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**Interior air of road vehicles —**

Part 3:

**Screening method for the determination  
of the emissions of volatile organic  
compounds from vehicle interior parts and  
materials — Micro-scale chamber method**

*Air intérieur des véhicules routiers —*

*Partie 3: Méthode de criblage pour la détermination des émissions de  
composés organiques volatils des parties et matériaux intérieurs des  
véhicules — Méthode de la micro-chambre*





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<b>Contents</b>	<b>Page</b>
<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>2</b>
<b>4 Principle</b> .....	<b>2</b>
<b>5 Apparatus</b> .....	<b>2</b>
<b>6 Test conditions</b> .....	<b>3</b>
<b>6.1 Temperature</b> .....	<b>4</b>
<b>6.2 Air or gas flow rate through the micro-scale chamber</b> .....	<b>4</b>
<b>6.3 Quality of supplied air or gas and background concentration of organic vapours</b> .....	<b>4</b>
<b>6.4 Control measures</b> .....	<b>4</b>
<b>7 Test specimens</b> .....	<b>4</b>
<b>7.1 General</b> .....	<b>4</b>
<b>7.2 Preparation of the test specimen</b> .....	<b>5</b>
<b>8 Cleaning micro-scale chamber components</b> .....	<b>5</b>
<b>9 Test method</b> .....	<b>5</b>
<b>9.1 Sampling media</b> .....	<b>5</b>
<b>9.2 Measuring background concentrations</b> .....	<b>6</b>
<b>9.3 Vapour sampling</b> .....	<b>6</b>
<b>9.4 Sealing the vapour sampling devices after gas sample collection</b> .....	<b>6</b>
<b>9.5 Analysis of vapour sampling devices</b> .....	<b>6</b>
<b>9.6 Storage of the test specimen between emissions tests (if required)</b> .....	<b>6</b>
<b>9.7 Cleaning the micro-scale chamber after use</b> .....	<b>6</b>
<b>10 Calculation of vapour concentrations and specific emission rates</b> .....	<b>7</b>
<b>11 Test report</b> .....	<b>8</b>
<b>12 Quality assurance/quality control</b> .....	<b>9</b>
<b>Annex A (informative) Micro-scale chamber principles</b> .....	<b>10</b>
<b>Annex B (informative) Micro-scale chamber — Example 1</b> .....	<b>12</b>
<b>Annex C (informative) Micro-scale chamber — Example 2</b> .....	<b>14</b>
<b>Annex D (informative) Micro-scale chamber — Example 3</b> .....	<b>16</b>
<b>Annex E (informative) Assessment of VOC recoveries</b> .....	<b>18</b>
<b>Annex F (informative) Calculation of specific emission rates from micro-scale chamber air concentration</b> .....	<b>19</b>
<b>Bibliography</b> .....	<b>21</b>

## 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 12219-3 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 6, *Indoor air*, in collaboration with Technical Committee ISO/TC 22, *Road vehicles*.

ISO 12219 consists of the following parts, under the general title *Interior air of road vehicles*:

- *Part 1: Whole vehicle test chamber — Specification and method for the determination of volatile organic compounds in cabin interiors*
- *Part 2: Screening method for the determination of the emissions of volatile organic compounds from vehicle interior parts and materials — Bag method*
- *Part 3: Screening method for the determination of the emissions of volatile organic compounds from vehicle interior parts and materials — Micro-scale chamber method*
- *Part 4: Screening method for the determination of the emissions of volatile organic compounds from vehicle interior parts and materials — Small chamber method*

The following part is under preparation:

- *Part 5: Screening method for the determination of the emissions of volatile organic compounds from vehicle interior parts and materials — Static chamber method*

## Introduction

Volatile organic compounds (VOCs) are widely used in industry and may be emitted by many everyday products and materials. They have attracted attention in recent years because of their impact on indoor air quality. After homes and workplaces, people spend a lot of time in their vehicles. It is important to determine the material emissions of interior parts and to reduce them to an acceptable level, if required. Therefore it is necessary to obtain comprehensive and reliable information about the types of organic compounds in the interior air of vehicles and also their concentrations.

Monitoring emissions from vehicle trim components can be performed in several ways and the approach selected depends upon the desired outcome and the material type. For example, to obtain emissions data from complete assemblies (e.g. a dashboard or seat), it is necessary to employ emissions chambers or bags that have sufficient volume to house the complete assembly (typically  $>1 \text{ m}^3$ ). Such tests may take several hours or even days to perform, depending on specified equilibration times and the requirements of the relevant test protocol.

This part of ISO 12219 outlines a method of measuring the types and levels of VOCs emitted using micro-scale chambers (References [2]–[4]). These allow qualitative and semiquantitative screening of product emissions after only minutes, rather than hours or days, of equilibration. Their capacity is limited so they are best suited to small assemblies or representative samples of homogeneous vehicle interior parts and materials. Multiple test specimens can also be readily evaluated from the same sample if required. Micro-scale chambers can provide an ideal quick screening tool for quality control of production and other in-house tests by manufacturers. They offer a complementary approach to large chamber or sampling bag approaches.

ISO 16000-3, ISO 16000-5,<sup>[5]</sup> ISO 16000-6, ISO 16000-9,<sup>[6]</sup> ISO 16000-10,<sup>[7]</sup> ISO 16000-11,<sup>[8]</sup> ISO 16000-24,<sup>[9]</sup> ISO 16000-25,<sup>[10]</sup> as well as ISO 16017-1<sup>[11]</sup> and ISO 16017-2,<sup>[12]</sup> also focus on VOC measurements.



# Interior air of road vehicles —

## Part 3: Screening method for the determination of the emissions of volatile organic compounds from vehicle interior parts and materials — Micro-scale chamber method

**WARNING** — It is the responsibility of the user of this part of ISO 12219 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. National regulations for precautions shall be followed.

### 1 Scope

This part of ISO 12219 specifies a fast, qualitative and semiquantitative screening method for vapour-phase organic compounds (volatile and some semi-volatile) released from vehicle trim materials under simulated real-use conditions using micro-scale test chambers. This method is intended for evaluating new car interior trim components but can, in principle, also be applied to used car components.

Target analytes include VOCs (conventionally defined as organic compounds in the volatility range of *n*-hexane to *n*-hexadecane) and volatile carbonyl compounds such as formaldehyde. The specified analytical procedure for VOCs is ISO 16000-6 and for formaldehyde and some other light carbonyl compounds is ISO 16000-3.

NOTE 1 Some compounds more volatile than *n*-hexane and less volatile than *n*-hexadecane can also be analysed (for more information, see: ISO 16000-6:2011, Annex D; ISO 16017-1;<sup>[1]</sup> and Annex E).

