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ISO/FDIS 17895

Paints and varnishes — Determination of volatile organic compound (VOC) — Gas- chromatographic method with headspace injection for VOC determination

Peintures et vernis — Détermination de la teneur en composés organiques volatils (COV) — Méthode par chromatographie en phase gazeuse avec injection dans l'espace de tête pour la détermination des COV

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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 document 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).

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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 35, *Paints and varnishes*, Subcommittee SC 16, *Chemical analysis*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 139, *Paints and varnishes*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 17895:2005), which has been technically revised.

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The main changes are as follows:

- the document has been technically revised;
- used methods have been further specified;
- normative references have been updated.

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.

Paints and varnishes — Determination of volatile organic compound (VOC) — Gas-chromatographic method with headspace injection for VOC determination

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of the document, and to determine the applicability of any other restrictions for this purpose.

1 Scope

This document specifies the sampling and testing of low volatile organic compound (VOC) coating materials and their raw materials. In particular, this document specifies a gas-chromatographic method to quantitatively determine the VOC content (i.e. the content of organic compounds with boiling points up to 250 °C) under standard conditions (101,325 kPa). It is applicable to VOC contents between 0,01 % and 0,1 % by mass.

This document does not apply to the determination of the semi-volatile organic compounds (SVOC) content, which is covered in ISO 11890-2.

This document does not apply to volatile organic and volatile inorganic compounds that cannot be determined by gas chromatography.

The procedure for identifying the appropriate method for the determination of VOC content and the SVOC content of coating materials and their raw materials is described in ISO/TR 5601.

2 Normative references

[ISO/FDIS 17895](https://standards.itih.ai/catalog/standards/iso/117b5cdf-0b57-45d6-b3c6-21e39a03b76e/iso-fdis-17895)

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 2811-1, *Paints and varnishes — Determination of density — Part 1: Pycnometer method*

ISO 2811-2, *Paints and varnishes — Determination of density — Part 2: Immersed body (plummet) method*

ISO 2811-3, *Paints and varnishes — Determination of density — Part 3: Oscillation method*

ISO 2811-4, *Paints and varnishes — Determination of density — Part 4: Pressure cup method*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

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

ISO and IEC maintain terminology 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

volatile organic compound

VOC

organic liquid and/or solid that evaporates spontaneously at the prevailing temperature and pressure of the atmosphere with which it is in contact

Note 1 to entry: As to current usage of the term VOC in the field of *coating materials* (3.9), see *volatile organic compound content* (3.3).

Note 2 to entry: Under US government legislation, the term VOC is restricted solely to those compounds that are photochemically active in the atmosphere (see ASTM D3960). Any other compound is then defined as being an exempt compound.

[SOURCE: ISO 4618:2023, 3.266, modified — Note 3 to entry has been removed.]

3.2

semi-volatile organic compound

SVOC

organic liquid and/or solid that evaporates spontaneously but slower in comparison to VOC at the prevailing temperature and pressure of the atmosphere with which it is in contact

Note 1 to entry: Regarding current usage of the term SVOC in the field of *coating materials* (3.9), see *semi-volatile organic compound content* (3.4).

[SOURCE: ISO 4618:2023, 3.227]

3.3

volatile organic compound content

VOC content

mass of the *volatile organic compounds* (3.1) present in a *coating material* (3.9), as determined under specified conditions

Note 1 to entry: The properties and the amounts of the compounds to be taken into account depend on the field of application of the coating material. For each field of application, the limiting values and the methods of determination or calculation are stipulated by regulations or by agreement.

Note 2 to entry: If the term VOC refers to compounds with a defined maximum boiling point, the compounds considered to be part of the VOC content are those with boiling points below and including that limit, and compounds with higher boiling points are considered to be *semi-volatile* (3.2) or non-volatile organic compounds.

[SOURCE: ISO 4618:2023, 3.267 modified — Note 2 to entry has been added.]

3.4

semi-volatile organic compound content

SVOC content

mass of the *semi-volatile organic compounds (SVOC)* (3.2) present in a *coating material* (3.9), as determined under specified conditions

Note 1 to entry: The properties and the amounts of the compounds to be taken into account depend on the field of application of the coating material. For each field of application, the limiting values and the methods of determination or calculation are stipulated by regulations or by agreement.

Note 2 to entry: If the term SVOC refers to compounds with a defined maximum boiling point and minimum boiling point, the compounds considered to be part of the SVOC content are as followed:

- compounds above the lower limit value (lower limit value not included) and;
- compounds lower or equal to the upper limit (upper limit included).

