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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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Foreword

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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 $>1\ m^3$). 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,[5] ISO 16000-6, ISO 16000-9,[6] ISO 16000-10,[7] ISO 16000-11,[8] ISO 16000-24,[9] ISO 16000-25,[10] as well as ISO 16017-1[11] and ISO 16017-2,[12] 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;[11] 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[1] and ISO 16000-9[6]) or test cells (ISO 16000-10[7]). 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® 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^3 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 ±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 (±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®[1)] 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[11] and Annex F for more information.

NOTE 1 Refer to ISO 16000-6:2011, Annex D or ISO 16017-1[11] for guidance on sorbent selection if VOCs eluting before n-C_6 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.

1) Tenax TA® 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 °C ± 2 °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 µg/m^3 for individual VOC and below 50 µg/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[8] 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).