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Interior air of road vehicles —

Part 4: Method for the determination of the emissions of volatile organic compounds from vehicle interior parts and materials — Small chamber method

Air intérieure des véhicules routiers —

Partie 4: Méthode pour la détermination des émissions de composés organiques volatils des parties et des matériaux intérieurs des véhicules — Méthode de la petite chambre

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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.

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ISO 12219-4 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: Method for the determination of the emissions of volatile organic compounds from vehicle interior parts and materials — Small chamber method*
- *Part 5: Screening method for the determination of the emissions of volatile organic compounds from vehicle interior parts and materials — Static chamber method*

The following parts are under preparation:

- *Part 6: Method for the determination of the emissions of semi-volatile organic compounds from vehicle interior parts and materials — Small chamber method*
- *Part 7: Odour determination in interior air of road vehicles and test chamber air of trim components by olfactory measurements*

Introduction

Volatile organic compounds (VOCs) are widely used in industry and can 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 $\geq 4 \text{ m}^3$). 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 in vehicle trim components under controlled conditions using a small emission test chamber (small chamber). It describes requirements for a small chamber and a test protocol. Measurements are carried out according to ISO 16000-6 (VOCs) and ISO 16000-3 (carbonyl compounds).

The capacity of a small chamber is not limited to small assemblies or representative samples of homogeneous car trim materials. Small chambers allow qualitative and quantitative VOC emission data to be measured and recorded. The subsequent VOC emission data can be used to develop a correlation between material level methods and the vehicle level method.

This part of ISO 12219 is based on VDA 276^[2] and ASTM D5116,^[1] and correlates to ISO 16000-9.^[4]

ISO 16000-3, ISO 16000-5,^[3] ISO 16000-6, ISO 16000-9,^[4] ISO 16000-10,^[5] ISO 16000-11,^[6] ISO 16000-24,^[7] ISO 16000-25,^[8] as well as ISO 16017-1^[9] and ISO 16017-2^[10] also focus on VOC measurements.

Interior air of road vehicles —

Part 4:

Method for the determination of the emissions of volatile organic compounds from vehicle interior parts and materials — Small chamber method

WARNING — It is the responsibility of the user of this part of ISO 12219 to establish appropriate safety and health practices and to 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 qualitative and quantitative analytical method for vapour-phase organic compounds (volatile and some semi-volatile) released from car trim materials under simulated real use conditions using small emission test chambers (small chamber). Small chambers are intended to provide a transfer function to vehicle level emissions. This method is intended for evaluating new car interior trim components but can, in principle, be applied to used car components.

Target compounds include VOCs (conventionally defined as organic compounds in the volatility range *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 Compounds more volatile than *n*-hexane and less volatile than *n*-hexadecane can also be analysed (see ISO 16000-6:2011, Annex D, and ISO 16017-1^[8] for more information).

This part of ISO 12219 is complementary to ASTM D5116^[1] and VDA 276,^[2] and provides third party test laboratories and manufacturing industry with an approach for:

- a) identifying the effect of real use conditions on specific VOC emissions data;
- b) comparing emissions from various assemblies with regards to specific VOC emissions;
- c) evaluating and sorting specific assemblies regarding specific VOC emissions data;
- d) providing specific VOC emissions data to develop and verify a correlation between material level methods and the vehicle level method;
- e) evaluating prototype, “low-emission” assemblies during development.

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 3833, *Road vehicles — Types — Terms and definitions*

ISO 16000-3:2011, *Indoor air — Part 3: Determination of formaldehyde and other carbonyl compounds in indoor air and test chamber air — 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® 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 3833 and the following apply.