Compounds with boiling points higher than the upper limit for SVOC are considered to be non-volatile organic compounds.

[SOURCE: ISO 4618:2023, 3.228, modified — Note 2 to entry has been modified.]

3.5

ready for use

state of a product when it is mixed in accordance with the manufacturer's instructions in the correct proportions and thinned, if required, using the correct thinners so that it is ready for application by the approved method

Note 1 to entry: In context of this VOC emission standard, only waterbased thinners will be of relevance.

3.6

surrogate standard

compound of known purity which is used to quantify unidentified *volatile organic compounds* (3.1)

3.7

marker compound

compound which is used to differentiate between *volatile organic compounds (VOC)* (3.1) and *semi-volatile organic compounds (SVOC)* (3.2), or SVOC and non-volatile organic compound if the differentiation is made on the basis of *retention time* (3.8)

3.8

retention time

t_R
time elapsed from injection of the sample component to the recording of the peak maximum

3.9

coating material

product, in liquid, paste or powder form, that, when applied to a substrate, forms a layer possessing protective, decorative and/or other specific properties

[SOURCE: ISO 4618:2023, 3.48]

4 Principle

The volatile organic compounds (VOC) in a very small amount of a thinned sample are fully evaporated in a headspace injector and then determined by gas-chromatographic analysis, as follows.

A few microlitres of the sample diluted with a buffer solution are heated to 150 °C in a septum-sealed vial. When fully evaporated, some of the vapour phase is transferred to a non-polar capillary column. The peak areas of all the components with retention times less than that of the marker compound (tetradecane, boiling point 250 °C) are integrated. Standard additions of a surrogate standard are employed at four concentration levels to determine the VOC content. The result is based on the response factor of the surrogate standard.

For a series of samples with the same matrix, quantification via an external standard can be applied.

NOTE The determination of individual components in the sample, or of standards prepared separately from coating materials or their raw materials which are as free of VOCs as possible, can be used to validate the analytical system.

5 Required information

For any particular application, the information required should preferably be agreed between the interested parties and may be derived, partially or totally, from an international or national standard or other document related to the product under test.

The method of calculation used shall be indicated (see [Clause 10](#)).

6 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

6.1 Gas chromatograph (GC)

6.1.1 General

All instrumental parts which come into contact with the test sample shall be made of a material (e.g. glass), which is resistant to the sample and does not change it chemically.

6.1.2 Headspace injector, preferably with an automatic sample changer.

6.1.3 Oven

The oven shall be capable of being heated between 40 °C and 300 °C both isothermally and under programmed temperature control. It shall be possible to set the oven temperature to within 1 °C. The final temperature of the temperature programme shall not exceed the maximum operating temperature of the capillary column (see [6.1.5](#)).

6.1.4 Detector

A flame ionization detector (FID) shall be used, for quantification of VOC compounds operated at temperatures between 230 °C and 300 °C. To prevent condensation, the detector temperature shall be at least 10 °C above the maximum oven temperature. The detector gas supply, injection volume, split ratio and gain setting shall be optimized so that the signals (peak areas) used for the calculation are proportional to the amount of substance.

6.1.5 Capillary column

A non-polar fused-silica capillary column shall be used, with a stationary phase consisting of 95 % to 100 % dimethylsilicone and 5 % to 0 % of phenylsilicone chemically bound to the column.

NOTE Capillary columns having a length of 30 m and an internal diameter of 0,32 mm coated with 95 % dimethylsilicone and 5 % phenylsilicone (film thickness approximately 1 µm) were found to be suitable in an interlaboratory trial.

6.1.6 Gas filters

[ISO/FDIS 17895
https://standards.iteh.ai/catalog/standards/iso/117b5cdf-0b57-45d6-b3c6-21e39a03b76e/iso-fdis-17895](https://standards.iteh.ai/catalog/standards/iso/117b5cdf-0b57-45d6-b3c6-21e39a03b76e/iso-fdis-17895)
Filters shall be installed in the gas chromatograph connection pipes to adsorb residual impurities in the gases (see [6.1.7](#)).

6.1.7 Gases

6.1.7.1 Carrier gas, dry, oxygen-free helium, nitrogen or hydrogen, having a purity of at least 99,996 % by volume.

6.1.7.2 Detector gases, hydrogen having a purity of at least 99,999 % by volume and purified or synthetic air, free of organic compounds.

6.1.8 Data processing

A suitable software shall be used for integration, calibration, quantification and other data handling processes.

