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Paints and varnishes — Determination of water content — Gas-chromatographic method

Peintures et vernis — Détermination de la teneur en eau — Méthode par chromatographie en phase gazeuse

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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).

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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 9, *General test methods for paints and varnishes*.

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 water content — Gas-chromatographic method

1 Scope

This document specifies a method for the determination of the water content of water-borne coating materials and their raw materials by using a gas chromatograph. The preferred working range of this test method is from a water mass fraction of 15 % to a water mass fraction of 90 % but the method can be applied outside of this range.

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

ISO 4618, *Paints and varnishes — Terms and definitions*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
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

[SOURCE: ISO 11890-2:2013, 3.4]

4 Principle

A suitable amount of the sample is internally standardized, diluted with appropriate organic solvent, and then injected into a gas chromatographic column that separates water from other components, after which the water is detected by a thermoconductivity detector and quantified from the peak areas using the internal standard.

5 Apparatus

5.1 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.

5.2 Sample injection 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 nonvolatile constituents, and shall be cleaned and provided with new glass wool packing or replaced as required to eliminate errors due to residues of binder or pigment (i.e. adsorption of compounds).

5.3 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 5.5).

5.4 Detector

Thermoconductivity detector (TCD), capable of being operated at temperatures up to 300 °C. The 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.

5.5 Capillary column

The column shall be made of glass or fused silica. Columns of sufficient length to resolve water and of maximum internal diameter 0,53 mm, based on bonded porous polymer technology shall be used. Columns should also show good stability and reproducibility with samples containing large amounts of water. Other columns proved to be equally suitable may also be used.

5.6 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.

5.7 Data processing

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

5.8 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)].

5.9 Gas filter

A filter shall be installed in the gas chromatograph connection pipes to adsorb residual impurities in the gas (see 5.10).