thermostatic oven

[a] Vent line.
[b] Humidity-controlled clean air [(50 ± 5) % RH] inlet.

Figure B.2 — Emission test (first step test)

B.3 Desorption test (second step test)

The specimen is removed from the micro-chamber before the test. In this example, the micro-chamber is heated in an oven during the thermal desorption step. It is heated from ambient temperature to 200 °C to 220 °C at a rate between 10 °C/min and 20 °C/min and maintained at that level for 40 min. A typical inlet gas flow is 5,4 l/h (90 ml/min) and a typical pump flow rate is 3,6 l/h (60 ml/min). The process of thermal desorption of SVOC adsorbed on chamber inner walls is shown in Figure B.3.



Key

1 clamp

2 transfer line (holding temperature of 200 °C to 220 °C or higher is maintained at all times)

3 sorbent tube (sampling of SVOC thermally desorbed from chamber inner walls)

4 sampling pump

5 micro-chamber

6 chamber heating device (200 °C to 220 °C)

[a] Vent line.

[b] Inert gas (He or N_2) inlet.

Figure B.3 — Desorption test (second step test)

B.4 Verification of the area specific emission rate by the difference in an examination micro-chamber

The purpose of this clause is to demonstrate the determination of area specific emission rate for a sample with different shapes of micro-chamber.

Table B.1 shows specifications for the two types of micro-chamber used for the verification test. PVC wallpaper was the SVOC emission source. The area specific emission rates determined in each micro-chamber are shown in Table B.2.

Table B.1 — Specifications for two types of micro-chamber

Examination conditions	Micro-chamber type A	Micro-chamber type B
Inner size of a micro-chamber, mm	∅ 82 × 120	∅ 123 × 53
Effective capacity of a micro-chamber, ml	630(633)	630(629)
Area of the glass in a micro-chamber (side face), m^2	0,030 9	0,020 5
Area of the glass in a micro-chamber (bottom face), m^2	0,005 3	0,011 9
Total area of the glass in a micro-chamber, m^2	0,041 5	0,044 3
Surface area of test specimen, A, m^2	0,005 3	0,005 9
Effective absorption glass area of a micro-chamber, $A_{\mathrm{g,eff}}$, m^2	0,036 2	0,038 4
Air flow rate for micro-chamber, $q_{V,\mathrm{c}}$, m^3/h	0,000 9	0,000 9
Surface area ratio: $A/A_{\mathrm{g,eff}}$	0,15	0,15
Area specific air flow rate: $q_{V,\mathrm{c}}/A$	0,17	0,15

Table B.2 — Area specific emission rates determined in two types of micro-chamber

Result	Micro-chamber type A	Micro-chamber type B
First step sampling		
Mass of DEHP, µg	<0,01	<0,01
Area specific emission rate of DEHP, $\mu g/m^2 \cdot h$	<0,1	<0,1
Second step sampling		
Mass of DEHP, µg	0,64	0,57
Area specific emission rate of DEHP, $\mu g/m^2 \cdot h$	5,0	4,0

Annex C
(informative)

Example of a method of micro-chamber recovery measurement

C.1 Injection of standard solution

This annex gives an example of a method of micro-chamber recovery measurement (see References [8][11][12][13]).

Remove the outlet line of a micro-chamber before placing in the heating oven. Inject an SVOC standard solution of volume 1 µl to 5 µl, containing 100 ng to 500 ng of:

D6 (dodecamethyl cyclohexasiloxane);

BHT (butylated hydroxytoluene);

TCEP [tris(2-chloroethyl) phosphate)];

