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Binders for paints and varnishes — Determination of saponification value — Titrimetric method

Liants pour peintures et vernis — Détermination de l'indice de saponification — Méthode titrimétrique

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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*.

This fourth edition cancels and replaces the third edition (ISO 3681:1996), which has been technically revised.

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

— the normative references in Clause 2 have been updated;

— a reference to ISO 4618 for terms and definitions relating to paints and varnishes has been added to Clause 3;

— the concentration of the phenolphthalein (5.4) solution has been lowered to 0,9 %.

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.

Binders for paints and varnishes — Determination of saponification value — Titrimetric method

1 Scope

This document specifies a titrimetric method for determining the esterified-acid content in binders for paints and varnishes, free acids and acid anhydrides being necessarily included in the result obtained.

Because different binders vary in their resistance to saponification, this document is of limited applicability. If necessary, completeness of saponification can be checked by repeating the test under more severe conditions achieved by the use of longer saponification time, more concentrated potassium hydroxide solution, or a higher-boiling alcohol as solvent.

Annex A specifies a procedure suitable for binders that saponify with difficulty.

The method is not applicable to those materials that show further reaction with alkalis beyond normal saponification.

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 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

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
saponification
formation of the alkali metal salts of derivatives of organic acids

3.2
saponification value
milligrams of potassium hydroxide (KOH) required for the *saponification* (3.1) of 1 g of the product tested

4 Principle

After a preliminary test to determine the saponification conditions (concentration of potassium hydroxide solution, saponification time, etc.) for the product to be tested, a test portion is boiled under reflux with potassium hydroxide solution under these conditions. The hot solution is titrated with standard volumetric hydrochloric acid, either in the presence of a colour indicator or potentiometrically.

5 Reagents

During the analysis, use only reagents of recognized analytical grade, and only water of at least grade 3 purity as defined in ISO 3696.

5.1 Toluene, or other suitable unsaponifiable solvent.

5.2 Potassium hydroxide solution, in isopropanol, ethanol or methanol, c(KOH) = 0,5 mol/l.

If more severe conditions for saponification are needed, 2 mol/l ethanolic potassium hydroxide solution may be used, or 1,2-ethanediol (ethylene glycol) or 2,2'-oxydiethanol (diethylene glycol) may be used as the solvent, in accordance with Clause 8 and Annex A.

Where isopropanol can be used instead of ethanol or methanol, it shall be used. The applicability of the solution in isopropanol is comparable to that of an ethanolic solution and its toxicity is less than that of a methanolic solution.

5.3 Hydrochloric acid, standard volumetric solution, c(HCl) = 0,5 mol/l, in a mixture of 4 parts by volume of methanol and 1 part by volume of water or in water.

5.4 Phenolphthalein or **thymolphthalein**, 9 g/l solution in 95 % (volume fraction) ethanol, in methanol or in isopropanol (see 5.2).

6 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

6.1 Conical flask, of capacity 250 ml, with a ground-glass joint.

6.2 Reflux condenser, with a ground-glass joint.

6.3 Burette or **pipette,** of capacity 25 ml or 50 ml, complying with the requirements of ISO 385 and ISO 648.

6.4 Potentiometric titration apparatus, fitted with a glass electrode and a reference electrode. The use of this apparatus is an optional alternative (see 9.3).

6.5 Magnetic stirrer.

6.6 Water bath or **oil bath.**

7 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.