
**Paints and varnishes — Determination of
volatile organic compound (VOC) content —
Part 2:
Gas-chromatographic method**

*Peintures et vernis — Détermination de la teneur en composés organiques
volatils (COV) —
Partie 2: Méthode par chromatographie en phase gazeuse*

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Printed 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 part of ISO 11890 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 11890-2 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*.

ISO 11890 consists of the following parts, under the general title *Paints and varnishes — Determination of volatile organic compound (VOC) content*:

- Part 1: *Difference method*
- Part 2: *Gas-chromatographic method*

Annex A forms a normative part of this part of ISO 11890. Annex B of this part of ISO 11890 is for information only.

Paints and varnishes — Determination of volatile organic compound (VOC) content —

Part 2: Gas-chromatographic method

1 Scope

This part of ISO 11890 is one of a series of standards dealing with the sampling and testing of paints, varnishes and related products.

It specifies a method for the determination of the volatile organic compound (VOC) content of paints, varnishes and their raw materials. This part shall be used where the expected VOC content is greater than 0,1 % by mass and less than about 15 % by mass. When the VOC content is greater than about 15 % by mass, the less complicated method given in ISO 11890-1 may be used.

This method assumes that the volatile matter is either water or organic. However, other volatile inorganic compounds may be present and may need to be quantified by another suitable method and allowed for in the calculations.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 11890. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 11890 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 760:1978, *Determination of water — Karl Fischer method (General method)*.

ISO 1513:1992, *Paints and varnishes — Examination and preparation of samples for testing*.

ISO 2811-1:1997, *Paints and varnishes — Determination of density — Part 1: Pycnometer method*.

ISO 2811-2:1997, *Paints and varnishes — Determination of density — Part 2: Immersed body (plummet) method*.

ISO 2811-3:1997, *Paints and varnishes — Determination of density — Part 3: Oscillation method*.

ISO 2811-4:1997, *Paints and varnishes — Determination of density — Part 4: Pressure cup method*.

ISO 4618-1:1998, *Paints and varnishes — Terms and definitions for coating materials — Part 1: General terms*.

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*.

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*.

ISO 11890-1:2000, *Paints and varnishes — Determination of volatile organic compound (VOC) content — Part 1: Difference method*.

ISO 15528:—¹⁾, *Paints, varnishes and raw materials for paints and varnishes — Sampling*.

ASTM D 3960-98, *Standard Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings*.

3 Terms and definitions

For the purposes of this part of ISO 11890, the following terms and definitions apply.

3.1

VOC

volatile organic compound

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

[ISO 4618-1:1998]

3.2

VOC content

volatile organic compound content

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

NOTE 1 The properties and the amount 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 agreement.

NOTE 2 Under certain US governmental legislation, the term VOC is restricted solely to those compounds that are photochemically active in the atmosphere (see ASTM D 3960). Any other compound is then defined as being an exempt compound.

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3.3

exempt compound

organic compound that does not participate in atmospheric photochemical reactions (see note 2 to definition 3.2)

3.4

ready for use

the 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

4 Principle

After preparation of the sample, the VOCs 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, they are quantified from the peak areas using an internal standard. Depending on the equipment used, it may also be possible to determine the water content by this method. A calculation is then performed to give the VOC content of the sample.

5 Required supplementary information

For any particular application, the test method specified in this part of ISO 11890 needs to be completed by supplementary information. The items of supplementary information are given in annex A.

1) To be published. (Revision of ISO 842:1984 and ISO 1512:1991)

6 Apparatus

6.1 Gas chromatograph

The apparatus shall be set up and used in accordance with the manufacturer's instructions. 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.

6.2 Sample injection system

6.2.1 General

Use one of the two types specified in 6.2.2 and 6.2.3

6.2.2 Hot-injection system with sample splitter (preferred system)

The instrument shall have a variable-temperature injection block with a sample splitter. The injection temperature shall be capable of being set to an accuracy of 1 °C. The split ratio shall be adjustable and capable of being monitored. The sample splitter insert shall contain silanized glass wool to retain non-volatile constituents, and shall be cleaned and provided with new glass wool packing or replaced as required to rule out errors due to residues of binder or pigment (i.e. adsorption of compounds). The occurrence of adsorption is revealed by peak tailing, in particular with components of low volatility.