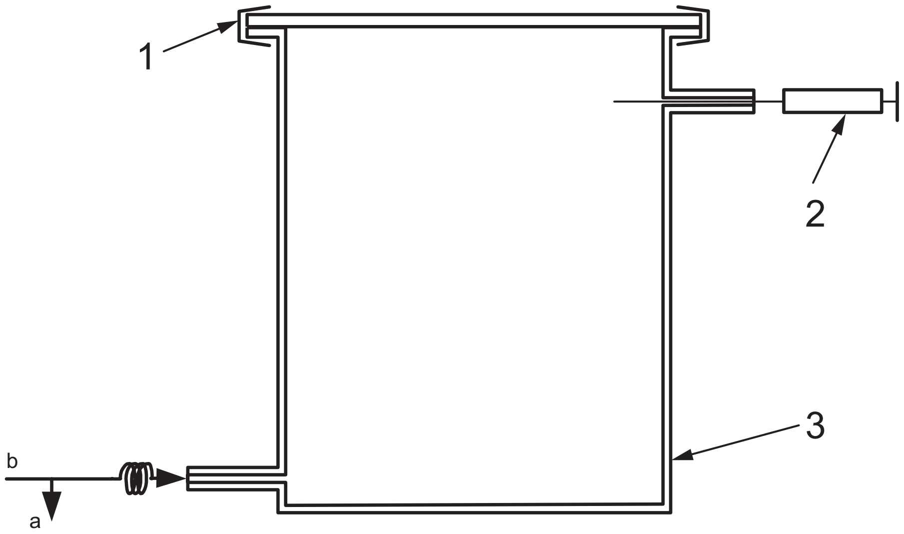
DBP (dibutyl phthalate);

DOA [di(2-ethylhexyl) adipate];

DEHP [di(2-ethylhexyl) phthalate]; and

DINP (diisononyl phthalate)

in acetone, into the micro-chamber by using a microsyringe. To prevent air leakage into the chamber during this procedure, introduce He or N_2 gas into the micro-chamber at slightly higher than ambient pressure (Figure C.1).



Key

1 clamp

2 microsyringe

3 micro-chamber

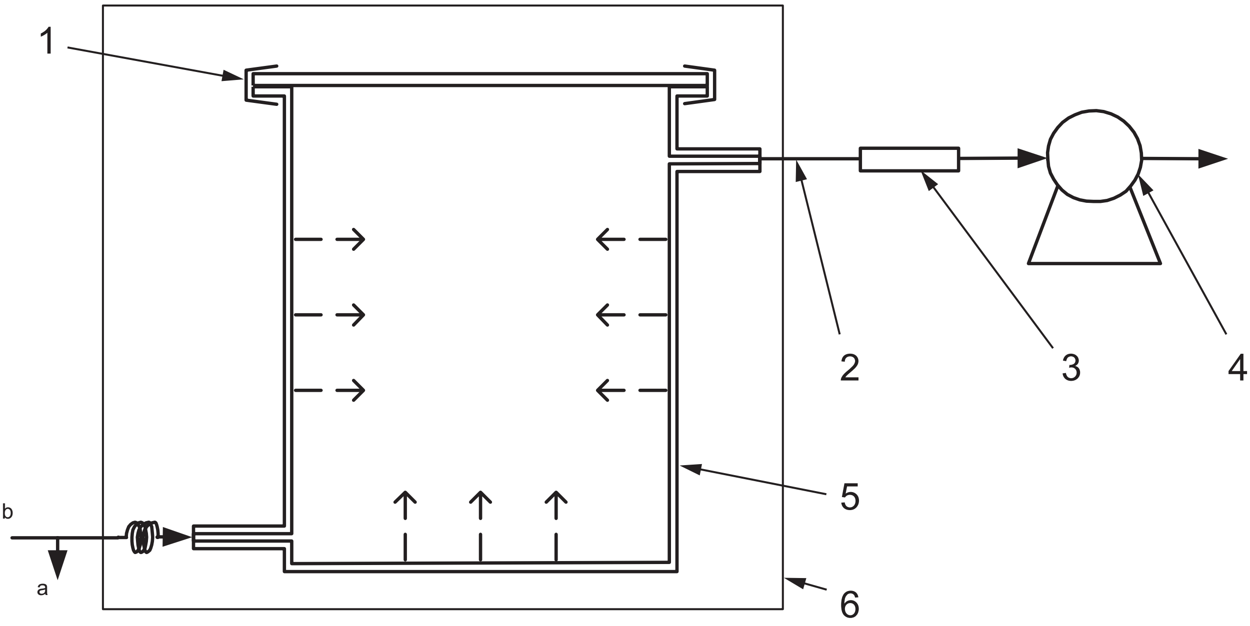
[a] Vent line.