NOTE 2 For dry, homogeneous materials, results from tests of volatile organic emissions carried out using micro-scale chambers on newly manufactured products have been found to correlate well with data obtained using standard (reference) methods and conventional emission test chambers (ISO 12219-4, VDA 276<sup>[1]</sup> and ISO 16000-9<sup>[6]</sup>) or test cells (ISO 16000-10<sup>[7]</sup>). Correlation with emission data obtained using bags (ISO 12219-2) has also been reported. The practice specified in this part of ISO 12219 is therefore complementary to existing standards.

This part of ISO 12219 provides third party test laboratories and manufacturing industry with a cost-effective approach for:

- a) monitoring and screening VOC emissions as part of routine quality control;
- b) monitoring product uniformity or conformity between formal certification tests;
- c) comparing emissions from products within a range (e.g. different colours or patterns);
- d) evaluating prototype, “low-emission” materials or products during development.

NOTE 3 All volatile carbonyl compounds except formaldehyde can be analysed according to ISO 16000-6.

### 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 12219-1, *Interior air of road vehicles — Part 1: Whole vehicle test chamber — Specification and method for the determination of volatile organic compounds in cabin interiors*

ISO 16000-3:2011, *Indoor air — Part 3: Determination of formaldehyde and other carbonyl compounds — Active sampling method*

ISO 16000-6:2011, *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 or MS–FID*

### 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16000-6, ISO 12219-1 and the following apply.

#### 3.1

##### **vehicle trim component**

vehicle interior part or material

### 4 Principle

The principle of the test is to determine the area (or mass) specific emission rate of VOCs emitted from automotive products. The test is performed in a micro-scale test chamber at a constant temperature and flow rate. The surface area (or mass) of the sample in the micro-scale test chamber is constant and, by measuring the mass or vapour-phase concentrations of emitted compounds, the area (or mass) specific emission rates of VOCs from the product under test can be determined at a given time,  $t$  (see Clause 10).

The results can be used to assess product performance with respect to emission levels — either by comparison with control levels or by comparing the data with results from other products or batches of product.

### 5 Apparatus

**5.1 General.** General specifications and requirements, which apply to all types of micro-scale chambers, are given in 5.1 to 5.5. General micro-scale chamber principles are also summarized in Annex A and specific micro-scale chamber examples are given in Annexes B to D.

The following key micro-scale chamber components are required:

- micro-scale chamber apparatus;
- heating mechanism;
- clean gas supply and optional humidification system,

**NOTE** Most samples contain sufficient inherent humidity to facilitate formaldehyde screening, according to ISO 16000-3, over the short duration of a micro-scale chamber test. Therefore humidification is normally not required for this screening method.

- appropriate monitoring and control systems (to ensure that the test is carried out according to specified conditions);
- appropriate vapour sampling tubes.

**5.2 Micro-scale chamber apparatus construction materials.** Micro-scale test chambers range in size from 30 cm<sup>3</sup> to 1 l (e.g. 44,5 ml, see Annex B). They are designed to operate at ambient or elevated temperatures and to permit the testing of vapour-phase organic emissions from various types of vehicle interior trim components, construction products, and consumer goods.

The micro-scale chamber apparatus can comprise one or multiple sealable, micro-scale chambers constructed of inert, non-emitting and non-absorbing materials, such as surface-treated (polished) or inert coated stainless steel or deactivated glass or quartz. In all cases, the requirements specified in 5.4 and 5.5 shall be fulfilled.

Any sealing materials e.g. gaskets or O-rings used for sealing the doors or lids of micro-scale chambers, shall be low emitting and low absorbing and shall not contribute significantly to the background vapour concentration. The O-rings or gaskets shall be easily removed to facilitate cleaning or replacement. The micro-scale chambers shall be easily dismantled and removed from any housings to facilitate cleaning as specified in Clause 8.



**5.3 Heating.** The micro-scale chambers shall be capable of heating the test specimen to a uniform prescribed temperature for the duration of the test. The temperature shall be maintained within  $\pm 2$  °C throughout the entire procedure (see also 6.1).

To facilitate cleaning (see Clause 8) the micro-scale test chamber should be capable of being heated to 100 °C or more.

**5.4 Air or gas supply and mixing facilities.** The apparatus shall include a means of supplying pure (low hydrocarbon content), optionally humidified, air or gas to the micro-scale chamber(s) at a controlled flow rate ( $\pm 3$  %). The supply air or gas shall not contain any VOCs at levels greater than the micro-scale chamber background requirements (6.3). Similarly, if the air or gas supply is humidified then the water used for humidification shall not contain interfering VOCs.

The positioning of the air or gas inlet and outlet, the volume of the micro-scale chamber, and the gas flow rate should ensure thorough mixing with no volumes of still air or gas within the micro-scale chamber. The air or gas inlet and outlet are usually positioned at right angles to the sample surface to optimize turbulence. Air or gas flow rates between 20 ml/min and 500 ml/min are typical for the types of micro-scale chamber described in Annexes B and C.

NOTE 1 Air is most commonly used, but pure inert gases such as nitrogen or helium are preferred for some applications.

NOTE 2 Turbulence and mixing within the micro-scale chamber examples given in Annexes B and C have been optimized by minimizing the air volume above the sample surface to 3,2 ml and 7,4 ml, respectively, and by orienting the inlet and outlet such that air enters and leaves the micro-scale chamber at right angles to the sample surface. In this configuration, air flow rates in excess of 10 ml/min and 20 ml/min, respectively, are sufficient to ensure turbulence and mixing as well as eliminating the risk of still air volumes. Note that inadequate turbulence and mixing, if it does occur, is identified by inadequate analyte recovery (see Annex E.)

**5.5 Air or gas leaks.** The micro-scale chamber is considered sufficiently leak-free if the inlet carrier air or gas flow differs from the total outlet air or gas flow by less than 5 %. This should be checked at the start of every recovery test (Annex E), background test (9.2), and emissions test (9.3).

**5.6 Air sampling.** Vapours shall be sampled from the micro-scale chamber exhaust by connecting a sample tube [conditioned sorbent tube for volatile or semi-volatile organics (see ISO 16000-6) or DNPH cartridge or equivalent for formaldehyde and other volatile carbonyl compounds] to the outlet coupling of the micro-scale chamber. Micro-scale chambers are typically closed systems in which all of the air or gas entering passes out into the vapour sampling tube.

Tenax TA<sup>®1</sup>) is the most commonly used sorbent for VOCs ranging in volatility from *n*-hexane to *n*-hexadecane. Other sorbents or sorbent combinations are available to extend this volatility range if required: see ISO 16000-6:2011, Annex D, ISO 16017-1<sup>[11]</sup> and Annex F for more information.

