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## Paints and varnishes — Determination of volatile organic compounds (VOC) and/or semi volatile organic compounds (SVOC) content —

### Part 2: Gas-chromatographic method

*Peintures et vernis — Détermination de la teneur en composés organiques volatils (COV) et composés organiques semi-volatils (COSV) —*

*Partie 2: Méthode par chromatographie en phase gazeuse*

Member bodies are requested to consult relevant national interests in ISO/TC 28 before casting their ballot to the e-Balloting application.

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ISO copyright office  
CP 401 • Ch. de Blandonnet 8  
CH-1214 Vernier, Geneva  
Phone: +41 22 749 01 11  
Fax: +41 22 749 09 47  
Email: [copyright@iso.org](mailto:copyright@iso.org)  
Website: [www.iso.org](http://www.iso.org)

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## Foreword

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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](http://www.iso.org/directives)).

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This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, 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 fourth edition cancels and replaces the third edition (ISO 11890-2:2013), which has been technically revised.

The main changes compared to the previous edition are as follows:

- the scope has been expanded to include the determination of semi volatile organic compounds (SVOC);
- the scope has been expanded to include concentration ranges from 0,01 % to 100 %;
- the specifications for determination of semi-volatile organic compounds have been added.

A list of all parts in the ISO 11890 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](http://www.iso.org/members.html).

## Introduction

This document is one of a series of standards dealing with the sampling and testing of coating materials and their raw materials. It specifies a method for the determination of the volatile organic compounds (VOC) content and the semi-volatile organic compounds (SVOC) content of coating materials and their raw materials.

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# Paints and varnishes — Determination of volatile organic compounds (VOC) and/or semi volatile organic compounds (SVOC) content —

## Part 2: Gas-chromatographic method

**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 is applicable for the determination of VOC and SVOC with an expected VOC and/or SVOC content greater than 0,01 % by mass up to 100 % by mass.

The method given in ISO 11890-1 is used when the VOC is greater than 15 % by mass. This document (method ISO 11890-2) applies when the system contains VOC and SVOC as the VOC result of ISO 11890-1 can be influenced by the SVOC. For VOC content smaller than 0,1 %, the head space method described in ISO 17895 is used as an alternative. ISO 11890-1 and ISO 17895 cannot be used for the determination of the SVOC content.

**NOTE 1** Some ingredients of coating materials and their raw materials can decompose during analysis and cause artificial VOC and SVOC signals. When determining VOC and SVOC for coating materials and their raw materials, these signals are artefacts of the method and are not taken into account (examples are given in [Annex B](#)).

This method assumes that the volatile matter is either water or organic. However, other volatile inorganic compounds can be present and might need to be quantified by another suitable method and allowed for in the calculations. The method defined in this document is not applicable for determination of water content.

**NOTE 2** If organic acids or bases and their corresponding salts are present in the coating material or its raw materials, the amount that is quantified by this method might not be accurate due to a change in the acid or base equilibrium.

### 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 760, *Determination of water — Karl Fischer method (General method)*

ISO 1513, *Paints and varnishes — Examination and preparation of test samples*

ISO 2811 (all parts), *Paints and varnishes — Determination of density*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

### 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 <http://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

#### 3.1 volatile organic compound

##### VOC

any 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, see *volatile organic compounds content (VOC content)* (3.4).

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* (3.6).

[SOURCE: ISO 4618:2014, 2.270]

#### 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: As to current usage of the term SVOC in the field of coating materials, see *semi-volatile organic compounds content (SVOC content)* (3.5).

#### 3.3 non-volatile organic compound

##### NVOC

organic liquid and/or solid not classified as VOC or SVOC

#### 3.4 volatile organic compounds content

##### VOC content

##### VOCC

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

Note 1 to entry: The properties and the amounts of the compounds to be taken into account will 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 or non-volatile organic compounds.

[SOURCE: ISO 4618:2014, 2.271, modified — Note 2 to entry has been added.]



**3.5**  
**semi-volatile organic compounds content**  
**SVOC content**  
**SVOCC**

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

Note 1 to entry: The properties and the amounts of the compounds to be taken into account will 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 those with boiling points below and including the upper and above the lower limit, and compounds with higher boiling points are considered to be non-volatile organic compounds.

**3.6**  
**exempt compound**

organic compound that does not participate in atmospheric photochemical reactions

Note 1 to entry: This expression is only relevant in some countries.

**3.7**  
**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

**3.8**  
**internal standard**

compound which is not present in the sample, is completely separated from the other components in the chromatogram, is inert with respect to the sample constituents, stable in the required temperature range and of known purity and which is added to the sample to control the dilution and the injection step of the analysis

**3.9**  
**surrogate standard**

compound of known purity which is used to quantify unidentified VOCs and SVOCs

**3.10**  
**marker compound**

compound which is used to differentiate between VOC and SVOC, or SVOC and NVOC if the differentiation has to be made on the basis of *retention time* (3.11)

**3.11**  
**retention time**

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

**3.12**  
**extraction solvent**

liquid used to extract the VOCs and SVOCs from the matrix under investigation

**3.13**  
**major peak**

peak that contributes significantly to either

- the VOC content, i.e.  $\geq 10\%$  of the absolute VOC content (as DEA equivalent) and  $\geq 0,1\%$  by mass (as DEA equivalent), or
- the SVOC content, i.e.  $\geq 10\%$  of the absolute SVOC content (as DEA equivalent) and  $\geq 0,1\%$  by mass (as DEA equivalent)

