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Paints and varnishes — Determination of the volatile organic compound content of low-VOC emulsion paints (in-can VOC)

Peintures et vernis — Détermination du contenu en composés organiques volatiles dans des peintures en émulsion à faible teneur en COV (COV en récipient)

Reference number
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Contents

Page

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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 17895 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*.

Introduction

The requirements imposed today by authorities, for health and environmental reasons, include the assessment of the content of residual monomers and organic saturated volatiles, sometimes down to minute traces.

This International Standard is one of a series of standards dealing with the VOC content of paints, varnishes and related products: ISO 11890-1 (see the Bibliography) specifies a method for determining VOC contents greater than 15 % (by mass), ISO 11890-2 is applicable to VOC contents between 0,1 % and 15 % (by mass).

This International Standard describes a method for determining VOC contents between 0,01 % and 0,1 % (by mass). In contrast to ISO 11890-1 and ISO 11890-2, this standard is applicable to volatile organic compounds with boiling points up to 250 °C.

Paints and varnishes — Determination of the volatile organic compound content of low-VOC emulsion paints (in-can VOC)

1 Scope

This International Standard specifies a gas-chromatographic method of quantitatively determining the volatile organic compound (VOC) content (i.e. the content of organic compounds with boiling points up to 250 °C) under standard conditions (101,325 kPa) of low VOC content emulsion paints (in-can VOC). The method is applicable to VOC contents between 0,01 % and 0,1 % (by mass).

The main purpose of the method is to qualify low-VOC emulsion paints, not routine quality control.

2 Normative references

The following referenced documents are indispensable for the application 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: Pyknometer 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*

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.

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 As to current usage of the term VOC in the field of coating materials, see volatile organic compound content (VOC content).

NOTE 2 Under U.S. government 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.

[ISO 4618]

3.2
volatile organic compound content
VOC content
mass of the volatile organic compounds present in a coating material, as determined under specified conditions

NOTE The properties and the amounts of 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[1)] or by agreement.

[ISO 4618]

3.3
in-can VOC
volatile organic compound present in a water-based emulsion paint

3.4
emulsion paint
latex paint
coating material in which the organic binder is an aqueous dispersion

[ISO 4618]

3.5
full evaporation
method of transferring the VOCs in a liquid sample from the liquid to the vapour phase

NOTE Although a headspace injector with septum-sealable vials is used for introduction of a test portion of the vapour phase into the chromatographic column, the full evaporation method differs substantially from conventional headspace analysis in which equilibrium is established. Since the vial contains very small amounts of sample, virtually all the VOCs enter the vapour phase when heated to a certain temperature [3].

3.6
stock reference compound mixture
mixture prepared from pure substances for use in the standard addition method

NOTE The concentration of the stock reference compound mixture is determined by the initial masses of the individual components in the sample and their degree of purity.

3.7
multiple standard addition method
method of determining the VOC content in which a known amount of stock reference compound mixture is added to the sample

4 Principle

The VOCs in a very small amount of 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 buffer solution are heated to 150 °C in a septum-sealed vial and, 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 tetradecane (boiling point 252,6 °C) are integrated. Standard additions of a stock reference compound mixture (see 3.6) are employed at four

1) For the purposes of this International Standard, the applicable regulations are given in European Commission decision 96/13/EC dated 1996-01-06 which defines all organic compounds having a boiling point (or initial boiling point) of not more than 250 °C at normal pressure (101,325 kPa) as volatile organic compounds (VOCs).

concentration levels to determine the VOC content. The result is based on the average response factor of the reference compound mixture.

NOTE Determination of individual components in the sample, or of standards prepared separately from emulsions or emulsion paints which are as free of VOCs as possible, can be used to validate the analytical system.

5 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

5.1 Gas chromatography system, consisting of a headspace injector, preferably with an automatic sample changer, a temperature-programmable gas chromatograph suitable for capillary gas chromatography, a flame-ionization detector or mass-selective detector and a data evaluation system. All the components of the headspace injector which come into contact with the sample (e.g. dispensing needle, dispensing valve, transfer tube) shall be capable of being heated.

5.2 Non-polar fused-silica capillary column, 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.

5.3 Microlitre syringe, of capacity 50 µl.

5.4 2 ml disposable plastic syringe.

5.5 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 International Standard result in fairly high pressures in the vials, care shall be taken to ensure that they are tightly sealed.

5.6 Volumetric flask, of capacity 1 l.

5.7 Analytical balance, accurate to 0,1 mg.

5.8 Top-pan balance, accurate to 0,1 g.

5.9 Refrigerator, for storing the reference compounds.

6 Reagents and materials

Unless otherwise specified, use only reagents of recognized analytical grade (purity greater than 99 %) and only grade 1 water as defined in ISO 3696.

6.1 Gases:

— **Carrier gas**: dry, oxygen-free helium, nitrogen or hydrogen having a purity of at least 99,995 % (by volume).

— **Detector gas mixture**: hydrogen having a purity of at least 99,995 % (by volume) and synthetic air free of organic compounds.

6.2 Stock reference compound mixture, containing the following representative reference compounds:

— diethylene glycol monobutyl ether;

— diethylene glycol monobutyl ether acetate;