NOTE 1 Refer to ISO 16000-6:2011, Annex D or ISO 16017-1<sup>[11]</sup> for guidance on sorbent selection if VOCs eluting before *n*-C<sub>6</sub> need to be analysed.

NOTE 2 Micro-scale chambers typically operate slightly (<20 %) above atmospheric pressure and incorporate mechanisms for controlling and maintaining gas flow at a constant rate, whether or not a sample tube is attached. Another benefit of this approach is that, provided all of the exhaust air flow is sampled, it means that a constant air flow can be maintained through the vapour sampling tubes without using pumps (see Annexes B to D.) This simplifies operation for routine industrial quality control checks.

## 6 Test conditions

In general, the following test conditions shall be achieved.

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1) Tenax TA<sup>®</sup> is the trade name of a product supplied by Buchem. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

## 6.1 Temperature

Emission rates are specific to a particular temperature, therefore it is essential to maintain a constant temperature of  $65\text{ °C} \pm 2\text{ °C}$  within the micro-scale chamber throughout the emission test.

Other temperatures may be used depending upon the objectives of the test and the agreement of all parties.

## 6.2 Air or gas flow rate through the micro-scale chamber

Maintain a constant air or gas flow rate through each individual micro-scale chamber throughout the emission test. Typical flow rates are of the order of 50 ml/min for screening surface emissions of VOCs. Higher gas flow rates (e.g. 100 ml/min to 200 ml/min) are normally recommended for testing VOC emissions from bulk materials where the sample is at the bottom of the micro-scale chamber and a larger micro-scale chamber volume is exposed. Higher flow rates are also recommended to minimize risk of sink effects when testing higher boiling semi-volatile organic compounds (SVOCs).

A flow rate of 250 ml/min is recommended for screening surface emissions of formaldehyde according to ISO16000-3.

NOTE Lower flow rates or shorter sampling times can be used for screening formaldehyde using micro-scale chambers; however, ISO 16000-3 detection limits can be compromised as a result.

Analyte recovery tests, such as that described in Annex E, shall be carried out regularly (e.g. once per month) and used as a check that air turbulence and mixing is adequate, and that there are no significant volumes of still air. Satisfactory recovery is demonstrated by  $>80\%$  recovery on the first sample tube and  $<20\%$  recovery on the second sample tube.

Record the air or gas flow rate and the results of the most recent analyte recovery test.

## 6.3 Quality of supplied air or gas and background concentration of organic vapours

Background levels of target compounds (including micro-scale chamber artefacts and contaminants in the supplied air or gas) shall be shown to be below 10 % of measured micro-scale chamber concentrations or below  $5\text{ }\mu\text{g}/\text{m}^3$  for individual VOC and below  $50\text{ }\mu\text{g}/\text{m}^3$  for total volatile organic compound (TVOC), whichever is higher. Similarly, if humidification is required, any water used shall not contain levels of organic compounds which could interfere with the results.

## 6.4 Control measures

Systems for measuring temperature and flow shall be independent of the means of controlling said conditions.

# 7 Test specimens

## 7.1 General

This part of ISO 12219 can be applied horizontally, i.e. to a wide range of car trim component materials. Studies of the emission of vapour-phase organic compounds from vehicle components in micro-scale chambers require proper handling of the test specimen prior to and during the testing period.

Samples that are taken straight from production to be analysed rapidly in an on-site laboratory should be placed in suitable clean, airtight, and non-outgassing (non-emitting) containers or packaging. Every sample shall be treated the same way in terms of type of storage container or packaging, method of test specimen preparation, and period between sample collection and analysis

If samples are to be stored for longer than 2 h before analysis or if they need to be transported to an off-site laboratory, more precautions are required with respect to sampling procedures, transport conditions, sample storage, test specimen preparation, etc. In this case, advice given in ISO 16000-11<sup>[8]</sup> should be followed.

NOTE For heterogeneous materials, it can be necessary to make measurements on multiple test specimens from the same sample to determine the mean specific emission rate.

## 7.2 Preparation of the test specimen

Test specimens often need to be cut (sectioned) to fit snugly within the micro-scale emissions chamber, thus minimizing or eliminating edge effects — see Annexes A to C. This is best performed using a punch to minimize heat generation. Identify and weigh each test specimen.

NOTE Sawing can heat the sample, which can compromise emissions testing.

For analysis of bulk emissions, sample mass shall be sufficient to determine the mass specific emission rate with enough sensitivity to meet test objectives.

The period of time between unpacking the sample and preparation of the test specimen shall be as short as possible, and shall be the same in each case. After preparation of the test specimen, it shall immediately be placed into the micro-scale chamber. This time shall be regarded as the start time of the emission test, i.e.  $t = t_0$ .

If it is appropriate to measure emissions from the bulk material (e.g. polymer resin pellets, adhesives or insulation fibres) representative samples can be placed directly into the micro-scale chamber with no additional preparation steps. If, in real use, only one surface of a material or product is exposed, care should be taken to prevent emissions from other surfaces and cut edges interfering with the test. The design of the micro-scale chamber can facilitate this by accommodating snug-fitting samples or by use of a collar or baffle that presses down on the surface of rigid planar materials, near the edge. This prevents ingress of emissions from cut edges and the rear surface of the sample for the short duration of the test (see Annexes B and C). Alternatively, the edges and rear surfaces of a test specimen shall be sealed with low-emitting aluminium adhesive tape or by using a suitable sample holder before the sample is placed in the micro-scale chamber.

## 8 Cleaning micro-scale chamber components

The blank air sample collected from an empty micro-scale chamber shall meet the requirements of 6.3. When the blank value cannot be achieved, the micro-scale chamber shall be cleaned. Examples of cleaning procedures are described in the following.

Micro-scale chambers can be cleaned by removing any O-rings or gaskets and by washing the micro-scale chamber components using alkaline detergent followed by two separate rinsings with clean water or by using an appropriate solvent. Dry thoroughly.

Alternatively, if the micro-scale chamber assembly can be heated, raise the temperature of the empty, sealed micro-scale chambers to 100 °C or more in a fast flow of pure gas until background artefacts are reduced to acceptable levels (see 6.3).

If the micro-scale chamber has an inert coating, care shall be taken not to damage the coating during cleaning (e.g. by using abrasive cleaners or cleaners with a high or low pH).

## 9 Test method

### 9.1 Sampling media

Select the correct sampling media — see 5.6. Sorbent tubes should be stringently conditioned before use (see ISO 16000-6).

