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Interior air of road vehicles —

Part 6: Method for the determination of the emissions of semi-volatile organic compounds from vehicle interior parts and materials at higher temperature — Small chamber method

Air intérieur des véhicules routiers —

Partie 6: Méthode pour la détermination des émissions de composés organiques semi-volatils des parties et matériaux intérieurs des véhicules à des températures élevées — Méthode de la petit chambre

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Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html .

The committee responsible for this document is Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 6, *Indoor air*.

A list of all the parts in the ISO 12219 can be found on the ISO website.

Introduction

Volatile and semi-volatile organic compounds (VOCs and SVOCs) 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 emission chambers or bags that have sufficient volume to house the complete assembly (typically ≥ 1 m^3). The performance of such tests may take several hours or even days, depending on specified equilibration times and the requirements of the relevant test protocol.

This document outlines a screening method for measuring the types and levels of VOCs and SVOCs in vehicle trim components under controlled conditions using a small emission test chamber (small chamber). The described screening method can be used to investigate the emissions of car interior trim under conditions of real use where elevated temperatures are prevailing in the cabin of road vehicles. For this purpose, tests are performed at 65 °C and 100 °C. ISO 12219-6 describes requirements for a small chamber and a test protocol. Measurements are carried out according to ISO 16000-6 (VOCs).

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

This document is based on VDA 276[2] and correlates to ISO 16000-9.

Besides the ISO 12219-series, there are parts of ISO 16000 which deal with the measurements of vapour-phase organic chemicals and vapour-phase chemical emissions:

- *Part 3: Determination of formaldehyde and other carbonyl compounds in indoor air and test chamber air — Active sampling method*
- *Part 5: Sampling strategy for volatile organic compounds (VOCs)*
- *Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA® sorbent, thermal desorption and gas-chromatography using MS or MS-FID*
- *Part 9: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test chamber method*
- *Part 10: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test cell method*
- *Part 11: Determination of the emission of volatile organic compounds from building products and furnishing — Sampling, storage of samples and preparation of test specimens*
- *Part 24: Performance test for evaluating the reduction of volatile organic compound (except formaldehyde) concentrations by sorptive building materials*
- *Part 25: Determination of the emission of semi-volatile organic compounds for building products — Micro chamber method*

Interior air of road vehicles —

Part 6:
Method for the determination of the emissions of semi-volatile organic compounds from vehicle interior parts and materials at higher temperature — Small chamber method

WARNING — This method is unsuitable for materials that are not stable at 100 °C in air. Application of this document for thermally unstable materials could lead to irreversible contamination of the test equipment.

1 Scope

This document describes a qualitative and quantitative analytical method for vapour-phase organic compounds released from car trim materials under simulated real use conditions, i.e. a vehicle is parked for several hours in direct sunlight. Under these conditions, some interior parts and materials reach higher temperatures than 65 °C (ISO 12219-4), e.g. a dashboard can reach temperatures up to 120 °C. This document can be implemented as an optional addition to ISO 12219-4 so that VOC, volatile carbonyl and SVOC testing can all be completed within one day. This part has been added to gain insight into the emission behaviour and emission potential of selected vehicle interior parts and materials exposed to higher temperatures. (By convention, 100 °C is set as the higher temperature.)

The test is performed in small emission test chambers (small chambers). These small chambers are intended to provide a transfer function for vehicle level emissions. This method is intended for evaluating new car interior trim components but can, in principle, be applied to used car components.

The specified analytical procedure for SVOCs and semi-volatile carbonyls is ISO 16000-6.

This document is complementary to existing standards[1],[2] and provides third party test laboratories and manufacturing industry with an approach for

- identifying the effect of real use conditions on specific VOC and SVOC emissions data,
- comparing emissions from various assemblies with regards to specific VOC and SVOC emissions,
- evaluating and sorting specific assemblies regarding specific VOC and SVOC emissions data,
- providing specific VOC and SVOC emissions data to develop and verify a correlation between component level methods and in vehicle air quality and
- evaluating prototype, “low-emission” assemblies during development.

The method described can be exclusively performed as a high temperature test or it can be performed in combination with the determination of VOCs at 65 °C in one run, which is described in ISO 12219-4.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 12219-4, *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*

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 12219-4 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
semi-volatile organic compound
SVOC
organic compound whose boiling point is in the range from (240 °C to 260 °C) to (380 °C to 400 °C)

Note 1 to entry: This classification has been defined by the World Health Organization[3].

