



มาตรฐานผลิตภัณฑ์อุตสาหกรรม

THAI INDUSTRIAL STANDARD

มอก. 2268 เล่ม 2 – 2549

ISO 11890 – 2 : 2000

# สีและวาร์นิช – การหาปริมาณสารประกอบอินทรีย์ ที่ระเหยได้

เล่ม 2 : วิธีก๊าซโครมาโทกราฟีค

PAINTS AND VARNISHES – DETERMINATION OF VOLATILE ORGANIC  
COMPOUND (VOC) CONTENT

PART 2 : GAS – CHROMATOGRAPHIC METHOD

สำนักงานมาตรฐานผลิตภัณฑ์อุตสาหกรรม

กระทรวงอุตสาหกรรม

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มาตรฐานผลิตภัณฑ์อุตสาหกรรม  
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สำนักงานมาตรฐานผลิตภัณฑ์อุตสาหกรรม  
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ประกาศในราชกิจจานุเบกษา ฉบับประกาศและงานทั่วไป เล่ม 123 ตอนที่ 64ง  
วันที่ 6 กรกฎาคม พุทธศักราช 2549

มาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้เป็นเล่มหนึ่งในชุดมาตรฐานผลิตภัณฑ์อุตสาหกรรมสีและวาร์นิช - การหาปริมาณสารประกอบอินทรีย์ที่ระเหยได้ เพื่อให้การทดสอบของหน่วยทดสอบ ผู้ทำ หรือผู้ที่เกี่ยวข้องเป็นมาตรฐานเดียวกัน จึงกำหนดมาตรฐานผลิตภัณฑ์อุตสาหกรรม สีและวาร์นิช - การหาปริมาณสารประกอบอินทรีย์ที่ระเหยได้ เล่ม 2 : วิธีก๊าซโครมาโทกราฟีค ขึ้น

มาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้กำหนดขึ้นโดยรับ ISO 11890-2 : 2000 Paints and varnishes - Determination of volatile organic compound (VOC) content-Part 2 : Gas - chromatographic method มาใช้ในระดับเหมือนกันทุกประการ (identical) โดยใช้ ISO ฉบับภาษาอังกฤษเป็นหลัก

มาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้กำหนดขึ้นเพื่อให้ทันกับความต้องการของผู้ใช้และจักได้แปลเป็นภาษาไทยในโอกาสอันสมควร หากมีข้อสงสัยโปรดติดต่อสอบถามที่สำนักงานมาตรฐานผลิตภัณฑ์อุตสาหกรรม

คณะกรรมการมาตรฐานผลิตภัณฑ์อุตสาหกรรมได้พิจารณามาตรฐานนี้แล้ว เห็นสมควรเสนอรัฐมนตรีประกาศตาม มาตรา 15 แห่งพระราชบัญญัติมาตรฐานผลิตภัณฑ์อุตสาหกรรม พ.ศ. 2511



## ประกาศกระทรวงอุตสาหกรรม

ฉบับที่ 3475 (พ.ศ. 2549)

ออกตามความในพระราชบัญญัติมาตรฐานผลิตภัณฑ์อุตสาหกรรม

พ.ศ. 2511

เรื่อง กำหนดมาตรฐานผลิตภัณฑ์อุตสาหกรรม

สีและวาร์นิช-การหาปริมาณสารประกอบอินทรีย์ที่ระเหยได้

เล่ม 2 : วิธีก๊าซโครมาโทกราฟีค

อาศัยอำนาจตามความในมาตรา 15 แห่งพระราชบัญญัติมาตรฐานผลิตภัณฑ์อุตสาหกรรม พ.ศ. 2511 รัฐมนตรีว่าการกระทรวงอุตสาหกรรมออกประกาศกำหนดมาตรฐานผลิตภัณฑ์อุตสาหกรรม สีและวาร์นิช-การหาปริมาณสารประกอบอินทรีย์ที่ระเหยได้ เล่ม 2 : วิธีก๊าซโครมาโทกราฟีค มาตรฐานเลขที่ มอก. 2268 เล่ม 2-2549 ไว้ดังมีรายการละเอียดต่อท้ายประกาศนี้

ประกาศ ณ วันที่ 24 มีนาคม พ.ศ. 2549

สุริยะ จึงรุ่งเรืองกิจ

รัฐมนตรีว่าการกระทรวงอุตสาหกรรม

# มาตรฐานผลิตภัณฑ์อุตสาหกรรม

## สีและวาร์นิช – การหาปริมาณสารประกอบอินทรีย์ ที่ระเหยได้

### เล่ม 2 : วิธีก๊าซโครมาโทกราฟี

#### บทนำ

มาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้กำหนดขึ้นโดยรับ ISO 11890-2 : 2000 Paints and varnishes – Determination of volatile organic compound (VOC) content – Part 2 : Gas – chromatographic method มาใช้ในระดับเหมือนกันทุกประการ (identical) โดยใช้ ISO ฉบับภาษาอังกฤษเป็นหลัก