## 9.2 Measuring background concentrations

Micro-scale chamber background levels shall be checked regularly, e.g. before the start of each batch of emission tests, to quantify any background contribution of vapour-phase organic compounds from the air or gas supply or from the micro-scale chamber apparatus itself.

The background check shall be carried out using representative conditioned sample tubes and by following the procedure specified in 9.3, but without a sample present.

Background concentrations shall meet the requirements of 6.3.

## 9.3 Vapour sampling

The test specimen shall be placed in the pre-heated micro-scale chamber (65 °C) and left to equilibrate for 20 min. Recommended flow rates for surface emissions testing using the types of micro-scale chamber illustrated in Annexes B to D are 50 ml/min (VOCs) or 250 ml/min (formaldehyde).

NOTE 1 Longer equilibration times can be necessary in order to measure semi-volatile components or when testing particularly dense materials.

After the equilibration period, a conditioned vapour sampling device shall be connected to the outlet port of the micro-scale chamber. This marks the beginning of vapour sample collection. Quickly check for leaks (see 5.5). Collect vapour for 15 min ± 1 min (VOC sampling) or for 2 h ± 0,2 h to 4 h ± 0,2 h (formaldehyde sampling).

NOTE 2 Most micro-scale chamber apparatus operates at slightly elevated pressure and pumps are not usually required to pull micro-scale chamber air through the vapour sampling tube (see 5.6).

NOTE 3 Lower flow rates or shorter sampling times can be used for screening formaldehyde using micro-scale chambers, but ISO 16000-3 detection limits can be compromised as a result (see 6.3).

NOTE 4 Lower flow rates or shorter sampling times can also be used to minimize risk of breakthrough for sampling the most volatile VOCs (see Clause 12), but in this case it is often preferable to use alternative, stronger, sorbents — see ISO 16000-6:2011, Annex D. For high VOC emissions, it is preferable to increase the split flow on the analytical system rather than reduce the sampling time or flow rate.

NOTE 5 Vapours exhausted from micro-scale chambers should be ducted into a fume hood when not being sampled to ensure that any chemicals emitted from the test specimen are isolated from the laboratory environment.

## 9.4 Sealing the vapour sampling devices after gas sample collection

Sample tubes shall be disconnected from the micro-scale chamber at the end of vapour sampling, resealed using appropriate caps and fittings (ISO 16000-6 and ISO 16000-3) and the duration of sampling immediately recorded. Sample tubes shall be stored carefully before analysis following guidance given in ISO 16000-6 or ISO 16000-3.

## 9.5 Analysis of vapour sampling devices

Analyse the vapour sampling devices according to ISO 16000-3 (formaldehyde) or 16000-6 (VOCs) in order to determine the mass of each target compound retained by the vapour sampling tubes. Analyte vapour concentrations can be determined from the respective masses if required — see ISO 16000-3:2011, Clause 10, ISO 16000-6:2011, Clause 11, and Annex F.

## 9.6 Storage of the test specimen between emissions tests (if required)

If the same sample is to be subjected to repeated tests using the micro-scale chamber, the test specimens shall be kept within the micro-scale chamber under pure air or gas flow.

## 9.7 Cleaning the micro-scale chamber after use

At the end of an emission test, the micro-scale chamber should be cleaned if necessary, according to Clause 8, to ensure that it meets background requirements for subsequent tests (see 6.3).

## 10 Calculation of vapour concentrations and specific emission rates

To accommodate differences in micro-scale chamber design, the results are usually expressed in area specific emission rates,  $q_{mA}$ , in micrograms per square metre hour, or as mass specific emission rates,  $q_{mm}$ , in micrograms per gram hour.

Most micro-scale chambers operate with all of the air flow directed through the vapour sampling tube. If this is the case and if blank levels meet the performance criteria specified in this part of ISO 12219, then the mass of individual vapours or total VOCs determined during analysis (see 9.5) can be used to calculate the area specific emission rate,  $q_{mA}$ , as well as the mass specific emission rate,  $q_{mm}$ , directly:

The area specific emission rate,  $q_{mA}$ , is given by:

$$q_{mA} = \frac{m_a}{At} \quad (1)$$

The mass specific emission rate,  $q_{mm}$ , is given by:

$$q_{mm} = \frac{m_a}{m_s t} \quad (2)$$

where

- $m_a$  is the mass, in micrograms, of the analyte;
- $A$  is the area, in square metres, of the sample;
- $m_s$  is the mass, in grams, of the sample;
- $t$  is the duration, in hours, of vapour sampling.

For this part of ISO 12219, the duration of sampling is 15 min (0,25 h) for VOCs or up to 4 h for formaldehyde.

### EXAMPLES

For the micro-scale chamber example shown in Annex B, with an exposed sample area of  $1,28 \times 10^{-3} \text{ m}^2$ , calculation of  $q_{mA}$  for VOCs using 15 min vapour sampling can be reduced to:

$$q_{mA} = \frac{m_a \text{ } \mu\text{g}}{3,2 \times 10^{-4} \text{ } \mu\text{g} \cdot \text{m}^{-2} \cdot \text{h}^{-1}}$$

For the micro-scale chamber examples shown in Annex C, with an exposed sample area of  $2,46 \times 10^{-3} \text{ m}^2$ , calculation of  $q_{mA}$  for VOCs using 15 min vapour sampling can be reduced to:

$$q_{mA} = \frac{m_a \text{ } \mu\text{g}}{6,15 \times 10^{-4} \text{ } \mu\text{g} \cdot \text{m}^{-2} \cdot \text{h}^{-1}}$$

When using the micro-scale chamber example shown in Annex B for measuring formaldehyde and other volatile carbonyl compounds with 4 h vapour sampling, calculation of  $q_{mA}$  can be reduced to:

$$q_{mA} = \frac{m_a \text{ } \mu\text{g}}{5,12 \times 10^{-3} \text{ } \mu\text{g} \cdot \text{m}^{-2} \cdot \text{h}^{-1}}$$

When using the micro-scale chamber examples shown in Annex C for measuring formaldehyde and other volatile carbonyl compounds with 4 h vapour sampling, calculation of  $q_{mA}$  can be reduced to:

$$q_{mA} = \frac{m_a \text{ } \mu\text{g}}{9,84 \times 10^{-3} \text{ } \mu\text{g} \cdot \text{m}^{-2} \cdot \text{h}^{-1}}$$

where  $m_a$  is the measured mass of the analyte.