**3.1
air change rate**
volume ratio of clean air brought into the small chamber per hour and the free small chamber volume measured in identical units

[SOURCE: ISO 16000-9:2006,⁴ definition 3.1, modified]

**3.2
air circulation**
ideal mixing of the small chamber

**3.3
air flow rate**
air volume entering into the small chamber per time

[SOURCE: ISO 16000-9:2006,⁴ definition 3.2, modified]

**3.4
air sample**
representative quantitatively preset volume of the atmosphere in the small chamber

**3.5
air speed**
medium air speed over the surface of the test specimen

**3.6
background concentration**
concentration of a specific volatile organic compound, VOC_x , (or group of volatile organic compounds) measured in the small chamber outlet

**3.7
clean air supply**
pressurized purified air or synthetic air in gas cylinders

**3.8
small chamber**
enclosure with controlled operational parameters for the destination of volatile organic compounds emitted from products

**3.9
small chamber concentration**
concentration of a specific volatile organic compound, VOC_x , (or group of volatile organic compounds) measured in the small chamber outlet

**3.10
emission**
organic substances that escape from the component under the existing test conditions

**3.11
ideal air mixing**
substance diffusing in an ideally mixed chamber, without time delay, completely and homogeneously throughout the whole chamber

**3.12
supply air**
sum of all gaseous volume flows conducted into the small chamber

Note 1 to entry: The supply air is quantified as a volume flow per time.

3.13**product loading factor**

ratio of exposed surface area (or mass or volume) of the test specimen and the free small chamber volume

3.14**recovery rate**

measured mass of a target volatile organic compound in the air leaving the small chamber during a given time period divided by the mass of target volatile organic compound added to the small chamber in the same time period

Note 1 to entry: The recovery provides information about the performance of the entire method.

Note 2 to entry: The recovery rate is expressed in per cent.

3.15**sample**

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

[SOURCE: ISO 16000-9:2006,⁴ definition 3.10]

3.16**specific emission rate**

q_x

product-specific rate describing the mass of a volatile organic compound emitted from a product per time at a given time from the start of the test

Note 1 to entry: Area-specific emission rate, q_A , is used in this part of ISO 12219. Several other specific emission rates can be defined according to different requirements, e.g. length-specific emission rate, q_l , volume-specific emission rate, q_V , and unit-specific emission rate, q_U .

Note 2 to entry: The term “area-specific emission rate” is sometimes used in parallel with the term “emission factor”.

[SOURCE: ISO 16000-9:2006,⁴ definition 3.11, modified]

Note 3 to entry: The specific emission rate is expressed in units of mass per time.

3.17**surface of the component****surface of the automotive interior product**

surface that is formed by the outline of the component or automotive interior product and penetrable by organic substances

3.18**target volatile organic compound**

product-specific volatile organic compound

3.19**test specimen**

part of the sample specifically prepared for emission testing in the small chamber in order to simulate the emission behaviour of the material or product that is tested

[SOURCE: ISO 16000-9:2006,⁴ definition 3.13, modified]

3.20

total volatile organic compounds

TVOCs

sum of volatile organic compounds, sampled on Tenax TA[®],¹⁾ which elute between and including *n*-hexane and *n*-hexadecane on a non-polar capillary column, detected with a flame ionization detector (TVOC-FID) or mass spectrometric detector (TVOC-MS), and quantified by converting the total area of the chromatogram in that analytical window to a nominal mass using the chromatographic response factor for toluene (toluene equivalents)

[SOURCE: ISO 16000-6:2011, definition 3.4, modified]

Note 1 to entry: While this part of ISO 12219 specifies the determination of individual VOCs, it is common in practice to generate a single concentration value to characterize the total amount of VOCs present in the air. This value is called the TVOC value. It should be emphasized that the TVOC value so obtained depends on the sampling and analytical methods used, and therefore should be interpreted taking into account the full description of these methods.

3.21

outlet air

air leaving the small chamber through a fixed opening

3.22

volatile organic compound

VOC

organic compound that is emitted from the test specimen and all those detected in the small chamber outlet air

[SOURCE: ISO 16000-9:2006,⁴ definition 3.15, modified]

Note 1 to entry: Due to practical reasons to be taken into account for test chambers, this definition differs slightly from that defined in ISO 16000-6:2011. In ISO 16000-6, the definition is based on the boiling point range (50 °C to 100 °C) to (240 °C to 260 °C).

Note 2 to entry: The emission test method described in this part of ISO 12219 is optimum for the range of compounds specified by the definition of total volatile organic compounds (TVOCs).