6.2 Microlitre syringe, having a capacity of 50 µl.

6.3 Disposable plastic syringe, having a capacity of 2 ml.

6.4 Septum-sealable vials, having a capacity of about 20 ml, with a butyl or silicone rubber septum coated with polytetrafluoroethylene (PTFE). Since the test conditions specified in this document result in fairly high pressures in the vials, care shall be taken to ensure that they are tightly sealed.

6.5 Volumetric flask, having a capacity of 1 l.

6.6 Analytical balance, accurate to 0,1 mg.

7 Reagents

7.1 Demineralized water, of grade 1 in accordance with ISO 3696.

Demineralized water tends to cause microbial contamination. Demineralized water with low microbial contamination [colony forming units (CFU) < 100] should be used, or it should be tested that a microbial contamination of the demineralized water has no influence on the measurement result. Formaldehyde as a preservative can potentially react with microorganisms.

7.2 Surrogate standard

The surrogate standard to be used is a mixture of the following representative reference compounds:

- diethylene glycol monobutyl ether;
- diethylene glycol monobutyl ether acetate;
- butanol;
- butyl acrylate;
- 2-ethylhexyl acrylate;
- styrene;
- vinyl acetate.

Weigh, to the nearest 1 mg, about 1 g of each of the reference compounds into a septum-sealable vial using the analytical balance (6.6), adding the high boilers before the low boilers. Remove the cap only to allow each reference compound to be introduced. Add approximately 5 mg to 10 mg of polymerisation inhibitor (7.5) (leading to about a concentration of 1 000 mg/kg of polymerisation inhibitor) to the mixture.

NOTE Evaporation of readily volatile substances during weighing can be reduced by precooling and pipetting the reference compounds.

An example of a chromatogram of the stock reference compound mixture is given in [Figure A.1](#).

7.3 Marker compound

The marker compound n-tetradecane characterizes the upper limit of the VOC range. Its retention time is the end point for integration.

7.4 Citrate buffer, pH 5,0, obtainable commercially in ready for use form or can be prepared in-house by mixing 20,265 g of citric acid and 7,840 g of sodium hydroxide filled up to 1 l with demineralized water (7.1) at 20 °C in a volumetric flask.

7.5 Polymerisation inhibitor, 2,6-di-tert-butyl-4-methylphenol or the acid sodium salt hydrate of *N,N*-dimethyldithiocarbamate.

8 Procedure

8.1 Number of determinations

Carry out triplicate determinations of the test sample without standard additions (see [8.2.3](#)) and of each of the four test samples containing standard additions (see [8.2.4](#)).

8.2 Sample preparation

8.2.1 General

Sample preparation involves diluting the sample (see [8.2.2](#)) and preparing the test samples with and without standard additions (see [8.2.3](#) and [8.2.4](#), respectively).

Carry out the sample preparation quickly since the original sample diluted with citrate buffer is prone to serum formation and losses can occur as the result of volatilization of individual compounds.

[Figure 1](#) shows a diagram of the sample-preparation plan.

8.2.2 Dilution of original sample

Weigh 10 g of the original sample and 10 g of citrate buffer ([7.4](#)) to the nearest 0,1 g into a 20 ml septum vial ([6.4](#)), seal and mix.

8.2.3 Preparation of test samples for analysis without multiple standard additions

Vigorously shake the sealed septum vial containing the diluted sample (prepared as in [8.2.2](#)), then immediately remove any excess vapour by piercing the septum with a 2 ml disposable syringe ([6.3](#)). For the analysis, weigh aliquots of (15 ± 3) mg to the nearest 0,1 mg into each of three vials ([6.4](#)) and seal the latter immediately.

NOTE Higher initial test sample masses result in errors in the result due to the increase in pressure.

8.2.4 Preparation of test samples for analysis with multiple standard additions

Prepare four diluted samples as in [8.2.3](#) and add, using a 50 μ l syringe ([6.2](#)), about 10 μ l, 20 μ l, 30 μ l and 40 μ l of surrogate standard ([7.2](#)) (a different volume to each vial). Note the added amount to the nearest 0,1 mg. Close the vials firmly and mix well.

Shake the sealed vials vigorously once again, then remove any excess vapour by piercing the septum with a 2 ml disposable syringe ([6.3](#)). For the analysis, weigh an aliquot of about (15 ± 3) mg of these diluted test samples containing standard additions to the nearest 0,1 mg into each of three empty vials ([6.4](#)) and seal the latter immediately.

NOTE If a standard addition for a certain coating matrix is not feasible, another representative coating matrix can be used alternatively for standard addition. Since the slope of the calibration curve is likely to be different when using another matrix, it was necessary to record this deviation in the test report.