DRAFT INTERNATIONAL STANDARD

ISO/DIS 4629-2

ISO/TC **35**/SC **10**

Secretariat: **DIN**

Voting begins on:
2014-12-18

Voting terminates on:
2015-05-18

Binders for paints and varnishes — Determination of hydroxyl value —

Part 2:
Titrimetric method using a catalyst

Liants pour peintures et vernis — Détermination de l'indice d'hydroxyle —

Partie 2: Méthode utilisant d'un cataluseur

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.

Reference number
ISO/DIS 4629-2:2014(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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 4629-2 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*.

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

— *Part 1: Method without using a catalyst*

— *Part 2: Method using a catalyst*

Introduction

There are several different methods standardized for determining the hydroxyl value of resins. The classic method using pyridine without a catalyst is specified in ISO 4629-1. The advantages of the method using a catalyst are:

— the solvents used are less hazardous to health;

— the solvent consumption is lower;

— the method is faster due to shorter reaction times;

— the endpoint of the titration is easier to see;

— polyols are more readily soluble.

Binders for paints and varnishes — Determination of hydroxyl value —

Part 2:
Titrimetric method using a catalyst

1 Scope

This International Standard specifies a titrimetric method for determining the hydroxyl value of resins, binders for paints and varnishes, primary alcohols, glycols and fats. Whether it can be applied for hydro carboxylic acids, phenolic hydroxyl groups, polyols such as trimethyl propane and substances containing aromatic groups have been activated for Friedel-Crafts acylation shall be decided on case-to-case basis.

Under the right conditions, the method is also applicable for determining the hydroxyl value of castor oil and its derivatives.

2 Normative references

The following 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 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*

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.

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

(Source: ISO 4629-1:—[1)], 3.1)

4 Principle

The hydroxyl groups in polyols are acetylated with acetic anhydride. The excess acetic anhydride is titrated with alcoholic potassium hydroxide solution.

1) Under preparation. Revision of ISO 4629:1996.

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 N-Methylpyrrolidone (NMP).

5.2 Potassium hydroxide solution, c = 0,5 mol/l in methanol.

5.3 Methyl ethyl ketone (MEK).

5.4 Demineralized water.

5.5 Acetylating reagent. Make up a 10 % solution of acetic anhydride in NMP.

5.6 Catalyst solution. Make up a 1 % solution of 4-N-dimethylaminopyridind in NMP.

5.7 Indicator solution. Make up a 1 % solution of either thymolphthalein or 0,5 % solution of phenolphthalein in NMP.

6 Apparatus

Ordinary laboratory equipment and glassware, together with the following.

6.1 Automatic titrator.

6.2 Analytical balance.

6.3 100 ml conical flask with grounded joint.

6.4 50 ml motorized piston burette.

6.5 Hot plate.

6.6 Magnetic stirrer.

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 triplicate by titrating the sample potentiometrically or using a colour indicator.

8.2 Test portion

The initial sample mass required for the determination depends on the expected hydroxyl value and shall be calculated using Equation 1:

$$m = \frac{300}{HV_e} \quad (1)$$

where

m is the initial sample mass, in grams;

HV_e, is the expected hydroxyl value, in milligrams of KOH per gram, of the product.

Weigh, to the nearest 1 mg, the test portion into the 100 ml conical flask (6.3).

8.3 Determination

Add 30 ml of catalyst solution (5.6) and 10 ml of acetylating reagent (5.5). Close the flask with the stopper and dissolve the sample by stirring and, if necessary, by heating the mixture. Then, while stirring continuously on the magnetic stirrer (6.6), allow the reaction to take place for at least 15 min at ambient temperature (23 ± 2) °C.

NOTE 1 All products containing secondary OH-groups require a reaction time of at least 60 min, and that is also found to be the case for polyols containing secondary OH-groups.

Stop the reaction by adding 3 ml of demineralized water (5.4) and stir for a further 12 min.

NOTE 2 All products containing secondary OH-groups require a hydrolysis time of at least 30 min.

Remove the flask from the stirrer, rinse the stopper and wall with methyl ethyl ketone (5.3), add 2 to 3 drops of indicator solution (5.7) and titrate with potassium hydroxide solution (5.2) until the colourless solution becomes blue (tymolphthalein) or pink (phenolphthalein).

8.4 Blank test

Carry out a blank test, following the same procedure, but omitting the test portion.

It shall be re-determined on a daily basis.

NOTE The value for the blank is around 40 ml.

8.5 Determination of acid value

Determine the acid value of the sample separately as specified in ISO 2114:2000, Method A.

9 Expression of results

Calculate the hydroxyl value HV, in milligrams of KOH per gram of the product, using Equation 2:

$$HV = \frac{(V_0 - V_1) \cdot c \cdot 56,1}{m} + AV \quad (2)$$

Where

V_0 is the volume, in millilitres, of potassium hydroxyl solution (5.2) required for the blank test (8.4);

V_1 is the volume, in millilitres, of potassium hydroxide solution (5.2) required for the determination (8.3);