3.23

volume of the small chamber

chamber volume minus the technical fittings and devices in the small chamber that take up volume

4 Symbols

Symbol	Meaning	Unit
<i>t</i>	time	hours or days
γ_X	mass concentration of substance X	micrograms per cubic metre
<i>q</i>	area specific air flow rate (= n/L_A)	cubic metres per square metre hour
q_u	unit specific emission rate	micrograms per hour
q_l	emission rate per length	micrograms per metre hour
q_A	emission rate per area	micrograms per square metre hour
q_V	emission rate per volume	micrograms per cubic metre hour

1) Tenax is the trademark 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.

q_m	emission rate per mass	micrograms per kilogram hour
n	air change rate	per hour
L_A	surface loading of chamber	square metres per cubic metre

5 Principle

A component or automotive interior product is inserted into an almost ideally mixed small chamber (0,5 m³ to 4,0 m³) and stored under conditions of preset temperature, humidity and air change or air renewal. Organic substances that escape the component accumulate in the small chamber and are conveyed through an airstream.

Air samples are taken at selected times. The concentration of gaseous air substances in the small chamber can be determined qualitatively and quantitatively using chemical analysis procedures and from that the rate of emission of target compounds from the test material can be determined.

6 Emission test bed preparation

6.1 Components

A test bed to determine gaseous emissions consists of the following functional components or operational elements:

- small chamber;
- air circulation;
- clean air supply;
- temperature, humidity, and flow control and regulation;
- sample line.

There are no mandatory guidelines for construction, arrangement, combination, and technical finish of these individual functional components. Hints for continual measurers for quality assurance are given in Clause 7.

6.2 Small chamber

6.2.1 General

The small chamber is an airtight container with the volume of 0,5 m³ to 4,0 m³. A typical standard small chamber size has a volume of 1 m³ ± 0,05 m³. The chamber volume shall be specified in the test report. Inside there is a device for mixing the air and a stand to guarantee the storage of the component without touching the walls. An inflow pipe and an outlet air pipe shall be provided to adjust the air change (air renewal) or to test the air. An example of a small chamber is shown diagrammatically in Figure 1.

6.2.2 Materials

General specifications and requirements, which apply to all types of small chambers, are provided in the following.

The small chamber method requires the following key components.

- Airtight small chamber apparatus.
- The wall surfaces of the small chamber and the rack for placement of the component made of electropolished high-quality steel. In the case of installations or parts in the interior for which

electropolished high-quality steel cannot be used due to technical reasons, materials shall be selected that have low emissions and absorption of organic substances.

- Heating mechanism and temperature control system.
- Sampling line, constructed of an inert, non-emitting and non-absorbing and adsorbing material and heated, if necessary, to prevent condensation/deposition on the inner walls. The length of the sampling line shall be as short as possible and is restricted to about 3 m.
- Clean air supply and humidification system.
- Appropriate monitoring and control systems (to ensure that the test is carried out according to specified conditions). Appropriate vapour sampling tubes are also required.

Any sealing materials (e.g. gaskets or O-rings) used for sealing the doors or lids of the small chambers, shall be low emitting and low absorbing. They shall not contribute significantly to the background vapour concentration. The O-rings or gaskets should be easily removed to facilitate cleaning or replacement. Surface areas of these parts that are in contact with the small chamber atmosphere shall not exceed in their sum 5 % of the small chamber walls.

6.2.3 Tightness

In order to avoid uncontrolled air convection, ensure that the leakage caused by non-tightness amounts to less than 0,5 % volume fraction of the small chamber per minute or 5 % volume fraction of the supply air in tests with air change (air renewal) at 1 000 Pa excess pressure. In order to avoid air inflow from outside, a small excess pressure with regard to the atmospheric pressure in the laboratory or a volume over-current shall be used. In particular, such excess pressure is necessary when taking air samples.

6.2.4 Air mixing

This method relies on the air inside the chamber being thoroughly mixed. Have available a suitable device for mixing the air that can also fulfil this prerequisite when testing large-volume, bulky material.

The flow rate (flow velocity) in the middle of the empty small chamber shall exceed 0,1 m/s.

NOTE 1 Adequate equipment for air velocity measurements are hot wire or film anemometers calibrated in the range 0,1 m/s to 0,5 m/s.