Note 2 to entry: Boiling points of some compounds are difficult or impossible to determine because they decompose before they boil at atmospheric pressure. Vapour pressure is another criterion for classification of compound volatility that can be used for classification of organic chemicals. SVOCs have vapour pressures between 10^{-2} mPa and 10 Pa.

[SOURCE: ISO 16000-25:2011, 3.16]

3.2
target semi-volatile organic compound
product-specific semi-volatile organic compound

4 Symbols

Symbol	Meaning	Unit
t	time	[h]
q	area specific air flow rate $q = n/L_A$	[$m^3 \cdot m^{-2} \cdot h^{-1}$]
q_A	emission rate per unit area	[$\mu g \cdot m^{-2} \cdot h^{-1}$]
q_m	emission rate per unit mass	[$\mu g \cdot kg^{-1} \cdot h^{-1}$]
n	air change rate	[h^{-1}]
n_L	specific leak rate	[h^{-1}]
L_A	surface loading of chamber	[$m^2 \cdot m^{-3}$]
V	air flow rate entering the small chamber	[$m^3 \cdot h^{-1}$]

5 Principle

A vehicle interior trim component or material sample, referred to as a test specimen, is inserted into a small chamber (0,5 m^3 to 4,0 m^3) and kept under controlled conditions of temperature, humidity and air change rate (air flow rate). The air inside the chamber is thoroughly mixed at all times so that the concentration of any organic substances emitted by the test specimen is uniform – both within the chamber and in the flow of air exhausting from the chamber.

The air exhausting from the chamber is sampled for semi-volatile organic compounds at selected times. Chemical analysis of these samples allows the chamber air concentration and specific emission rates from the test specimen to be determined.

6 Emission test bed preparation

6.1 General

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

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

General guidelines regarding suitable construction materials and configurations of test apparatus are given below. Recommendations for continuous monitoring of the chamber air for quality assurance purposes are also 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 m^3 to 4,0 m^3. A typical standard small chamber has a volume of 1 m^3 ± 0,05 m^3. The chamber volume shall be specified in the test report. Inside the chamber, there is a device for mixing the air and a stand to guarantee positioning of the component (see 6.2.2) 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 in the form of a flow chart is shown in Figure 1.

6.2.2 Materials

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

The small chamber method requires the following key components.

6.2.2.1 Airtight small chamber apparatus.

6.2.2.2 Appropriate wall surfaces and rack. The wall surfaces of the small chamber and the rack for supporting the test specimen should be made of electropolished high-quality steel. When testing materials or components that are not compatible with hot stainless steel (e.g. test specimens which emit odorous reactive substances such as some sulfur-containing compounds), the chamber shall be constructed of inert materials that don’t emit or absorb organic vapours.

6.2.2.3 Heating mechanism and temperature control system.

6.2.2.4 Sampling line, constructed of an inert, non-emitting and non-adsorbing material which is 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. It is strongly recommended to heat up the sampling line to 120 °C to prevent condensation.

6.2.2.5 Clean air supply and humidification system.

6.2.2.6 Appropriate monitoring and control systems (to ensure that the test is carried out according to specified conditions).

6.2.2.7 Appropriate vapour sampling tubes are also required.

6.2.2.8 Appropriate sealing materials (e.g. gaskets or O-rings). Any sealing materials used for sealing the doors or lids of the small chambers, shall be compatible with high temperatures and exhibit low emission and low absorption properties even at elevated temperatures. They shall not contribute significantly to the background vapour concentration. The O-rings or gaskets should be easily removed to facilitate cleaning or replacement (see 6.2.5). Surfaces 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 sample loss, any leakage shall be either

- less than 0,1 % of the volume of the small chamber per minute or
- less than 5 % of the incoming air (delivery air/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.

6.2.4 Air mixing

This method relies on the air inside the chamber being thoroughly mixed. A suitable device for mixing the air is required, 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 Suitable equipment for measuring air velocity includes hot wire or film anemometers calibrated in the range 0,1 m/s to 0,5 m/s.