### 3.14

#### **minor peak**

peak that contributes only to a minor extent to either

- the VOC content, i.e. < 0,1 % by mass (as DEA equivalent) and/or < 10 % of the absolute VOC content (as DEA equivalent), or
- the SVOC content, i.e. < 0,1 % by mass (as DEA equivalent) and/or < 10 % of the absolute SVOC content (as DEA equivalent)

### 3.15

#### **reagent**

substance used in chemical/biochemical analysis or other reactions

[SOURCE: ISO 20391-1:2018, 3.19]

## 4 Principle

After preparation of the sample, the VOCs, SVOCs and NVOCs are separated by a gas chromatographic technique. Either a hot or a cold sample injection system is used, depending on the sample type. Hot injection is the preferred method. After the compounds have been identified, e.g. via GC-MS, they are quantified from the peak areas with respect to their compound specific relative response with the help of an internal standard, via GC-FID. Non-identifiable substances are quantified with respect to a surrogate standard that can be identical to the internal standard. A calculation is performed to give the VOC and/or SVOC content of the sample.

## 5 Required information

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

The required information can include the following points:

- a) the organic compound(s) to be determined (see [Clause 9](#));
- b) the experimental conditions to be used (see [8.5](#));
- c) the classification criteria for VOC and/or SVOC;
- d) which of the organic compounds in a) are exempt compounds (if relevant);
- e) the method of calculation to be used (see [Clause 10](#) and [Clause 11](#)).

## 6 Apparatus

### 6.1 Gas chromatograph

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

Use one of the two types specified in [6.1.1](#) and [6.1.2](#).

### 6.1.1 Hot-injection system (preferred system)

The instrument shall have a variable-temperature injection block with sample splitter. The injection temperature shall be capable of being set to an accuracy of 1 °C. Standard operating temperature shall be between 250 °C and 280 °C.

NOTE It is useful to use silanized glass wool to retain non-volatile constituents. The active sides of silanized glass wool can be a sink for organic compounds and significantly influence the recovery rate in the lower range of the method. The occurrence of adsorption is revealed by peak tailing, in particular with components of low volatility and/or high polarity.

### 6.1.2 Cold-injection system (programmed temperature vaporizer, PTV)

The cold-injection system shall be provided with temperature programming for heating from ambient to 300 °C including a sample splitter for split operation.

NOTE It is useful to use silanized glass wool to retain non-volatile constituents. The active sides of silanized glass wool can be a sink for organic compounds and significantly influence the recovery rate in the lower range of the method. The occurrence of adsorption is revealed by peak tailing, in particular with components of low volatility and/or high polarity.

### 6.1.3 Selection of sample injection system

The choice between hot-injection and cold-injection will depend on the type of product under test. It is preferred to use the cold-injection system for products which, at high temperature, release substances which interfere with the determination. Cleavage products of binders or additives can be reduced by the use of cold injection system.

Indications of cleavage or decomposition reactions can be obtained by looking for changes in the chromatogram (for example the occurrence of foreign peaks or an increase or decrease in peak size) at various sample injector temperatures (see [Annex B](#)).

## 6.2 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 [6.4](#)).

## 6.3 Detector

The following two detectors shall be used.

**6.3.1 Mass spectrometer or other mass-selective detector (MS or MSD)**, for the identification of VOC and SVOC compounds. To prevent condensation, the detector temperature shall be at least 10 °C above the maximum oven temperature.

**6.3.2 Flame ionization detector (FID)**, for quantification of VOC and SVOC 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.

#### 6.4 Capillary column

The column shall be made of glass or fused silica. Columns of sufficient length to resolve volatiles and of maximum internal diameter 0,32 mm, coated with 5 % phenyl-modified poly(dimethylsiloxane) or 6 % (cyanopropyl-phenyl)-methylpolysiloxane at a suitable film thickness shall be used.

NOTE Columns such as DB-5, HP-5, or DB-1301 are suitable<sup>1)</sup>. DB-5, HP-5 or DB-1301 are GC columns characterized as equivalent to USP phase G27 or G43. Other columns equivalent USP phase G27 or G43, which meet the pre-set performance criteria of this method can be used as well.

#### 6.5 Analytical system performance criteria

The analytical system performance criteria shall be demonstrated. The limit of quantification (LOQ) for the VOC content and SVOC content, respectively, is 0,01 % by mass. For single compounds, the LOQ is assumed to be 0,005 % by mass using the response factor for diethyladipate (i.e. in DEA equivalents).

NOTE The limit of quantification can deviate for single compounds. If necessary, the compound specific limit of quantification can be determined for the considered single compound(s).

Chromatographic resolution shall be sufficient to separate the compounds DEA and tetradecane (C14), as well as docosane (C22) and dibutylsebacate (DBS). At least a resolution of 1 shall be reached.

For the quantification of VOC content and/or SVOC content at concentrations below 0,1 % by mass the LOQ of triethylene glycol (TEG) shall be proven to be at least 0,005 % by mass, based on the sample weight.

#### 6.6 Qualitative-analysis equipment

If the separated components are to be identified by a mass-selective detector, the instrument shall be coupled to the gas chromatograph.

#### 6.7 Injection syringe

The injection syringe shall have a capacity of at least twice the volume of the sample to be injected into the gas chromatograph.

#### 6.8 Data processing

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

#### 6.9 Sample vials

Use vials made of chemically inert material (e.g. glass) which can be sealed with a suitable septum cap [e.g. a rubber membrane coated with poly(tetra fluoro ethylene)].

#### 6.10 Gas filters

Filters shall be installed in the gas chromatograph connection pipes to adsorb residual impurities in the gases (6.11).

#### 6.11 Gases

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

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1) DB-5, HP-5 and DB-1301 are the trade names of products. 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.