[b] Inert gas (He or N_2) inlet.

Figure C.1 — Recovery measurement in a micro-chamber, step 1

C.2 Heating and sampling

Start heating and sampling simultaneously. Increase the temperature of the chamber heating device from room temperature to 200 °C to 220 °C at a rate of 10 °C/min to 20 °C/min and maintain at that level for 40 min. After sampling, determine the compounds sorbed on to the Tenax TA®[1] packing in the adsorbent tube by TD-GC/MS (Figure C.2).



Key

1 clamp
2 transfer line
3 sorbent tube
4 sampling pump
5 micro-chamber
6 chamber heating device (200 °C to 220 °C)

a Vent line.
b Inert gas (He or N_2) inlet.

Figure C.2 — Recovery measurement in a micro-chamber, step 2

C.3 Confirmation of recovery

Directly inject a similar volume of the SVOC standard solution as added into the micro-chamber (C.1) into a sorbent tube packed with Tenax TA®. Determine the components adsorbed on to the packing by TD-GC/MS. Take the value detected for each compound as 100 %. The ratio of the result obtained from the standard addition into the micro-chamber (C.1 and C.2) to that from the direct injection is treated as the recovery.

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1) Tenax TA® is the trade name of a product manufactured 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.

Annex D
(informative)

Selection of test specimens

D.1 Selection of test specimens from sheet-, panel-, and board-type products

Select the centre part of sheet, panel, and board products as the cutting position for test specimens.

D.2 Selection of test specimens from roll-type products

Remove the packaging from the roll and select an appropriate part of the sample centre. Select the specimen so as to include a large mass of colours making up the pattern.

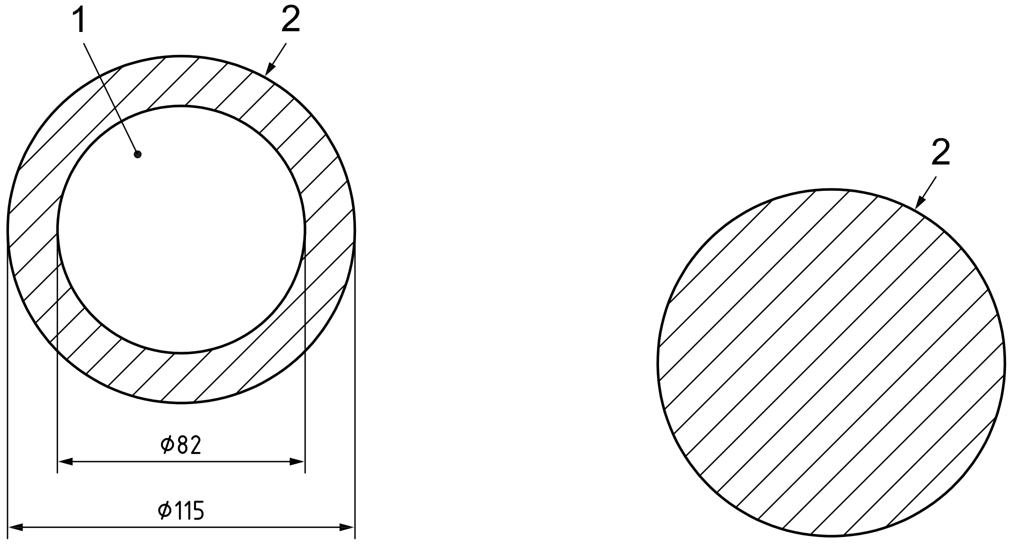
D.3 Selection of test specimens from adhesive- and paint-type products

For building products subject to evaporation in use (liquid adhesives and paints), prepare the test specimen by coating a glass plate, aluminium plate or similar, with the product.

D.4 Sealing of test specimens

Seal the edge and back of the test specimen with aluminium foil or similar. Examples of the sealing of test specimens are shown in Figures D.1 and D.2.

Dimensions in millimetres



a) Front face of test specimen **b) Rear face of test specimen**

Key

1 measured surface

2 aluminium foil

Figure D.1 — Principle of sealing the test specimen

a) Wallpaper b) Floor covering

Figure D.2 — Actual examples of sealed test specimens

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