#### ขอบข่าย

มาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ กำหนดวิธีทดสอบหาปริมาณสารประกอบอินทรีย์ที่ระเหยได้ในสี วาร์นิช และวัสดุที่เกี่ยวข้องที่มีปริมาณสารประกอบอินทรีย์ที่ระเหยได้ มากกว่าร้อยละ 0.1 และน้อยกว่าร้อยละ 15 โดยมวล ด้วยวิธีก๊าซโครมาโทกราฟี รายละเอียดให้เป็นไปตาม ISO 11890-2 : 2000 ข้อ 1

#### เอกสารอ้างอิง

รายละเอียดให้เป็นไปตาม ISO 11890- 2 : 2000 ข้อ 2

#### บทนิยาม

รายละเอียดให้เป็นไปตาม ISO 11890- 2 : 2000 ข้อ 3

#### หลักการ

รายละเอียดให้เป็นไปตาม ISO 11890- 2 : 2000 ข้อ 4

#### ข้อมูลเพิ่มเติม

รายละเอียดให้เป็นไปตาม ISO 11890- 2 : 2000 ข้อ 5

#### เครื่องมือ

รายละเอียดให้เป็นไปตาม ISO 11890- 2 : 2000 ข้อ 6

### **สารเคมี**

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### **การชักตัวอย่าง**

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### **การคำนวณ**

รายละเอียดให้เป็นไปตาม ISO 11890- 2 : 2000 ข้อ 10

### **การแสดงผลทดสอบ**

รายละเอียดให้เป็นไปตาม ISO 11890- 2 : 2000 ข้อ 11

### **ความเที่ยง**

รายละเอียดให้เป็นไปตาม ISO 11890- 2 : 2000 ข้อ 12

### **การรายงานผลทดสอบ**

รายละเอียดให้เป็นไปตาม ISO 11890- 2 : 2000 ข้อ 13

# 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:—<sup>1)</sup>, *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.

[Adapted from ISO 4618-1:1998]

#### 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.

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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.

#### 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.

## 8 Sampling

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).

### 10.3 Method 2: VOC content, in grams per litre, of the product “ready for use”

$$\text{VOC} = \sum_{i=1}^{i=n} m_i \times \rho_s \times 1\,000$$

where

VOC is the VOC content, in grams per litre, 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);

$\rho_s$  is the density, in grams per millilitre, of the test sample at 23 °C (see 9.1);

1 000 is a conversion factor.

### 10.4 Method 3: VOC content, in grams per litre, of the product “ready for use” less water

$$\text{VOC}_{\text{lw}} = \left( \frac{\sum_{i=1}^{i=n} m_i}{1 - \rho_s \times \frac{m_w}{\rho_w}} \right) \times \rho_s \times 1\,000$$

where

$\text{VOC}_{\text{lw}}$  is the VOC content, in grams per litre, of the product “ready for use” less water;

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

$m_w$  is the mass, in grams, of water in 1 g of the test sample (see 9.2);

$\rho_s$  is the density, in grams per millilitre, of the test sample at 23 °C (see 9.1);

$\rho_w$  is the density, in grams per millilitre, of water at 23 °C (= 0,997 537 g/ml);

1 000 is a conversion factor.

### 10.5 Method 4: VOC content, in grams per litre, of the product “ready for use” less water and less exempt compounds (only required if national legislation applies)

$$\text{VOC}_{\text{lwe}} = \left( \frac{\sum_{i=1}^{i=n} m_i}{1 - \rho_s \times \frac{m_w}{\rho_w} - \rho_s \times \sum_{i=1}^{i=n} \frac{m_{\text{eci}}}{\rho_{\text{eci}}}} \right) \times \rho_s \times 1\,000$$

where

$\text{VOC}_{\text{lwe}}$  is the VOC content, in grams per litre, of the product “ready for use” less water and less exempt compounds;

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

$m_w$  is the mass, in grams, of water in 1 g of the test sample (see 9.2);

$m_{\text{eci}}$  is the mass, in grams, of exempt compound  $i$  in 1 g of the test sample;

$\rho_s$  is the density, in grams per millilitre, of the test sample at 23 °C (see 9.1);

$\rho_w$  is the density, in grams per millilitre, of water at 23 °C (= 0,997 537 g/ml);

$\rho_{eci}$  is the density, in grams per millilitre, of exempt compound  $i$ ;

1 000 is a conversion factor.

## 11 Expression of results

If the two results (duplicates) differ by more than the value indicated in 12.2, repeat the procedure.

Calculate the mean of two valid results (replicates). For values greater than 1 % by mass, report to the nearest 0,1 %; for values less than or equal to 1 % by mass, report the result to the nearest 0,01 %.

## 12 Precision

### 12.1 General

The precision of the test method was determined by interlaboratory testing in accordance with ISO 5725-1 and ISO 5725-2. Four different materials were tested by five to seven laboratories. Some of the results were not considered when calculating the precision because they were not in the scope of the test method (see footnote "a" to Table 1). Their VOC content was above 15 % by mass but they were tested for a better comparison with the precision of ISO 11890-1.