For further information or in cases where only part of the air exhausting from the micro-scale chamber is sampled,  $q_{mA}$  (or  $q_{mm}$ ) shall be determined from the vapour-phase concentration of target analytes within the micro-scale chamber, see Annex G or ISO 16000-3, ISO 16000-6, ISO 16000-9<sup>[6]</sup> and ISO 16000-10.<sup>[7]</sup>

## 11 Test report

The following is a guide to the details required in a test report if the micro-scale chamber results are to be used for evaluation of emissions in absolute terms. A simpler report is acceptable if this test is carried out for routine, in-house quality control purposes:

- a) test laboratory:
  - laboratory details (if appropriate),
  - name of the responsible person,
  - reference to this practice,
  - reference to the appropriate in-house protocol or detailed description of the equipment and methods used;
- b) sample or test specimen description:
  - type and batch number of product (and brand name if appropriate),
  - sample selection process (e.g. random),
  - product history, e.g. date of production or date of arrival at the test laboratory, if appropriate,
  - description of packaging, if appropriate;
- c) test specimen preparation:
  - date and time of unpacking (if appropriate),
  - method of preparation, including thickness and substrate;
- d) experimental conditions and procedures:
  - micro-scale chamber conditions (temperature, air or gas flow, humidity) (if appropriate),
  - test specimen area or mass and means of application (for liquid products, e.g. paint, describe sample substrate and coating procedure, paint thickness and coating density),
  - describe how the test specimen was presented within the micro-scale chamber — e.g. for surface only emission testing or for emission testing from whole components or bulk materials,
  - sampling of emitted compounds (sorbent used, volume sampled, sampling duration and times after introduction into the micro-scale chamber),
  - analytical conditions used [i.e. thermal desorption parameters, GC column selected, GC-MS (or GC-MS-FID) conditions, etc.];
- e) data analysis: describe the method used to calculate the specific emission rate or vapour concentration data;
- f) results: specific emission rates shall be reported for each test specimen, for individual organic vapours and/or TVOC (if required), at the times of gas sampling;
- g) quality assurance/quality control:
  - background micro-scale chamber concentrations of target compounds,
  - recovery data (see Annex E or ISO 16000-6).
  - recovery data for the three compounds that are representative of the range of target organic vapours,

- results of duplicate sampling or analysis,
- quality of the environmental variables (temperature, air or gas selected, air or gas flow, air or gas change rate, humidity) (if appropriate).

## 12 Quality assurance/quality control

If the micro-scale chamber tests are to be used for quick screening of product emissions as part of routine quality control or for in-house intercomparison of products by the manufacturer, the performance guidelines given in this part of ISO 12219 are sufficient.

However, if the micro-scale chamber tests are to be used in absolute terms — i.e. for estimating the contribution of a product to atmospheric concentrations of organic vapours in car cabin air — an appropriate level of quality control shall be employed following ISO 16000-3, ISO 16000-6, and ISO 16000-9<sup>[6]</sup> including:

- field blanks are prepared according to Clause 8;
- the field blank level is acceptable if artefact peaks are no greater than 10 % of the typical areas of the analytes of interest;
- the desorption efficiency of VOCs and carbonyl compounds shall be checked according to ISO 16000-3 and ISO 16000-6 — a recovery of at least 95 % shall be demonstrated for  $n$ -C<sub>6</sub> to  $n$ -C<sub>16</sub> hydrocarbons (ISO 16000-6);
- the collection efficiency can be controlled by always taking sample volumes below the safe sampling volume, see ISO 16000-6 — breakthrough can be checked using back-up tubes or by collecting samples at a range of different sampling volumes below the safe sampling volume;
- repeatability of the air sampling and analytical method shall be determined — a coefficient of variation  $\leq 15$  % (ISO 16000-3 and ISO 16000-6) from the duplicate measurements should be reached;

NOTE The repeatability of the emission test is influenced by any inhomogeneity of the material under test.

- documentation illustrating traceable calibrations for temperature, humidity, and flow measurements.

## **Annex A** (informative)

### **Micro-scale chamber principles**

#### **A.1 General**

Micro-scale chambers operate under the same fundamental principles as conventional small environmental test chambers and can be used for surface-only emissions testing or bulk emissions testing. However, their small size and ability to operate at elevated temperatures allows them to equilibrate more quickly. This facilitates the generation of fast, repeatable emissions-screening data that can be correlated with longer-term reference tests and makes them a useful quality control tool for manufacturing industry.

The guidelines in A.2 to A.5 should be considered.

#### **A.2 Size and shape**

Micro-chambers typically have a volume of 40 ml to 120 ml and are constructed with a flat base and circular perimeter to facilitate the simple preparation of test specimens which fit snugly against the inner diameter of the micro-scale chamber such that interference from edge and rear surface emissions is minimized.

#### **A.3 Materials**

Construction materials should be robust (suitable for use in a factory environment), inert, non-outgassing, non-absorbent and tolerant of temperatures above 100 °C. Suitable materials include stainless steel or inert coated stainless steel and surface-treated glass or quartz. Seals should be low emitting and tolerant of high temperatures.

#### **A.4 Functions and features**

Micro-scale chambers should offer the capability to readily analyse surface-specific emissions from planar materials or bulk material emissions from moulded or particulate products.

An air inlet and an air outlet are required and should be orientated to facilitate maximum air mixing and eliminate risk of still air volumes and sink effects.

Connection to the vapour sampling tube should normally allow the entire flow of air exhausted from the micro-scale chamber to be sampled. Alternatively, steps should be taken to ensure that the vapour sample collected is representative of the air within the micro-scale chamber.

If the micro-scale chamber can function at uniform inlet and sampled air flow rates, whether or not a vapour sampling tube is attached, but without requiring an air sampling pump, this simplifies operation and is an advantage for use by manufacturing industry.

It should be possible to heat the micro-scale chamber uniformly to the temperatures required by this part of ISO 12219.

It should be possible to readily dismantle the micro-scale chamber apparatus for cleaning.

#### **A.5 Performance criteria**

The area specific emission rate data generated by the micro-scale chamber under the conditions specified in this part of ISO 12219 is indicative of that obtained from the same sample using conventional small chambers.

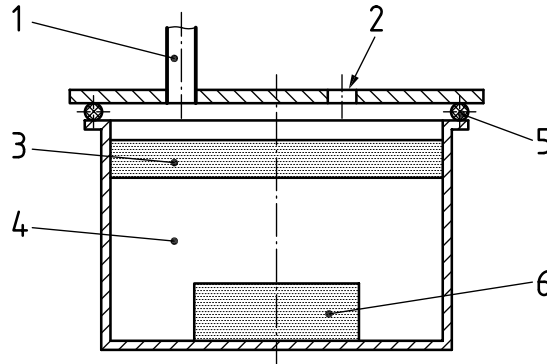


Micro-scale chamber emission profiles should be repeatable.

Micro-scale chambers should exhibit negligible background and significant sink effects as defined in the normative text of this part of ISO 12219.