6.2.5 Cleaning

Ensure, through suitable cleaning procedures, such as thermal desorption at high temperatures, that thorough cleaning between tests is carried out. The background sample of the empty small chamber shall meet the background requirements described in 7.3. When the background value cannot be achieved, the small chamber shall be cleaned. A detailed description is given in 9.2.

6.3 Small chamber temperature control

The chamber temperature shall be precisely controlled because there is a strong link between temperature and chemical emission rate. The chamber shall be able to maintain a given temperature to within ± 1 °C. Emission rates are specific to a particular temperature, therefore it is essential to maintain a constant temperature within the small chamber throughout the emission test/comparative analyses.

6.4 Air humidification

The humidification of the supply air in the small chamber shall be carried out in such a way that the formation of steam, vapour, and aerosols is ruled out. Maintain a relative humidity of 5 % at 65 °C in the supply air using the humidification unit. This corresponds to a relative humidity of 50 % at 21 °C or a dew point of 10,4 °C. It is typical to maintain a targeted dew point temperature and then to reheat to a

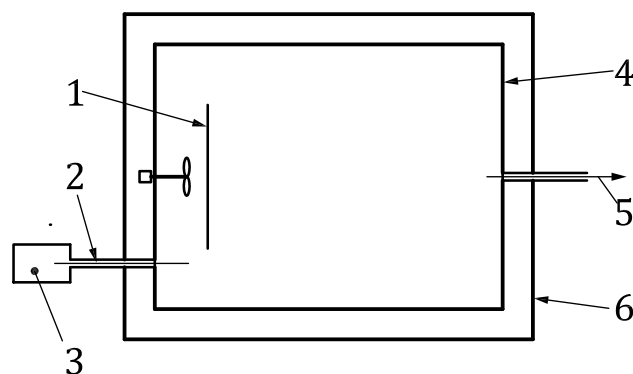
specified air temperature by the feedback from a RH sensor in the chamber. Maintaining a constant dew point and air temperature produces very constant relative humidity.

6.5 Clean air supply

Tests with air change or air renewal and the taking of air samples require a flow of clean air. An air change or air renewal of 0,4 per hour, under the test conditions (65 °C, ambient pressure) shall be set with an accuracy of $\pm 5\%$.

The sum of the concentrations of volatile organic hydrocarbon combinations (TVOCs, see 3.20) in the supply air shall not be more than $50\ \mu\text{g}/\text{m}^3$ in the sum and $5\ \mu\text{g}/\text{m}^3$ for individual substances before the start of a test.

Particulate matter in the clean supply shall be reduced through a particle filter (e.g. HEPA filter) in order to avoid an adsorption of compounds.



Key

- | | | | |
|---|----------------------------|---|-------------------------------------|
| 1 | air circulation | 4 | small chamber |
| 2 | controlled humidified flow | 5 | sample line |
| 3 | clean air supply | 6 | temperature-controlled test cabinet |

Figure 1 — Emission test bed of the small chamber

7 Quality control

7.1 General

The minimum requirements for small chamber emissions are listed below.

Errors can occur through the integration of numerous technical functions in a small chamber which requires a regular and conscientious inspection of the whole system. Since these errors can have an effect in part directly on a test result, the small chamber shall be integrated into a creditable quality assurance system or a comparable continual observation method.

Several important test methods for measuring test parameters are described in 7.2 to 7.4.

7.2 Airtightness

7.2.1 General

Check the airtightness of the small chamber at an excess pressure of 1 000 Pa by measuring the pressure drop in a period of 2 h. The sensitiveness of the pressure pick-off/pressure transducer should be smaller

than 100 Pa with an accuracy of $\pm 5\%$. The average value of the specific leak rate, n_L , referring to the small chamber volume in thousandths per hour in this period is calculated using Formula (1):

$$n_L = \frac{1\,000}{t} \left[\frac{p_1}{p_2} - 1 \right] \quad (1)$$

where

- p_1 is the absolute pressure in the small chamber at the start of the test, in pascals;
- p_2 is the absolute pressure in the small chamber at the end of the test, in pascals;
- t is the period of time of the leak rate determination, in hours.