### 12.2 Repeatability limit $r$

The repeatability limit  $r$  is the value below which the absolute difference between two single test results, each the mean of duplicates, obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method, may be expected to lie.

The repeatability for five repeated determinations made using this test method, expressed as the repeatability coefficient of variation lies between 1 % and 8 %.

### 12.3 Reproducibility limit $R$

The reproducibility limit  $R$  is the value below which the absolute difference between two test results, each the mean of duplicates, obtained on identical material by operators in different laboratories using the standardized test method, may be expected to lie.

The reproducibility for this test method, expressed as the reproducibility coefficient of variation, lies between 2 % and 11 %.

**Table 1 — Results of interlaboratory testing**

Parameter	Dispersion	Cathodic electro-deposition paint	Water-based paint	Two-component varnish <sup>a</sup>
Number of laboratories	5	7	5	6
Number of repeated determinations	5	5	5	5
Mean value, % by mass	0,36	11,68	11,83	43,36
Reproducibility standard deviation	0,04	0,24	1,22	4,73
Reproducibility coefficient of variation	10,4	2,0	10,3	10,9
Repeatability standard deviation	0,01	0,17	0,88	0,64
Repeatability coefficient of variation	2,6	1,5	7,4	1,5
<sup>a</sup> Not considered for the precision because the mean value is above 15 % by mass.				

### 13 Test report

The test report shall contain at least the following information:

- a reference to this part of ISO 11890 (ISO 11890-2);
- all details necessary for complete identification of the product tested (manufacturer, trade name, batch number, etc.);
- the items of supplementary information referred to in annex A;
- a reference to the international or national standard, product specification or other document supplying the information referred to in c) above;
- the results of the test, as indicated in clause 10, and the method of calculation used (10.2, 10.3, 10.4 or 10.5);
- any deviation from the test method specified;
- the date of the test.

## **Annex A** (normative)

### **Required supplementary information**

The items of supplementary information listed in this annex shall be supplied as appropriate to enable the method to be carried out.

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.

- a) The organic compound(s) to be determined (see clause 9).
- b) The experimental conditions to be used (see clause 9).
- c) Which of the organic compounds in a) are exempt compounds.
- d) The method of calculation to be used (see clause 10).

## **Annex B** (informative)

### **Examples of gas-chromatographic conditions**

#### **B.1 Hot injection of a water-thinnable product**

Injector temperature:	250 °C
Split ratio:	1:40
Injection volume:	0,5 µl, automatic injection
Oven temperature programme:	initial temperature: 70 °C isothermal holding time: 3 min heating rate: 10 °C/min final temperature: 200 °C isothermal holding time: 15 min
Detector temperature:	260 °C
Carrier gas:	helium column inlet pressure: 100 kPa
Column:	length: 25 m internal diameter: 0,2 mm poly(ethylene glycol) coated film thickness: 0,2 µm

#### **B.2 Cold injection of a water-thinnable product**

Cold-injection system temperature programme:	injection temperature: 30 °C heating rate: 10 °C/s first holding temperature: 100 °C holding time: 10 s heating rate: 10 °C/s second holding temperature: 260 °C holding time: 240 s split ratio: 1:20
Injection volume:	0,2 µl
Oven temperature programme:	initial temperature: 50 °C isothermal holding time: 4 min heating rate: 8 °C/min final temperature: 240 °C isothermal holding time: 10 min
Detector temperature:	280 °C

Carrier gas:	hydrogen column inlet pressure: 150 kPa
Column:	length: 50 m internal diameter: 0,32 mm poly(dimethylsiloxane) coated film thickness: 1,0 µm

### B.3 Hot injection of a product containing no water

Injector temperature:	250 °C
Split ratio:	1:100
Injection volume:	0,2 µl, automatic injection
Oven temperature programme:	initial temperature: 40 °C heating rate: 3 °C/min final temperature: 175 °C isothermal holding time: 15 min
Detector temperature:	260 °C
Carrier gas:	helium column inlet pressure: 170 kPa
Column:	length: 50 m internal diameter: 0,2 mm poly(dimethylsiloxane) coated film thickness: 0,25 µm

### B.4 Cold injection of a product containing no water

Cold-injection system temperature programme:	injection temperature: 40 °C heating rate: 10 °C/s first holding temperature: 100 °C holding time: 10 s heating rate: 10 °C/s second holding temperature: 250 °C holding time: 200 s split ratio: 1:100
Injection volume:	0,2 µl
Oven temperature programme:	initial temperature: 40 °C heating rate: 3 °C/min final temperature: 175 °C isothermal holding time: 10 min
Detector temperature:	260 °C

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Carrier gas:

helium

column inlet pressure: 170 kPa

Column:

length: 50 m

internal diameter: 0,32 mm

poly(dimethylsiloxane) coated

film thickness: 0,25  $\mu$ m