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6.2.3 Cold-injection system with sample splitter

The cold-injection system shall be provided with temperature programming for heating from ambient to 300 °C and shall have a sample splitter inlet which is made of an inert material such as glass. The sample splitter shall be provided with silanized glass wool packing and shall be maintained as specified in 6.2.2. The split ratio shall be adjustable and capable of being monitored.

Method precision will be increased if the injection system, especially the hot-injection system, is coupled to an auto-injector. The manufacturer's instructions shall be followed when an auto-injector is used.

6.2.4 Selection of sample injection system

The choice between hot injection and cold injection will depend on the type of product under test. It will be necessary to use the cold-injection system for products which at high temperature release substances which interfere with the determination.

Indications of cleavage or decomposition reactions may 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.

The hot-injection system covers all of the volatile constituents, compounds and cleavage products of the binders and additives. Cleavage products of the binders or additives which are identical to a constituent of the product can be separated by a cold-injection system, since they elute later as a result of the programmed increase in injection block temperature.

6.3 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 column (see 6.5).

6.4 Detector

Any of the following three detectors may be used:

6.4.1 Flame ionization detector (FID), capable of being operated at temperatures up to 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.

6.4.2 Suitably calibrated and tuned mass spectrometer or other mass-selective detector.

6.4.3 Suitably calibrated Fourier-transform infrared spectrometer (FT-IR spectrometer).

6.5 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 poly(dimethylsiloxane) or poly(ethylene glycol) at a suitable film thickness, have been shown to give good peak separation. The stationary phase and column length shall be chosen to suit the particular separation (see examples in annex B).

6.6 Qualitative-analysis equipment

If the separated components are to be identified by a mass-selective detector or FT-IR spectrometer, the instrument shall be coupled to the gas chromatograph and operated in accordance with the manufacturer's instructions.

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 Chart recorder

Compensating chart recorders are suitable for plotting the gas chromatogram.

6.9 Integrator

An electronic data-processing system (integrator or computer) shall be used to measure the peak areas. The integration parameters used in calibration and analysis shall be identical.

6.10 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 polytetrafluoroethylene).

6.11 Gas filters

Filters shall be installed in the gas chromatograph connection pipes to adsorb residual impurities in the gases (see 6.12).

6.12 Gases

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

6.12.2 Detector gases: hydrogen having a purity of at least 99,999 % by volume and air (synthetic), free of organic compounds.

6.12.3 Auxiliary gas: nitrogen or helium of the same quality as the carrier gas.

7 Reagents

7.1 Internal standard

The internal standard shall be a compound which is not present in the sample and is completely separated from the other components in the chromatogram. It shall be inert with respect to the sample constituents, stable in the required temperature range, and of known purity. Compounds such as isobutanol and diethylene glycol dimethyl ether have been found suitable.

7.2 Calibration compounds

The compounds used for calibration shall have a purity of at least 99 % by mass or shall be of known purity.

7.3 Dilution solvent

Use an organic solvent suitable for diluting the sample. It shall have a purity of at least 99 % by mass or shall be of known purity and it shall not contain any substances which interfere with the determination, e.g. causing overlapping peaks in the chromatogram. Always carry out a separate run injecting the solvent alone in order to observe contaminants and possible interference peaks, especially in trace analysis.

NOTE Solvents such as methanol and tetrahydrofuran have been found suitable.

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8 Sampling

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Take a representative sample of the product to be tested (or of each product in the case of a multi-coat system), as specified in ISO 15528.

Examine and prepare each sample for testing as specified in ISO 1513, preparing the final sample for testing in the "ready for use" state.

9 Procedure

9.1 Density

If required by the calculation (see 10.3 to 10.5), determine the density of the sample using the part of ISO 2811 which will give the best precision for the type of sample concerned. Determine the density at 23 °C.

9.2 Water content

Determine the water content, as a percentage by mass, by the method given in ISO 760, selecting the reagents so that there will be no interference from the compounds contained in the sample. If the compounds are not known, then determine them qualitatively (see 9.4).