7.2.2 Alternative procedure 1

The tightness of the small chamber is determined by measuring the half-life $t_{1/2}$ of the pressure drop in the small chamber with an excess pressure of 1 000 Pa. Half-life is the period of time in which the excess pressure has reached half of its initial value. With it, the specific leak rate, n_L , %, with reference the small chamber expressed as a percentage per hour, can be determined according to Formula (2):

$$n_{L, \%} = \frac{100}{t} \frac{\Delta p}{p} \frac{\ln 2}{t_{1/2}} \quad (2)$$

where

- $\Delta p/p$ is the relative excess pressure with reference to the ambient pressure;
- $t_{1/2}$ is the period of time until the decrease of the excess pressure to half of the initial value, in hours.

In each procedure, the tightness is determined in a small chamber at a temperature of 65 °C.

7.2.3 Alternative procedure 2

The small chamber shall be airtight if the leakage is less than 5 % of the supply air flow.

7.2.4 Air speeds

The air speeds are measured centrally in the empty small chamber. Hot wire, film or impeller wheel anemometers can be used for measuring.

7.3 Recovery and sink effects

The recovery of organic vapours can be determined by introducing a known mass of one or more specific target VOCs into the chamber on a suitable substrate and by sampling the total mass of vapour recovered from the small chamber exhaust. Examples of compounds commonly used for recovery tests include: toluene, *n*-dodecane, and surrogate polar compounds, e.g. 2-ethylhexanol. The addition of a known mass of a compound into the small chamber can be performed via a syringe injected on a substrate or with a known mass of the compound in the supply air. The substrate shall be placed in the middle of the small chamber. Contact with the walls of the chamber should be avoided. A set of several substrates shall be arranged in such a way that an air flow through the circulation is achieved from all sides in the best way possible. Attention should be paid to a reasonable product loading factor. The small chamber shall be closed or locked immediately after placing the compound on the substrate.

The tests of analytic recovery shall be carried out under normal test conditions, e.g. normal time, 65 °C, and an exchange rate of 0,4 times per hour.

NOTE 1 If a particular small chamber consistently underperforms with respect to the recovery, this is most likely to be the result of leaks or sink effects within the small chamber.

NOTE 2 Low recovery of hygroscopic VOCs may occur in humidified air.

NOTE 3 Sink effects, leaks or poor calibration can cause difficulties with achieving the desired recovery. Sink and adsorption characteristics are very much dependent on the type of compound emitted. Additional recovery tests using target VOCs with different molecular weight and polarity can be used to increase understanding of these effects.

The mass of vapour recovered experimentally should ideally be greater than 80 % of that introduced.

7.4 Supply air

7.4.1 General

The small chamber shall have facilities (e.g. electronic mass flow controller) capable of continuously controlling the temperature, relative air humidity, and air flow. These shall be monitored and recorded continuously with instruments meeting the following accuracy:

- temperature ± 1 °C
- relative air humidity $\pm 0,5$ % RH at 65 °C or ± 5 % RH at 21 °C
- standard air flow rate ± 5 %

The air speed should be regularly checked with a minimum frequency of every 12 months with an accuracy of $\pm 0,1$ m/s. A hot-wire anemometer is typically used for the measurement. The air speed should be measured in at least one position in the centre of the empty small chamber.

The air change rate shall be regularly checked with a minimum frequency of every 12 months using a calibrated gas meter. The air change rate shall not vary by more than ± 5 % of the set value.

If the test is carried out on the outlet with a gas volume meter or flow meter that is not permanently installed, be aware that the back pressure introduced by the instrument can lower the flow rate through the small chamber.

7.4.2 Background concentration values

Background concentration values, for substances for quantitative analytical methods should be recorded with the help of regular checks.

A screening procedure, for example Tenax¹⁾ and thermal desorption with a sensitivity of at least 10 % for individual substances, shall be used for a quantitative blank value observation.

The chamber blank level is acceptable if the concentrations of individual target compounds that have been determined with this procedure are no greater than 10 % of the same target compounds measured.

7.4.3 Temperature and humidity

Maintenance within tolerances of temperature and relative humidity shall be verified with a combined temperature and humidity sensor or gauge as $\pm 0,5$ °C or ± 5 % respectively.