6.2.5 Cleaning

The chamber shall be easy to clean – mechanically and thermally, including ready access to seals and gaskets. See 7.3 for performance criteria relating to background levels and see 9.2 and 9.3.4 for information on cleaning procedures.

6.3 Small chamber temperature control

The chamber temperature shall be precisely controlled because there is a strong link between temperature and the chemical emission rate. The chamber shall be able to maintain a given temperature 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 a very constant relative humidity.

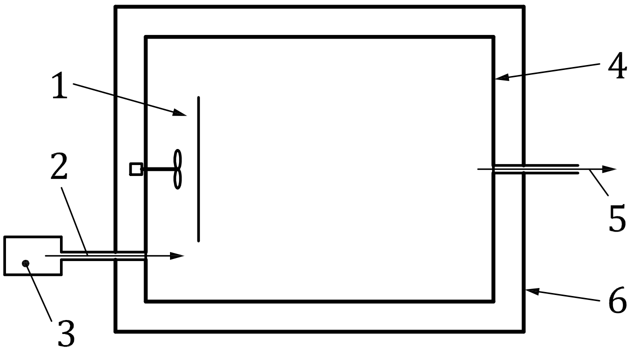
6.5 Clean air supply

Tests with air change or air renewal and air sampling require a flow of clean air. An air change or air renewal of 0,4 h^{-1}, under the test conditions (65 °C, ambient pressure), shall be set with an accuracy of ± 5 %. When regulating the supply air flow, the temperature and pressure conditions at which the mass flow controller was calibrated shall be taken into account.

To prevent localized cooling of the chamber and material/component near the air inlet, the inlet air supply is normally heated up in the heating jacket between the small chamber and the outer test cabinet.

Before the start of a test, the sum of the concentrations of volatile and semi-volatile organic hydrocarbon combinations in the supply air shall not be more than 50 µg m^{-3}; for individual substances, the concentration shall not be more than 5 µg m^{-3}.

Particulate matter in the clean air supply shall be reduced through a particle filter in order to avoid the adsorption of compounds.



Key

1	air circulation	4	small chamber
2	controlled humidified flow	5	heated 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 within a small chamber; therefore, regular and thorough inspections of the whole system are required. Since these errors can affect a test result, the inspection of 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 below.

7.2 Airtightness

7.2.1 General

The airtightness of the small chamber is checked at an excess pressure of 1 000 Pa by measuring the pressure drop over a time period of 2 h. The sensitiveness of the pressure pick-off/pressure transducer should be smaller than 100 Pa with an accuracy of ± 5 %. The average value of the specific leak rate, n_L, referring to the small chamber volume in thousandth per hour during this period is calculated:

$$n_\mathrm{L} = \frac{1\,000}{t}\left(\frac{p_1}{p_2} - 1\right) \tag{1}$$

where

p_1 is the absolute pressure in the small chamber at the start of the test, in Pa;

p_2 is the absolute pressure in the small chamber at the end of the test, in Pa;

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, $n_{L,\%}$, referring to the small chamber in percentage per hour, can be determined according to Formula (2). The tightness is determined at a small chamber temperature of 65 °C.

$$n_{\mathrm{L},\%} = \frac{100}{t}\frac{\Delta p}{p}\frac{\ln 2}{t_{1/2}} \tag{2}$$

where

$\frac{\Delta p}{p}$ is the relative excess pressure compared to the ambient pressure;

$t_{1/2}$ is the period of time elapsed until the decrease of the excess pressure to half of the initial value, in hours.

7.2.3 Alternative procedure 2

The small chamber shall be airtight if the leakage is less than 5 % of the supply air flow. The tightness is determined at a small chamber temperature of 65 °C.

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 organic compound(s) 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, dihexylphthalate, and surrogate polar compounds, e.g. 2-ethylhexanol.

The addition of a known mass of a compound into the small chamber can be performed either via a syringe injected into a substrate or through 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. If there is a set of several substrates, this set shall be arranged in such a way that air flow through the chamber 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 analyte recovery shall be carried out under typical test conditions, e.g. typical time, 65 °C, and an exchange rate of 0,4 h^{-1}.

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 organic compounds can 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 highly dependent on the type of compound emitted. Additional recovery tests using selected organic compounds 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 or tested and reported for the actual compound of interest.