It shall be possible to maintain a uniform air flow rate through the micro-scale chamber, with or without a vapour-sampling tube attached.

It shall be possible to maintain a constant micro-scale chamber temperature throughout the test.



**Key**

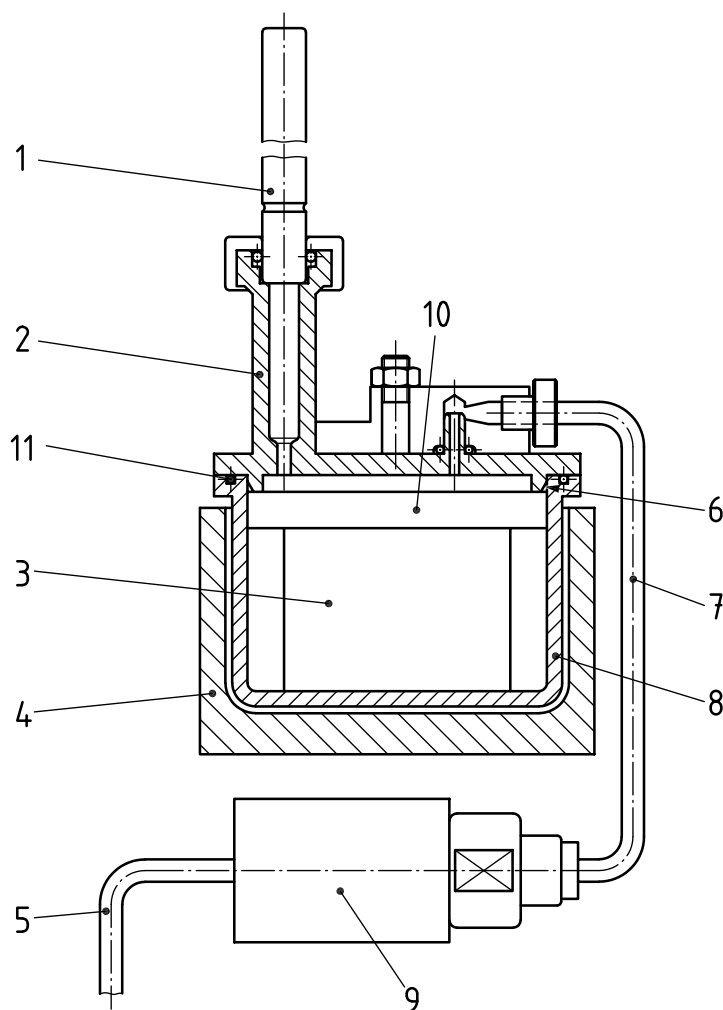
- 1 air outlet and connection to vapour sampling tube
- 2 heated air inlet
- 3 sample for surface emission testing
- 4 in surface emissions testing, the space below the sample is filled with metal plates (typically aluminium)
- 5 low emitting seal
- 6 sample for bulk emission testing

**Figure A.1 — Schematic diagram of key micro-scale chamber components**

## Annex B (informative)

### Micro-scale chamber — Example 1

A first typical micro-scale chamber is shown in Figure B.1. Dimensions and performance specifications for this type of micro-scale chamber are listed in Table B.1.



**Key**

- |   |                           |
|---|---------------------------|
| 1 vapour-sampling tube                              | 7 air supply (heated)     |
| 2 air outlet  | 8 micro-scale chamber     |
| 3 metal spacers (typically aluminium)               | 9 flow-controlling device |
| 4 heated block                                      | 10 sample                 |
| 5 pressurized air supply                            | 11 low-emitting seal      |
| 6 collar to exclude edge and rear surface emissions |                           |

**Figure B.1 — First example of a micro-scale chamber shown in cross-section as used for surface emission testing**

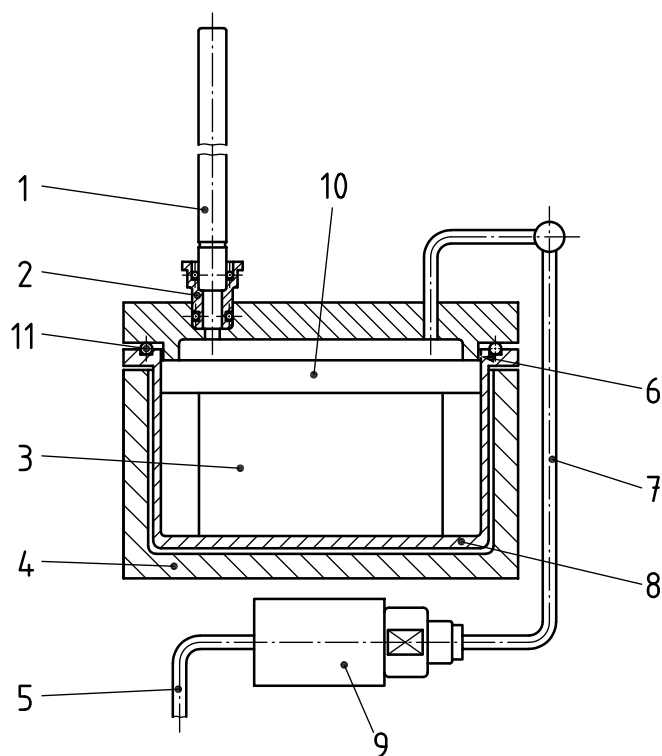
Table B.1 — Dimensions and ancillary data for the micro-scale chamber example shown in Figure B.1

<b>Empty micro-chamber (bulk emissions testing)</b>					
Chamber diameter, mm	45				
Chamber depth, mm	28				
Volume (empty chamber), ml	44,5				
Gas flow rate range, ml/min	10 to 500				
Gas change rate range, h <sup>-1</sup>	13 to 674				
<b>Test material sealed against collar on lid (surface emission testing)</b>					
Volume, m <sup>3</sup>	3,20 × 10 <sup>-6</sup>				
Diameter of sample exposed, mm	40,4				
Maximum exposed surface area, m <sup>2</sup>	1,28 × 10 <sup>-3</sup>				
Load factor, m <sup>2</sup> • m <sup>-3</sup>	400				
Gas flow rate, ml/min (range 10 ml/min to 500 ml/min)	50	100	250	500	
Corresponding gas change rate, h <sup>-1</sup>	938	1 875	4 690	9 375	
Area specific gas flow rate, $q_{VA}$ , m <sup>3</sup> • h <sup>-1</sup> • m <sup>-2</sup>	~2,34	~4,69	~11,7	~23,4	

## Annex C (informative)

### Micro-scale chamber — Example 2

A second typical micro-scale chamber is shown in Figure C.1. Dimensions and performance specifications for this type of micro-scale chamber are listed in Table C.1.