If there is sufficient experience with possible condensation effects inside the chamber, the humidity can also be measured in the supply air.

8 Component or automotive interior product

8.1 General

When determining volatile organic emissions, the conditions the component or automotive interior product was exposed to before the test, can have a considerable effect on the results, especially in quantitative tests. Therefore, it is necessary to standardize the past history of the component or automotive interior product as much as possible.

The test samples shall have been put through all of the steps of production intended for the series production process with all of the pertinent auxiliary production equipment. Deviations shall be documented in precise detail in the test report.

8.2 History of the component or automotive interior product

If it is necessary for test purposes to dismantle a component partly, to separate it or to change it in any way, then this procedure shall be documented. Soiling should be avoided.

Possibly, the adsorption of substances from the environment that have not been in the component originally also has to be expected. The past history of the component before the test shall be documented as completely as possible.

8.3 Packaging, transport and storage of the component or automotive interior product

The component shall be thoroughly protected from chemical contamination or any physical exposure, e.g. heat, light and humidity, until the start of the test.

For solid products, this can usually be achieved by wrapping each specimen separately in aluminium foil and in a polyethylene bag or alternatively, in aluminized packaging lined with polyethylene or clear poly(vinyl fluoride) film. Liquid products shall be shipped in unopened can, tubes, etc.

During temporary storage and during transporting, the component shall be kept in its packaging and the temperature of 23 °C should not be exceeded during any period.

The component shall be labelled with the details of the type of product, day of manufacture (if known) and/or any identification numbers or batch numbers.

The storage time should be not longer than 3 months.

Storage can affect the emission properties due to aging of the component. It is recommended that the storage time of the sample be minimized prior to testing.

9 Standard emission test procedure

9.1 General

In this clause, the procedures, arrangements and requirements of the standard emission test in the chamber are described. This test gives a broad overview over the emission spectrum of components under intensified climatic conditions.

The test temperature in the first conditioning phase is 65 °C with a humidity of the supply air of approx. 5 % RH (this corresponds to 45 % RH. at 23 °C). The air change rate or air renewal is 0,4 exchanges per hour.

By default, air samples are taken to determine the concentrations of carbonyl compounds and VOCs. In special cases, additional samples for the determination of such compounds as *N*-nitrosamines, isocyanates, and amines can be taken. Also an additional conditioning phase and measurements, e.g. flame-ionization detection (FID), can be subsequently added to the procedure. The test procedure shall be documented in the report.

Determine the test and blank sample concentrations of VOCs, formaldehyde and other carbonyl compounds by selecting the appropriate sampling media, e.g. sorbent tube for VOCs or DNPH cartridge (or equivalent) for aldehydes (see ISO 16000-6 or ISO 16000-3, respectively for more details).

The specified analytical procedure is valid for the determination of VOCs ranging in concentration from sub- $\mu\text{g}/\text{m}^3$ to several mg/m^3 . The method is applicable to the measurement of non-polar and slightly polar VOCs ranging in volatility from $n\text{-C}_6$ to $n\text{-C}_{16}$.

9.2 Cleaning and purification

All pollutant-subjected surfaces in the small chamber shall be in a purified condition before the start of a component test. If there are any background concentrations, they have to be kept so low that the quality of the results from analytical methods are not influenced by them (blank values less than 10 % of the sample values).

The small chamber should be freed of all particles or similar remains of the components with mechanical purification methods. Remove any O-rings or gaskets and clean the small chamber components using an alkaline detergent, followed by two separate rinsings with distilled water or by using an appropriate solvent and drying thoroughly.

Alternatively, the small chamber assembly can be heated for cleaning. Raise the empty, sealed small chamber to a temperature of 180 °C or more in a fast flow of pure gas until background artefacts are reduced to negligible levels (see 6.5).

If the small chamber has an inert coating, care shall be taken not to damage the coating during cleaning (e.g. by using abrasive cleaners and/or high pH).

9.3 Test

Actual testing extends from unpacking the specimen from the emissions-tight laminated aluminium foil to removal of the specimen from the small chamber upon conclusion of all conditioning and sampling operations.