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 parameters shall be monitored and recorded continuously with instruments meeting the following accuracy:

—	temperature	± 1 °C;
—	relative air humidity	± 0,5 % RH at 65 °C or ± 5 % RH at 23 °C;
—	standard air flow rate	± 5 %.

The air speed should be regularly checked (with a minimum frequency of every 12 months and a resulting accuracy of ± 0,1 m/s). A hot-wire anemometer is typically used for this 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) by 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 shall be checked prior to every test; see 9.3.6.

A screening procedure, for example using Tenax® sampling tubes[1)] with thermal desorption and GCMS analysis (as described in ISO 16000-6), with a sensitivity of at least 1 µg m^{-3} for individual substances, shall be used for a quantitative blank value observation.

[1)] Tenax TA® is the trade name of a product manufactured by Buchem, Inc. It is unique and has a sole manufacturer; however, it is available from many suppliers. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

The sum concentration of VOC that has been determined with this procedure should be below 50 µg m^{-3} and the individual concentration of any of the substances should not exceed 5 µg m^{-3}.

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 ± 1 °C or ± 0,5 % at 65 °C, respectively.

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

8 Test specimen

8.1 General

When determining (semi-)volatile organic emissions, the conditions the test specimen 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 way test specimens are treated before testing as much as possible.

The test specimens 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 test specimen

If it is necessary to partially dismantle or modify a component or material in any way in order to test it, this shall be documented.

Test specimens should be protected from contamination as much as possible; however, it is inevitable that they will absorb or adsorb some substances from the environment. The past history of the component before the test shall be documented as completely as possible.

8.3 Packaging, transport and storage of the test specimen

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. The container shall be filled properly in order to avoid any uncontrolled head-space.

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.

Storage can affect the emission properties due to aging of the component. Therefore, the storage time should be no longer than 3 months.

To reduce the influence of aging effects, it is recommended to keep the storage time of the component as short as possible.

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 extreme climatic conditions.

The test temperature in the first conditioning phase according to ISO 12219-4 is 65 °C with a humidity of the incoming air of approximately 5 % RH (this corresponds to 45 % RH at 23 °C). The air change rate or air renewal is 0,4 h^{-1}.

The test temperature in the second conditioning phase is 100 °C.

By default, air samples are taken to determine the concentrations of semi-volatile organic compounds (SVOC). In special cases, additional samples for the determination of such compounds as carbonyls, N-nitrosamines, isocyanates, and amines, can be taken. The test procedure shall be documented in the report.

Determine the test sample concentrations of SVOCs by selecting the appropriate sampling media, e.g. a sorbent tube for SVOCs (see ISO 16000-6 for more details).

The specified analytical procedure is valid for the determination of (semi-)volatile organic compounds (SVOCs) ranging in concentration from sub- µg m^{-3} to several mg m^{-3}.

9.2 Cleaning and purification

All exposed inner surfaces of the small chamber shall be clean before the start of a component test. If there are any background concentrations, they shall 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 rinsing with distilled water or by using an appropriate solvent and drying thoroughly.

Alternatively or additionally, the small chamber assembly can be heated for cleaning. Raise the empty, sealed small chamber to a temperature of 180 °C or up to 230 °C in a fast flow of pure gas until background artefacts are reduced to negligible levels (see 6.5 and 9.3.4).

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

9.3.1 General

Actual testing extends from unpacking the specimen from its air-tight packaging to removal of the specimen from the small chamber upon conclusion of all conditioning and sampling operations.

9.3.2 Preconditioning the sample prior to test

The test specimens are removed from the packaging 1 week before the test (the packaging materials shall be removed in the case of components, if applicable) and stored at 23 °C ± 2 °C and 50 % RH ± 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.

9.3.3 Preparation

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

9.3.4 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.

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

9.3.6 Background concentration sampling — Phase 3

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

9.3.7 Inserting the test specimen — 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 and further cannot significantly alter the surface air velocity. Attention should be paid to a reasonable product loading factor.

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

9.3.8 Conditioning at 65 °C — Phase 5 according to ISO 12219-4

The test has to be started immediately after closing the small chamber either by starting an automated 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.

