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Date: 30 June 2023

Origin: International

Latest date for receipt of comments: 23 August 2023

Project No. 2020/03482

Responsible committee: EH/2/5 Emissions to internal environments

Interested committees:

Title: Draft BS ISO 12219-11 Interior air of road vehicles. Part 11: Thermal desorption analysis of organic emissions for the characterization on non-metallic materials for vehicles.

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Introduction

This draft standard is based on national and international discussions. Your comments on this draft are invited and will assist in the preparation of the consequent standard.

For international standards, comments will be reviewed by the relevant UK national committee before sending the consensus UK vote and comments to the international committee, which will then decide appropriate action. If the international standard is approved, it is usual for the text to be published as a British Standard.

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Submission of Comments

- **Annotated drafts are not acceptable and will be rejected.**
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Template for comments and secretariat observations

Date: xx/xx/20xx	Document: ISO/DIS xxxx
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1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/ Figure/Table/Note	Type of comment	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	

DRAFT INTERNATIONAL STANDARD

ISO/DIS 12219-11

ISO/TC 146/SC 6

Secretariat: DIN

Voting begins on:
2023-06-30

Voting terminates on:
2023-09-22

Interior air of road vehicles —

Part 11:

Thermal desorption analysis of organic emissions for the characterization on non-metallic materials for vehicles

ICS: 13.040.20; 43.020

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Reference number
ISO/DIS 12219-11:2023(E)

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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

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For an explanation of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 6, *Indoor air*.

A list of all parts in the ISO 16000 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document describes an analytical method to determine emissions from non-metallic materials used for molded parts in motor vehicles.

The materials are characterized in terms of the type and quantity of organic substances that can be outgassed from them. For this purpose, two semi-quantitative sum values are determined, which allow an estimation of the emissions of volatile organic compounds (VOC value) and the proportion of condensable substances (fog value). Furthermore, individual substances of the emission are determined. During the analysis, the samples are thermally extracted, the emissions are separated by gas chromatography and detected by mass spectrometry. The present test method provides values that are valid only for conditions described here.

Interior air of road vehicles —

Part 11:

Thermal desorption analysis of organic emissions for the characterization on non-metallic materials for vehicles

1 Scope

The analytical method is intended for the determination of emissions from non-metallic materials for moulded parts in motor vehicles such as:

Textiles, carpets, adhesives, sealing compounds, forms, leather, plastic parts, films and sheets, paints or material combinations.

The materials are characterized with regard to the type and quantity of the organic substances outgassed from them.

In this method two semi-quantitative cumulative values are determined which allow the emission of volatile organic compounds (VOC value) and the portion of condensable substances (FOG value) to be estimated. Furthermore, single substance emissions are determined.

During the analysis, the samples are thermally extracted, and the emissions are separated by gas chromatography and detected by mass spectrometry.

This test method provides values which are only valid under the conditions described in this document. The results which can be achieved using this method are not appropriate.

For making further estimations of any kind of the health effects of the emitted substances, to be used in any form as a basis for the estimation of concentrations which might be found in the interior of a complete vehicle in stationary condition, while driving or in conditions similar to driving.

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.

3 Terms and definitions

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

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

VOC value

total of the readily volatile to medium volatile substances. It is calculated as toluene equivalent. The method described in this document allows substances in the boiling/elution range from the beginning of the chromatogram up to n-Pentacosane (C25) to be determined and analysed.

Note 1 to entry: It is known that the retention area between C1 and C4 cannot be completely quantified.

Note 2 to entry: It can be assumed that these substances can be detected when analysing the air of the vehicle interior. For the analysis, the sample is heated at 90°C for 30 min. The VOC value is determined by two measurements. The higher value of the measurements is indicated as the result.

3.2

FOG value

total of substances with low volatility which elute from the retention time of n-Tetradecane (inclusive). It is calculated as hexadecane equivalent. Substances in the boiling range of n-Alkanes C14 to C32 are determined and analysed.

3.3

Retention index

Index calculated with a series of n-alkanes

3.4

Sample

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

[SOURCE: ISO 12219-4:2013; 3.15]

3.5

Specimen

article prepared from a sample

[SOURCE: SOURCEISO 15906:2007]

4 Symbols and abbreviated terms

The symbol and the abbreviated terms used in this document are given below.

VOC	volatile organic compound
FOG	low volatile “fogging” compounds

5 Sampling and storage

The age of the sample to be analysed should be defined. A representative sample should be taken for examination. Contamination should be prevented. Each sample should be welded within 8 hours of its productions airtight in aluminium composite foils.

Alternatively, the sample should be fully wrapped twice in thick aluminium foil (30 µm) so that it is airtight, folding the edges several times. The sample should then be sealed in a polyethylene bag and sent to the laboratory. The sample should be delivered to the laboratory without delay. During transportation and until unpacking of the sample, a temperature of 23 °C should not be exceeded. Immediately before commencement of the analysis, the samples should be stored uncovered at room temperature (23° max) for 7 days. Any deviation from this procedure should be documented.

For further information see Annexes A.1 and A.2.