**Key**

- |   |                           |
|---|---------------------------|
| 1 vapour sampling tube                              | 7 air supply (heated)     |
| 2 air outlet  | 8 micro-scale chamber     |
| 3 metal spacers (typically aluminium)               | 9 flow-controlling device |
| 4 heated block                                      | 10 sample                 |
| 5 pressurized air supply                            | 11 low-emitting seal      |
| 6 collar to exclude edge and rear surface emissions |                           |

**Figure C.1 — Second micro-scale chamber example shown in cross-section as used for surface emission testing**

Table C.1 — Dimensions and ancillary data for the second micro-scale chamber example

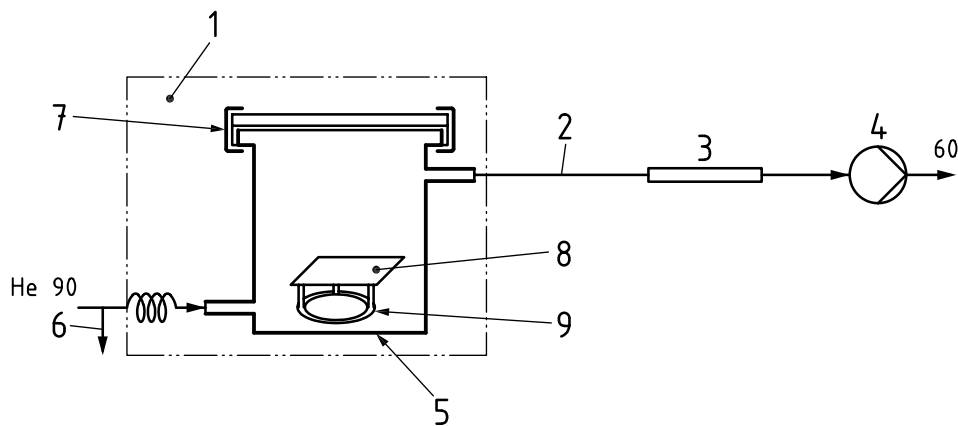
<b>Empty micro-chamber (bulk emission testing)</b>					
Chamber diameter, mm	64				
Chamber depth, mm	36				
Volume (empty chamber), m <sup>3</sup>	$1,14 \times 10^{-4}$				
Gas flow rate range, ml/min	10 to 500				
Gas change rate range, h <sup>-1</sup>	10 to 500				
<b>Test material sealed against collar on lid (surface emission testing)</b>					
Volume, m <sup>3</sup>	$7,38 \times 10^{-6}$				
Diameter of sample exposed, mm	56,0				
Maximum exposed surface area, m <sup>2</sup>	$2,46 \cdot 10^{-3}$				
Load factor, m <sup>2</sup> • m <sup>-3</sup>	333				
Gas flow rate, ml/min (range 10 ml/min to 500 ml/min)	50	100	250	500	
Corresponding gas change rate, h <sup>-1</sup>	407	813	2 033	4 065	
Area specific gas flow rate, $q_{VA}$ , m <sup>3</sup> • h <sup>-1</sup> • m <sup>-2</sup>	~1,22	~2,44	~6,10	~12,2	

## Annex D (informative)

### Micro-scale chamber — Example 3

A third typical micro-scale chamber is shown in Figures D.1 and D.2. Dimensions and performance specifications for this type of micro-scale chamber are listed in Table D.1.

Flow rates in millilitres per minute

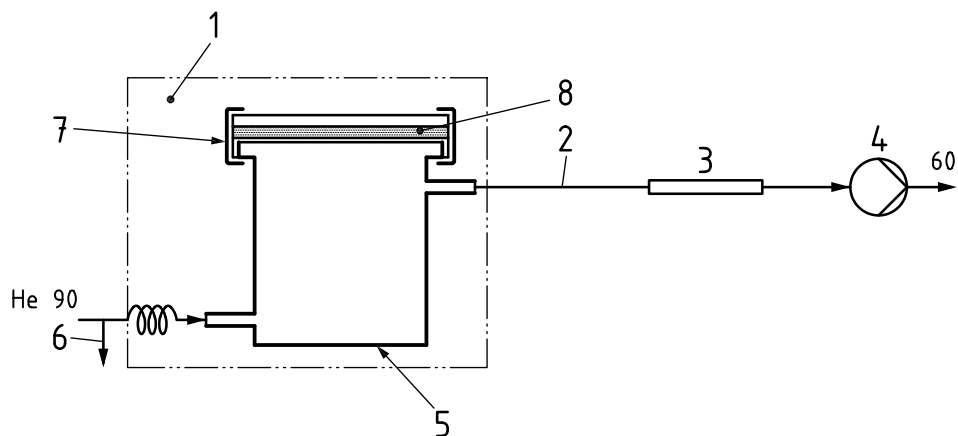


**Key**

- |   |                       |                                |
|---|-----------------------|--------------------------------|
| 1 climate chamber (adjusted to 65 °C)       | 4 sampling pump       | 7 clamp                        |
| 2 transfer line (minimum temperature 65 °C) | 5 micro-scale chamber | 8 automotive interior material |
| 3 Tenax TA <sup>®</sup> (1) tube            | 6 vent line           | 9 quartz stand                 |

**Figure D.1 — Third micro-scale chamber example (bulk emission testing)**

Flow rates in millilitres per minute



**Key**

- |   |                                |
|---|--------------------------------|
| 1 climate chamber (adjusted to 65 °C)       | 5 micro-scale chamber          |
| 2 transfer line (minimum temperature 65 °C) | 6 vent line                    |
| 3 Tenax TA <sup>®</sup> (1) tube            | 7 clamp                        |
| 4 sampling pump                             | 8 automotive interior material |

**Figure D.2 — Third micro-scale chamber example (surface emission testing)**

Table D.1 — Dimensions and specifications for micro-chamber

Micro-chamber property	Dimension
Chamber diameter, mm	82
Chamber depth, mm	125
Volume (empty chamber), m <sup>3</sup>	$6,3 \times 10^{-4}$
Gas flow rate range, ml/min	30 to 100
Gas change rate range, h <sup>-1</sup>	4 to 10

## Annex E (informative)

### Assessment of VOC recoveries

The recovery of organic vapours can be determined by introducing a known representative mass of one or more specific target analytes into the micro-scale chamber on a suitable inert substrate — e.g. a 1 mm thickness polytetrafluoroethylene or metal disk — and by sampling the total mass of vapour recovered from the micro-scale chamber exhaust. The air flow and temperature shall be allowed to equilibrate under analytical conditions before the test compound is introduced. Examples of compounds commonly used for recovery tests include: toluene, *n*-dodecane, and deuterated equivalents.