There is no air sampling in Phase 5 unless there is the intention to sample VOC and/or carbonyls in accordance to ISO 12219-4 (see Clause 10) or special compounds. So, additional samples for the determination of e.g. N-nitrosamines, isocyanates, amines, etc. can be taken. 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 incoming air flow to the small chamber to avoid underpressure. The taking of the air sampling for the VOCs and carbonyls with a sampling duration of 30 min is described in the respective air analytical determination methods ISO 16000-3 and ISO 16000-6.

9.3.9 Heating up and conditioning at 100 °C — Phase 6

After 5 h, the humidification of the incoming air/delivery air/supply air is switched off and the small chamber is heated up to 100 °C 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 conditioning of Phase 6 is performed for 3 h.

9.3.10 Air sampling at 100 °C — Phase 7

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 incoming air flow to the small chamber to avoid underpressure. The air sampling for the SVOCs with a sampling duration of 30 min is described in ISO 16000-6. The recovery of SVOCs (particularly those boiling above n-C_{22}) is facilitated by inserting a short (5 mm to 10 mm) bed of loosely packed quartz wool in front of the adsorbent (see ISO 16000-6:2011, D.4.1).

To prevent condensation of substances in the walls of the sampling ports, it is strongly recommended to heat up the sampling ports to 120 °C.

Care should be taken that the air sampling tubes will not become too hot which could result in a breakthrough of substances and / or damage of the adsorbing material. If necessary, both effects can be minimised by using glass tubes. Annex A shows infrared pictures of the sampling tubes during 65 °C and 100 °C sampling.

It is advisable to carry out replicate sampling.

9.3.11 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).

Table 1 — Test procedure by phase

Phase	Time h:min	Temperature target ° C	Air change target h^{-1}	Dew point bath temperature / air supply moisture[a] °C / % RH	Procedure
1		max (230)	maximum	off	Cleaning the chamber (e.g. overnight)
2		65	maximum	10,4 / 5,0	Set temperature to 65 °C and allow the chamber to equilibrate at 65 °C
3	0:00	65	0,40[b]	10,4 / 5,0	Measure the background concentration for 30 min
4	0:30	65	0,40	10,4 / 5,0	Insert test specimen
5a	4:30	65	0,40	10,4 / 5,0	Start VOC air sampling[d]
5b	5:00	65	0,40	10,4 / 5,0	End of air sampling[d]
6	5:00	100	0,44[c]	off	Start heating up to 100 °C[e]
7a	8:00	100	0,44[c]	off	Start SVOC air sampling
7b	8:30	100	0,44[c]	off	End of air sampling

[a] Dew point bath temperature and relative humidity are referenced to a temperature of 65 °C and a pressure of 101,325 kPa.

[b] In order to set the established air change rate of 0,40 h^{-1}, e.g. in a 1,00 m^3 test chamber, a supply air flow of 323 l h^{-1} (at standard temperature and pressure of 0 °C and 101,325 kPa, respectively) shall be set with an accuracy of ± 5 %.

[c] The air flow rate with reference to standard temperature and pressure entering the small chamber is the same for both operating modes (65 °C and 100 °C). Larger air change rates at elevated temperatures merely result from thermal effects according to the ideal gas law: $V_2=(T_2/T_1)\times V_2$ (with T_1 = 338 K and T_2 = 373 K).

[d] Optional sampling period. Only required if analysis of VOCs is anticipated (see Clause 10).

[e] The final temperature of 100 °C should be reached within 1 h at the most.

10 Determination of VOCs at 65 °C and SVOCs at 100 °C in one run

The determination of VOCs at 65 °C and SVOCs at 100 °C in one run is possible. Measure the VOCs at a temperature of 65 °C as described in ISO 12219-4. Follow this by a test phase at 100 °C for the SVOC sampling. After air sampling at 65 °C, change the sampling tubes and use new sampling tubes for the air sampling at 100°C. A typical temperature profile is shown in Annex A.

11 Calculation of the 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 text.

At a given test condition, the mass concentration of substance X (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 SVOCs, the compounds found both in the material and in the background shall be subtracted compound by compound. 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:

$$\gamma_{\mathrm{x}} = q_A \frac{L_A}{n} = \frac{q_A}{q} \tag{3}$$

where

L_A is the surface loading of the chamber, in m^2 m^{-3};

n is the air change rate, in h^{-1}.

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, γ_x depends on the area-specific air flow rate.

