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**Binders for paints and varnishes —  
Determination of hydroxyl value —**

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

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

*Partie 1: Méthode titrimétrique sans catalyseur*

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# Contents

Page

<b>Foreword</b> .....	<b>iv</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>1</b>
<b>4 Principle</b> .....	<b>2</b>
<b>5 Reagents</b> .....	<b>2</b>
<b>6 Apparatus</b> .....	<b>3</b>
<b>7 Sampling</b> .....	<b>3</b>
<b>8 Procedure</b> .....	<b>3</b>
8.1 Number of determinations.....	3
8.2 Test portion.....	3
8.3 Determination .....	4
8.4 Blank test.....	5
8.5 Determination of acid value .....	5
<b>9 Expression of results</b> .....	<b>5</b>
<b>10 Precision</b> .....	<b>5</b>
<b>11 Test report</b> .....	<b>6</b>
<b>Bibliography</b> .....	<b>7</b>

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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.

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](http://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](http://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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](http://www.iso.org/foreword)

The committee responsible for this document is ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*.

This first edition of ISO 4629-1 cancels and replaces ISO 4629:1996, which has been technically revised with the following changes:

- 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 paints 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 —

## 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 has to be taken into account.

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

### 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, *Laboratory glassware — Burettes*

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

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 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

##### 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** [ $\text{C}_6\text{H}_4(\text{COOH})(\text{COOK})$ ].

**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 ( $\text{CH}_3\text{C}_6\text{H}_4\text{SO}_3\text{H}\cdot\text{H}_2\text{O}$ ) 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,  $c(\text{KOH}) \approx 0,5 \text{ mol/l}$ , in methanol.

Ethanol may 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

Weigh, 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 phenolphthalein solution (5.7) as indication, until a red coloration that remains for at least 10 s appears.

Calculate the actual concentration,  $c$ , in moles of hydroxyl ions ( $\text{OH}^-$ ) per litre, of the potassium hydroxide solution, using [Formula \(1\)](#):

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

where