6 Apparatus

- Directly coupled thermal desorption / gas chromatography system
- Gas chromatograph (electronic control of pneumatics is recommended)
- Cryofocussing with programmable temperature and with split
- Mass-selective detector, mass spectral database
- Desorption tubes with inert surface, internal diameter 4-5 mm

- Chromatographic quartz glass capillary separation column with dimensions 50 m x 0,32 mm x 0,52 µm; stationary phase: 5% phenyl-methyl-siloxane
- Software for equipment control
- Software for analysis of chromatograms
- The recovery rates of the single substances (calculated as toluene equivalent) of the control mixture describe in clause 6.2.1 should be between 60 and 140 % under VOC conditions, whereby the recovery of the toluene should be at least 80 % and no more than 120 %.
- It should be possible to fall below the following decision limits (DIN 32645, confidence interval: VB=95%):
 - In the VOC run toluene < 0,04 µg, and Icosane (C20) < 0,06 µg
 - In the FOG run n-Alkane C32 < 0,2µg

For the determination of the decision limits, different amounts of the test substances dissolved in methanol (for VOC run) and n-Pentane (for FOG) resp. should be injected into desorption tubes filled with Tenax and desorbed at 280°C. The split characteristics and GC conditions should conform to the VOC/FOG method parameters.

- Parameters for gas chromatography for calibration and control analysis:

For toluene calibration and the analysis of the control mix the same parameters should be set as for the VOC sample run. The only difference is a later start of the data logging to mask out the methanol peak.

Hexadecane calibration is carried out under the GC conditions of the FOG analysis. Compared to the sample runs, the GC runs can be shortened by abandoning the oven temperature program after elution of the calibration substances.

7 Analysis

7.1 General information on thermal desorption analysis

In a thermal desorption analysis (TDS), small volumes of material are heated in a desorber tube in a defined process. The volatile substances emitted during this process are cryofocussed by means of an inert gas stream in the cold trap of an evaporator with programmable temperature.

Following the heating phase, the cold trap is heated up quickly to 280°C. The focussed substances evaporate during this process. They are split in the gas-chromatographic separation column and detected by mass spectrometry.

Calibration with reference substances allows for a semi-quantitative estimation to be carried out, expressed as µg/g².

Toluene for the VOC analysis and n-Hexadecane for the FOG value (see 3.3) are used as quantitative references substances. Based on the mass spectra and retention indices, substances can be assigned to the signal peaks.

7.2 Cleaning of the desorption tubes

All desorption tubes used should be completely free of contamination. New tubes should also be cleaned carefully before initial use by means of an appropriate method. The following procedure is recommended for glass tubes:

To clean them, store the tubes in an alkaline cleaning solution for several hours, preferably overnight, Then rinse thoroughly, first under running hot water for at least one minute, and then rinse again using

deionized water. Then dry the tubes in a drying oven for approx. 45 min at 105°C and store free of contamination (e.g. wrapped airtight in aluminium foil) until used.

7.3 Checking of system

The performance of the equipment system should be checked during the sample series by analysing a control standard solution (see. 7.3.1).

The control standard solution should contain non-polar, polar alkaline and acidic components which would demonstrate a noticeable peak tailing in the event of undesired adsorption effects. In addition, it is possible to determine any loss of material due to leakages. Peaks in close succession such as o-Xylene and n-Nonane are used for checking the separation performance of the chromatography column. Under the chromatographic conditions chosen, these two substance peaks must be almost baseline separated.

All substances contained in this control mixture should be uniquely identified during the search in the mass spectral database.

The performance of the mass-selective detector is to be assured by mass and sensitivity tuning., during which the manufacturer's specifications should be achieved. In addition, an air/water check should be performed to check the overall system for leakages.

The TDS (GC system should also be checked for memory effects by carrying out a blank run with an empty desorption tube at least before each sample series. If negative effects such as strong peak tailing, interfering blank run peaks or significant loss of materials occur, the system should be cleaned. If appropriate, the GC column, glass line, transfer line or seals, for example, should be replaced.

The documentation of the results of the control runs for each sample series within the framework of quality control is recommended (control chart). The following parameters are examples of expedient control variables:

- Peak area characteristics of the control substances
- Calculation of concentrations of control substances as toluene equivalents
- Retention times

7.3.1 Control standard solution

The following substances dissolved in methanol have proved suitable for checking the system (listed in elution sequence under VOC condition):

Table 1 — Control mixture

Retention index ¹	Component	Retention index	Component
670	Benzene	1100	n-Undecane
700	n-Heptane	1110	2,6-Dimethylphenol
766	Toluene	1200	n-Dodecane
800	n-Octane	1300	n-Tridecane
870	p-Xylene	1400	n-Tetradecane
895	o-Xylene	1435	Dicyclohexylamine
900	n-Nonane	1500	n-Pentadecane
1000	n-Decane	1600	n-Hexadecane
1030	2-Ethylhexanol-1	2390	Di-(2-ethylhexyl)-adipate
¹ The retention indices listed here are referenced to n-alkanes and represent approximate values.			

It is possible to use a commercially available certified substance mixture with comparable volatility, polarity and concentration for system control.