NOTE 1 Typical compounds likely to cause interference are ketones and aldehydes. Reagent manufacturers normally publish literature for guidance on correct reagent selection.

NOTE 2 If the product to be tested is well characterized and known not to contain water, it may not be necessary to determine the water content, which can be assumed to be zero.

9.3 Gas-chromatographic conditions

9.3.1 The gas-chromatographic conditions used will depend on the product to be analysed and shall be optimized each time using a known calibration mixture. See annex B for examples of conditions for use with hot-injection and cold-injection systems.

9.3.2 The injection volume and the split ratio shall be coordinated so as not to exceed the capacity of the column and to remain within the linear range of the detector. Asymmetrical peaks will give an indication of overloading of the gas-chromatographic system.

9.4 Qualitative analysis of product

If the organic compounds in the product are not known, then determine them qualitatively. A gas chromatograph coupled to a mass-selective detector or FT-IR spectrometer (6.6), with the chromatograph programmed to the same settings as will be used in 9.3, is the preferred method.

9.5 Calibration

9.5.1 Where suitable compounds are commercially available, the response factor shall be determined using the following technique:

9.5.1.1 Weigh, into a sample vial (6.10), to the nearest 0,1 mg, amounts of the compounds determined in 9.4 which are of the same order of magnitude as their respective contents in the product under test.

Weigh out a similar amount of the internal standard (7.1) into the sample vial, dilute the mixture with the dilution solvent (7.3), and inject it under the same conditions as will be used for the test sample.

9.5.1.2 Optimize the instrumental parameters as indicated in 9.3.

9.5.1.3 Again inject a suitable amount of the calibration mixture into the gas chromatograph. Calculate the response factors for each of the compounds using the following equation:

$$r_i = \frac{m_{ci} \times A_{is}}{m_{is} \times A_{ci}}$$

where

r_i is the response factor for compound i ;

m_{is} is the mass, in grams, of internal standard in the calibration mixture;

m_{ci} is the mass, in grams, of compound i in the calibration mixture;

A_{is} is the peak area of the internal standard;

A_{ci} is the peak area of compound i .

9.5.2 Where unidentified peaks are found or where suitable compounds are not commercially available, a response factor of 1,0 shall be assumed.

9.6 Sample preparation

Weigh, to the nearest 0,1 mg, 1 g to 3 g of the sample and an amount of the internal standard which is of the same order of magnitude as the analytes into a sample vial. Dilute the test sample with a suitable volume of dilution solvent, seal the vial and homogenize the contents.

NOTE Pigmented or otherwise difficult samples may be cleaned up by centrifuging.

9.7 Quantitative determination of compound content

9.7.1 Set the instrumental parameters as optimized during calibration.

9.7.2 Inject 0,1 µl to 1 µl of the test sample into the gas chromatograph and record the chromatogram. Determine the peak areas for each compound and then determine the mass of each compound present in the paint using the following equation:

$$m_i = \frac{r_i \times A_i \times m_{is}}{m_s \times A_{is}}$$

where

m_i is the mass, in grams, of compound i in 1 g of the product;

r_i is the response factor for compound i (see 9.5.1.3);

A_i is the peak area of compound i ;

A_{is} is the peak area of the internal standard;

m_{is} is the mass, in grams, of internal standard in the test sample (see 9.6);

m_s is the mass, in grams, of the test sample (see 9.6).

NOTE Some solvents, such as naphtha, will elute as a series of peaks. With most recording integrators, the total area can be summed and treated as one peak, provided no other compounds coelute over this interval. If the integrator does not have this capability, then the total area will have to be summed manually. The equation above can then be used to determine the amount of solvent in the test sample.

10 Calculation

10.1 General

Calculate the VOC content by the method specified in the referring specification. If no particular method is specified, calculate the VOC content by method 1.

Method 1 is the preferred calculation method as the precision is better since it does not involve the determination of density (which introduces the potential for additional errors).

10.2 Method 1: VOC content, as a percentage by mass, of the product “ready for use”

$$\text{VOC} = \sum_{i=1}^{i=n} m_i \times 100$$

where

VOC is the VOC content, as a percentage by mass, of the product “ready for use”;

m_i is the mass, in grams, of compound i in 1 g of the test sample (see 9.7.2).