9.3.1 Preliminary storage

The test specimens are removed from the packaging 1 week before the test (the protectors shall be removed in the case of components, if applicable) and stored at 23 °C \pm 2 °C and 50 % RH \pm 10 % RH. In order to prevent contamination of the test specimens with hydrocarbons, attention shall be paid to appropriate air exchange in the storage room.

The parts shall be stored individually with sufficient space between them. Ensure that all surfaces of the test specimen can be ventilated without obstruction and that the parts are not placed on their visible sides. Flat-spread materials in particular (e.g. leather, fabrics, foils, plastics plates) shall be placed on a rack or grating.

Deviations from the preliminary storage procedure described shall be documented in precise detail in the test report.

9.3.2 Preparation

The emission test bed of the entire small chamber shall be checked carefully for all necessary functions before the start of the test.

9.3.3 Cleaning — Phase 1

The small chamber is cleaned by heating to 180 °C or max 230 °C, if possible overnight. The cleaned small chamber is checked by a background measurement.

9.3.4 Preconditioning — Phase 2

The small chamber shall be conditioned to a temperature of 65 °C ± 2 °C and a relative humidity from 4,5 % to 5,5 % or a corresponding water dew point from 8,6 °C to 11,6 °C.

Humidification of the supply air shall be started in good time in order to achieve the right humidity in the small chamber before the start of the test.

9.3.5 Background concentration sampling — Phase 3

An air sample of the emission small chamber background concentration should be taken prior to the start of a new emission test.

9.3.6 Inserting the component or automotive interior product — Phase 4

The component shall be put on a stand in the middle of the small chamber configured as it is in the vehicle, if possible. Contact with the walls of the chamber should be avoided. A set of several components shall be arranged in such a way that an air flow through the circulation is achieved from all sides in the best way possible. Ensure that the components cannot move during the whole duration of the test. Attention should be paid to a reasonable product loading factor.

The small chamber shall be closed or locked immediately after inserting the components.

9.3.7 Conditioning and air sampling at 65 °C — Phase 5

The test shall be started immediately after closing the small chamber either by starting an automatized test programme or by switching to the test conditions manually. The climate parameters and sampling procedures of the individual test phases are summarized in Table 1.

The exhaust air, at the small chamber outlet, shall be used for sampling, although separate sampling ports in the small chamber can be used. The sum of sampling air flows should be less than 80 % of the supply air flow to the small chamber to avoid underpressure. The taking of the air sampling for the VOCs and carbonyl compounds with a sampling duration of 30 min is described in the respective air analytical determination methods.

Table 1 — Test procedure by phase

Phases	Time h:min	Temperature target °C	Air change target 1/h	Procedure
1		Max. (230)	Max.	Preheat the chamber (e.g. overnight)
2		65	Max.	Set temperature to 65 °C and allow to chamber to equilibrate at 65 °C
3	0:00	65	0,40	Measure the background concentration for 30 min
4	0:30	65	0,40	Insert sample
5	4:30	65	0,40	Start air sampling

Air sampling duration for concentration measurements depends on the analytical methods to be used and where appropriate duplicate air samples shall be taken.

NOTE Earlier sampling times can be applied if more volatile compounds are of interest.

Additional samples for the determination of such compounds as *N*-nitrosamines, isocyanates, and amines can be taken. Also an additional conditioning phase and measurements at elevated temperatures can be subsequently added to the procedure. The additional samples and measurements, e.g. FID, should be documented in the report.

9.3.8 End of testing

An emission test ends upon deactivation of the climate-regulating system or upon the opening of the emissions test room. Directly upon conclusion of the emissions test, the test room shall be subjected to a cleaning operation (see 9.2).

10 Calculation of emission rate

Area or mass-specific emission rates (q_A or q_m) can be derived from small chamber air concentrations using the conventional calculations described in ISO 16000-9 and ISO 16000-10 and as reproduced in the following.

