DRAFT INTERNATIONAL STANDARD

ISO/DIS 4629-1

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

Part 1: Titrimetric method without using a catalyst

Liants pour peintures et vernis — Détermination de l'indice d'hydroxyle — Méthode titrimétrique — Partie 1: Méthode titrimétrique sans catalyseur

ICS: 87.060.20

ISO/CEN PARALLEL PROCESSING

This draft has been developed within the International Organization for Standardization (ISO), and processed under the **ISO lead** mode of collaboration as defined in the Vienna Agreement.

This draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel five month enquiry.

Should this draft be accepted, a final draft, established on the basis of comments received, will be submitted to a parallel two-month approval vote in ISO and formal vote in CEN.

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.

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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 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 4629-1 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for paints and varnishes*.

ISO 4629-1 cancels and replaces ISO 4629:1996, which has been technically revised. The main changes are:

a) the standard has been numbered as ISO 4629-1;

b) the standard has been editorially revised and the normative references have been updated;

c) the concentration of the phenolphthalein-indicator solution has been changed.

ISO 4629 consists of the following parts, under the general title *Binders for apints and varnishes — Determination of hydroxyl value*:

— *Part 1: Titrimetric method without using a catalyst*

— *Part 2: Titrimetric method using a catalyst*

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

Part 1:
Titrimetric method without using a catalyst

1 Scope

This part of ISO 4629 specifies a titrimetric method for determining the free hydroxyl groups in binders and binder solutions for paints and varnishes. The hydroxyl groups may be present as polyhydric alcohols, partial esters, polyester end groups or hydroxylated fatty acids.

This method is not applicable to resins containing both hydroxyl groups and epoxy groups, because the latter will also be included in the result. Also the method is not applicable to cellulose nitrate or to phenolic resins.

NOTE 1 If, in the case of binder solutions, the hydroxyl value of the binder only is to be determined, the possibility that other constituents of the binder solution may contain hydroxyl groups will have to be taken into account.

NOTE 2 A method for the determination of the hydroxyl value of epoxy resins is specified in ISO 7142.[1]

2 Normative references

The following referenced documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385-1, *Laboratory glassware — Burettes — Part 1: General requirements*

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

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

ISO 2114:2000, *Plastics (polyester resins) and paints and varnishes (binders) — Determination of partial acid value and total acid value*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
hydroxyl value
number of milligrams of potassium hydroxide (KOH) corresponding to hydroxyl groups that have been acetylated under specified test conditions in 1 g of the product tested

4 Principle

The hydroxyl groups contained in a test portion are acetylated with acetic anhydride. The excess acetic anhydride is hydrolysed and the resulting acetic acid is titrated with potassium hydroxide solution, either in the presence of a colour indicator or potentiometrically.

NOTE Primary and secondary amines, if present, will also be acetylated. In such cases, this will have to be allowed when calculating the hydroxyl value.

5 Reagents

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

5.1 Potassium hydrogen phthalate [$C_6H_4(COOH)_2HK$].

5.2 Ethyl acetate, anhydrous.

5.3 Toluene/butanol mixture, 1 + 2 by volume.

5.4 Pyridine/water mixture, 3 + 1 by volume.

5.5 Acetylating reagent.

Dissolve 4,0 g of p-toluenesulfonic acid monohydrate ($CH_3C_6H_4SO_3H{\cdot}H_2O$) in 100 ml of ethyl acetate (5.2), preferably using a magnetic stirrer.

To this solution, add slowly, while stirring, 33 ml of acetic anhydride. Check that 5 ml of this reagent requires on titration a volume of between 40 ml and 50 ml of potassium hydroxide solution (5.6) for neutralization.

5.6 Potassium hydroxide, standard volumetric solution, c(KOH) ≈ 0,5 mol/l, in methanol.

NOTE Ethanol can also be used if the product to be tested is soluble in ethanol.

5.6.1 Preparation

Weigh, to the nearest 0,05 g, 28 g of potassium hydroxide, dissolve in the minimum quantity of water in a 1 000 ml one-mark flask, dilute to the mark with methanol and mix well.

5.6.2 Standardization

Weight, to the nearest 0,01 g, 2,5 g of potassium hydrogen phthalate (5.1), previously dried at about 120 °C to constant mass and allowed to cool in a desiccator, into a 250 ml flask. Add 150 ml freshly boiled and cooled water and swirl until dissolved.

Titrate the potassium hydroxide solution prepared in 5.6.1, using phenoplphthalein solution (5.7) as indicatiop, until a red coloration that remains for at least 10 s appears.

Calculate the actual concentration c, in moles of hydroxyl ions (OH^-) per litre, of the potassium hydroxide solution, using equation (1):

$$c = \frac{m}{V} \cdot \frac{1\,000}{204,22} \qquad (1)$$

Where

m is the mass, in grams, of potassium hydrogen phthalate taken;

V is the volume, in millilitres, of potassium hydroxide solution used for the titration;

204,22 is the relative molecular mass, in grams per mole, of potassium hydrogen phthalate.

5.7 Phenolphthalein, 5 g/l solution in 95 % (by volume) ethanol, in methanol or in isopropanol.

5.8 Mixed-indicator solution.

Mix 3 volumes of a 1 g/l ethanolic solution of thymol blue with 1 volume of a 1 g/l ethanolic solution of cresol red.

6 Apparatus

Ordinary laboratory equipment and glassware, together with the following.

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

6.2 Reflux condenser, with a ground-glass joint to the conical flask (6.1).

6.3 Microburette or **pipette** complying with the requirements of class A of ISO 648, of capacity 5 ml, for acetylating reagent (5.5).100 ml conical flask with grounded joint.

WARNING — If a pipette is used, this should not be a mouth pipette in view of the corrosive nature of the reagent.

6.4 Burette, of capacity 50 ml, complying with the requirements of ISO 385-1, for the potassium hydroxide solution (5.6).

6.5 Heating apparatus, e.g. an oil bath or a sand bath, capable of being maintained at (50 ± 1) °C.

6.6 Potentiometric titration apparatus, fitted with a glass electrode and a reference electrode. The use of his apparatus is an optional alternative (see 8.3).

7 Sampling

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

8 Procedure

8.1 Number of determinations

Carry out the determination in duplicate

8.2 Test portion

By reference to Table 1, select the appropriate mass of test portion to be taken. If the hydroxyl value cannot be predicted, take a test portion of 2,0 g and carry out a preliminary determination.