**NOTE** If the test compounds are prepared in solution (e.g. 1 % volume fraction toluene in solvent), use a solvent such as methanol, which is not well retained by Tenax TA<sup>®</sup>,<sup>1)</sup> to minimize analytical interference.

Once the micro-scale chamber has been allowed to equilibrate under analytical conditions, place the substrate into the micro-scale chamber and introduce a small volume (typically 0,5 µl to 2 µl) of the test solution. Immediately seal the micro-scale chamber, connect a conditioned vapour sampling tube (typically packed with Tenax TA<sup>®</sup><sup>1)</sup> sorbent) and switch on the air flow. Replace the vapour sampling tube after 15 min with a fresh conditioned vapour sampling tube. Subsequently analyse each vapour sampling tube using normal test conditions.

If tests on recovery of very volatile organic compounds are required, it may be preferable to introduce a permeation- or diffusion-controlled source of the compound of interest into the micro-chamber to prevent risk of evaporative loss before the micro-chamber can be sealed.

Satisfactory recovery is demonstrated by >80 % recovery on the first sample tube and <20 % recovery on the second sample tube.

If these conditions are not met, remove the substrate used to load the test compound and place it in a clean micro-scale chamber under the same conditions of temperature and flow. Continue the experiment by collecting two additional pairs of 15 min vapour samples in parallel from both the original micro-scale chamber (that had the substrate and test compound removed) and the new micro-scale chamber to which the substrate and any residual test compound have just been added.

If these secondary tests show continued presence of the test compound on the substrate, but no carry-over into the micro-scale chamber apparatus, incomplete recovery has been shown to be a function of slow vaporization of the target compound from the substrate, not of carry-over into the micro-scale chamber apparatus. In this case, adequate recovery has been demonstrated through the apparatus. However, if these tests show that no test compound remains on the substrate, but that losses in the apparatus exceed 20 %, this indicates unsatisfactory recovery of that compound. Possible causes could be leaks or contamination within the apparatus. These shall be addressed before using the apparatus for sample tests.

To a greater or lesser extent, depending on micro-chamber design, it is expected that heavier SVOCs (e.g. compounds less volatile than *n*-C<sub>18–22</sub>) may condense on the inner walls of the apparatus, thus compromising complete recovery. For this reason, quantitative SVOC studies using micro-scale chambers may need to be carried out over extended periods (e.g. 8 h or 24 h) or by using an alternative, two-step procedure, such as that specified in ISO 16000-25.<sup>[10]</sup>



## Annex F (informative)

### Calculation of specific emission rates from micro-scale chamber air concentration

The area specific emission rate,  $q_{mA}$ , in micrograms per square metre hour, can be derived from micro-scale chamber air concentrations using the conventional calculations described in ISO 16000-9<sup>[6]</sup> and ISO 16000-10<sup>[7]</sup> and as reproduced below.

$$\gamma_{VOC} = q_{mA} \frac{L}{n} = \frac{q_{mA}}{q_{VA}} \quad (F.1)$$

converted to

$$q_{mA} = \gamma_{VOC} q_{VA} \quad (F.2)$$

with

$$q_{VA} = \frac{n}{L} \quad (F.3)$$

where

- $\gamma_{VOC}$  is the VOC concentration, in micrograms per cubic metre, in sample air;
- $n$  is the number of air exchanges per hour;
- $L$  is the loading factor (sample area/chamber volume), in square metres per cubic metre;
- $q_{VA}$  is the area specific flow rate, in cubic metres per square metre hour.

#### EXAMPLE

The micro-scale chamber described in Annex B, has a loading factor

$$L = \frac{1,28 \times 10^{-3}}{3,20 \times 10^{-6}} = 400 \text{ m}^2 \cdot \text{m}^{-3}$$

When using it for surface emission testing of VOCs with an air or gas flow rate of 50 ml/min ( $0,003 \text{ m}^3 \cdot \text{h}^{-1}$ ), the air or gas change rate

$$n = \frac{0,003}{3,20 \times 10^{-6}} = 938 \text{ h}^{-1}$$

and the area specific air or gas flow rate

$$q_{VA} = \frac{938}{400} = 2,34 \text{ m}^3 \cdot \text{h}^{-1} \cdot \text{m}^{-2}$$

(See Table B.1.)

The VOC concentration in air,  $\gamma_{VOC}$ , in micrograms per metre cubed, is determined from the measured analyte mass and volume of air or gas sampled as specified in ISO 16000-6, Clause 11, or ISO 16000-3, Clause 10, using the correction for 23 °C and 101,3 kPa.

The area specific emission rate,  $q_{mA}$ , for VOCs using this style of micro-scale chamber under these conditions is therefore,

$\gamma_{\text{VOC}} q_{\text{VA}}$

i.e. for this style of micro-scale chamber

$$q_{mA} = \gamma_{\text{VOC}} \times 2,34 \mu\text{g} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$$

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- [1] VDA 276, *Bestimmung organischer Emissionen aus Bauteilen für den Kfz-Innenraum mit einer 1 m<sup>3</sup>-Prüfkammer* [Measurement of the emissions of automotive interior assemblies in a 1 m<sup>3</sup> test chamber, standard VDA]
- [2] SCHRIPP T., NACHTWEY B., TOELKE J., SALTHAMMER T., UHDE E., WENSING M. BAHADIR, M. A microscale device for measuring emissions from materials for indoor use. *Anal. Bioanal. Chem.* 2007, **387**, pp. 1907–1919
- [3] WILLIAMS G.J., PHARAOH M. *PARD Report: Correlation between the VDA 276 test and micro-chamber testing*. Issued by WMG. University of Warwick, UK, 2009
- [4] KIM, M.G. Determination of chemical emissions from car interior parts. Presentation to Joint Working Group 13 of ISO/TC 146/SC 6 and ISO/TC 22 (N 0069) and series of papers in press

### Documents on VOCs cited informatively

- [5] ISO 16000-5, *Indoor air — Part 5: Sampling strategy for volatile organic compounds (VOCs)*
- [6] ISO 16000-9, *Indoor air — Part 9: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test chamber method*
- [7] ISO 16000-10, *Indoor air — Part 10: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test cell method*
- [8] 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*
- [9] ISO 16000-24, *Indoor air — Part 24: Performance test for evaluating the reduction of volatile organic compound (except formaldehyde) concentrations by sorptive building materials*
- [10] ISO 16000-25, *Indoor air — Part 25: Determination of the emission of semi-volatile organic compounds by building products — Micro-chamber method*
- [11] 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*
- [12] ISO 16017-2, *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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