At a given test condition, γ_X depends on the area-specific emission rate of the test specimen and the air flow rate through the emission test chamber. For individual VOCs, the compounds found both in the material and in the background shall be subtracted compound by compound. For TVOCs, the measured background shall be subtracted. The relation between γ_X , the area-specific emission rate (q_A) and the area-specific air flow rate (q) of the emission test chamber can be expressed as:

$$\gamma_X = q_A \frac{L_A}{n} = \frac{q_A}{q} \quad (3)$$

at time t . Formula (3) shows that the area-specific air flow rate, q , equals the n/L_A ratio. For a given product tested under given emission test chamber conditions, the concentration of VOC_X depends on the area-specific air flow rate.

The measured concentration, γ_X , of a VOC in the outlet air from the emission test chamber shall be converted to an area-specific emission rate, $q_A \gamma_X$ is the mean concentration of a VOC_X calculated from duplicate air samples as described in 9.3.7.

$$q_A = \gamma_X q \quad (4)$$

at time t . The result shall be related to the time of the emission measurement after placing the test specimen in the emission test chamber and may be reported quantitatively as the area-specific emission rate, of individual VOCs and/or TVOCs according to the objective of the test.

The sum of emitted compounds, TVOCs, should be regarded only as a factor specific to the product studied and only to be used for comparison of products with similar target VOC profiles.

The VOC mass concentration (ρ) in air is calculated as described in ISO 16000-6:2011, Clause 11 or ISO 16000-3:2011, Clause 10, using the correction for 23 °C and 101,3 kPa.

11 Test report

The following is a guide to the details required in a test report if the small 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 part of ISO 12219 (ISO 12219-4:2013),
 - reference to the appropriate in-house protocol or detailed description of the equipment and methods used;

- b) sample and 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) and test specimen preparation,
 - conditions of storage,
 - method of preparation, including thickness and compound;
- d) experimental conditions and procedures:
 - small chamber conditions (temperature, air/gas flow, humidity (if appropriate)),
 - test specimen area and means of application (for liquid products, e.g. paint, describe sample compound and coating procedure, paint thickness),
 - sampling of emitted compounds (adsorbent used, volume sampled, sampling duration and times after introduction into the small chamber),
 - analytical conditions used [i.e. thermal desorption parameters, gas chromatography (GC) column selected, GC-MS, where MS is mass spectrometry, 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 VOCs and/or TVOCs (if required), at the times of gas sampling;
- g) quality assurance/quality control:
 - small chamber background concentrations of target volatile organic compounds,
 - recovery data of a known mass of toluene and *n*-dodecane by injection as described in 7.3),
 - results of duplicate sampling/analysis,
 - quality of the environmental variables [e.g. temperature, air or gas selected, air or gas flow, air or gas change rate, humidity (if appropriate)].

12 Quality assurance/quality control (QA/QC)

An appropriate level of quality control shall be employed in accordance with ISO 16000-3 and ISO 16000-6 including the following.

- Chamber blanks are prepared according to 9.3.5.
- The chamber blank level is acceptable if the mass of target compounds are no greater than 10 % of the typical target compounds measured.
- Desorption efficiency of VOCs or carbonyl compounds should be checked in accordance with ISO 16000-3 and ISO 16000-6.

- The collection efficiency can be controlled by using back-up tubes or taking samples of volumes less than the safe sampling volume.
- Repeatability of the air sampling and analytical method shall be determined. A coefficient of variation $\leq 15\%$ (determined as specified in ISO 16000-3 and ISO 16000-6) from the duplicate measurements should be reached.

NOTE The repeatability of the emission test can be influenced by the inhomogeneity of the material under test.

- The recovery of C_6 to C_{16} hydrocarbons of the sorbent tubes shall be 95 % (ISO 16000-6) or better and the recovery of C_6 to C_{16} hydrocarbons should ideally be greater than 80 % for the chamber (see 7.3).
- Documentation illustrating traceable calibrations for temperature, humidity, and flow measurements.

Bibliography

- [1] ASTM D5116, *Standard guide for small-scale environmental chamber determinations of organic emissions from indoor materials/products*
- [2] VDA 276, *Bestimmung organischer Emissionen aus Bauteilen für den Kfz-Innenraum mit einer 1 m³-Prüfkammer* [Determination of organic substances as emitted from automotive interior products using a 1 m test cabinet]

Documents on VOCs cited informatively

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