The measured concentration, γ_{x}, of a SVOC in the outlet air from the emission test chamber shall be converted to an area-specific emission rate.

$$q_A = \gamma_{\mathrm{x}} q \tag{4}$$

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 SVOCs according to the objective of the test.

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

12 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:

   1) laboratory details (if appropriate);

   2) name of the responsible person;

   3) reference to this practice;

   4) reference to the appropriate in-house protocol or detailed description of the equipment and methods used.

b) Test specimen description:

   1) type and batch number of the product (and brand name, if appropriate);

   2) test specimen selection process (e.g. random);

   3) product history (e.g. date of production or date of arrival at the test laboratory, if appropriate);

   4) description of the packaging, if appropriate.

c) Test specimen preparation:

   1) date and time of unpacking (if appropriate) and test specimen preparation;

   2) conditions of storage and any deviations (see 9.3.2);

   3) method of preparation, including thickness and compound.

d) Experimental conditions and procedures:

   1) volume of the small chamber (see 6.2.1);

   2) small chamber conditions [temperature, air/gas flow, humidity (if appropriate)];

   3) test specimen area and means of application (for liquid products, e.g. paint, describe compound and coating procedure, paint thickness);

   4) sampling of emitted compounds (adsorbent used, volume sampled, sampling duration and sampling times after introduction into the small chamber);

   5) analytical conditions used (i.e. thermal desorption parameters, GC column selected, GC-MS conditions, etc.);

   6) any deviations from the processing of the test specimens (see 8.1);

   7) test procedure used (see 9.1)

e) Data analysis:

   1) description of the method used to calculate the specific emission rate or vapour concentration data of VOCs or SVOCs.

f) Results:

   1) specific emission rates each test specimen, for individual SVOCs and/or TVOC (if required), at the times of gas sampling.

g) Quality assurance/ quality control:

   1) small chamber background concentrations of semi-volatile organic compounds;

   2) recovery data (to evaluate the overall performance);

   3) results of duplicate sampling/analysis; quality of the environmental variables [e.g. temperature, air or gas selected, air/gas flow, air/gas change rate, humidity (if appropriate)].

13 Quality assurance/quality control (QA/QC)

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

- chamber blanks should be prepared according to 9.3.6;
- the chamber blank level is acceptable if the mass of each individual compound is no greater than 10 % of the level of that compound that would typically be collected from that type of sample under the given test conditions;
- the desorption efficiency of SVOCs should be checked as described in ISO 16000-6;
- the collection efficiency can be controlled by using back-up tubes or taking samples of different sampling volumes less than the safe sampling volume;
- the repeatability of the air sampling and analytical method shall be determined; a coefficient of variation ≤ 15 % (determined as specified in ISO 16000-6) from the duplicate measurements should be reached;

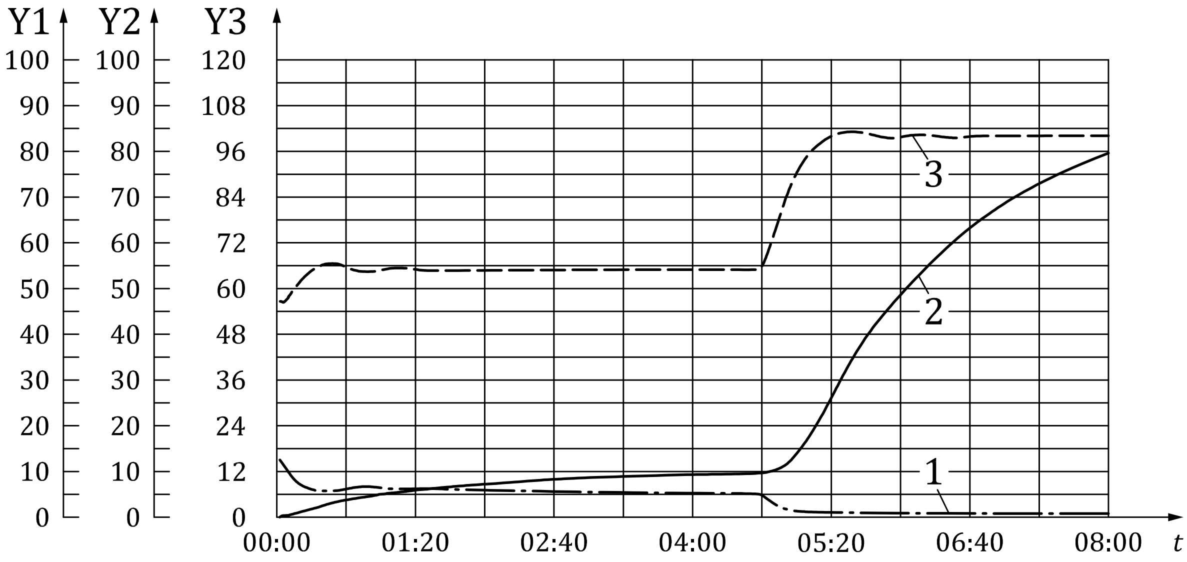
  NOTE The repeatability of the emission test will be influenced by the inhomogeneity of the material under test.

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

Annex A
(informative)

Typical test conditions and example for the experimental setup

The temperature profile of the individual test phases for the determination of VOCs at 65 °C (according to ISO 12219-4) and SVOCs at 100 °C in one run is shown in Figure A.1.



Key

t time, in h

Y1 relative humidity, in %

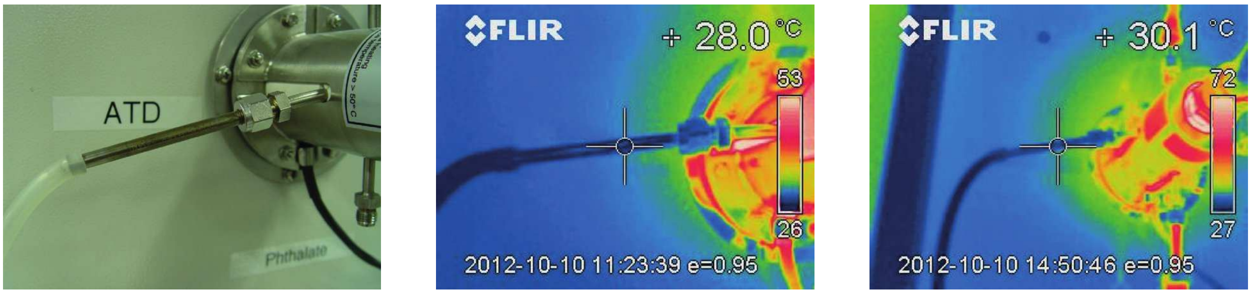
Y2 FID signal, in ppm

Y3 temperature, in °C

Grey shaded areas indicate time periods of air sampling.

Figure A.1 — Operating parameters of the standard emission test cycle for the determination of volatile and semi-volatile compounds from components using a 1 m^3 test chamber

The following figures depict the temperature distribution at the sampling unit when the chamber is operated at 65 °C and 100 °C, respectively. As shown in Figures A.2 b) and c), the temperature in the stainless steel tube filled with Tenax® used for the sampling of VOCs is only slightly elevated by about 2 °C while operating the chamber at 100 °C compared to the 65 °C operation mode.



a) Picture of the experimental setup

b) Thermogram of the experimental setup while operating the chamber at 65 °C and after a sampling period of 10 minutes

c) Thermogram of the experimental setup while operating the chamber at 100 °C and after a sampling period of 10 minutes

In subfigures a) and b), the cross marks the temperature measurement position.

Figure A.2 — Stainless steel tube filled with Tenax®

Bibliography

[1] ASTM D 5116-10, *Standard Guide for Small-Scale Environmental Chamber Determinations of Organic Emissions From Indoor Materials/Products*

[2] VDA 276:2005, *Bestimmung organischer Emissionen aus Bauteilen für den Kfz-Innenraum mit einer 1 m3 Prüfkammer. Teil 1: Standard Emissionsprüfung; Determination of organic substances as emitted from automotive interior products using a 1 m3 test cabinet – Part 1: Standard-Emission test*

[3] World Health Organization. Indoor air quality: Organic pollutants. Copenhagen: WHO Regional Office for Europe, 1989. 70 p. (EURO Reports and Studies No. 111.) Available (viewed 2011-11-16) at: http://whqlibdoc.who.int/euro/r&s/EURO_R&S_111.pdf

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[5] ISO 16000-9, *Indoor air — Part 9: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test chamber method*

[6] ISO 16000-10, *Indoor air — Part 10: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test cell method*

[7] 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*

[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*

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