
**Plastics — Polyols for use in the
production of polyurethane —
Determination of hydroxyl number**

*Plastiques — Polyols pour la production du polyuréthane —
Détermination de l'indice d'hydroxyle*

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ISO 14900:2001

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

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14900 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

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Introduction

International Standards have been published which deal with the determination of hydroxyl values of unsaturated-polyester resins (ISO 2554), non-ionic surface active agents (ISO 4326, ISO 4327), binders for paints and varnishes (ISO 4629) and industrial polyglycols (ISO 6796). The two methods in this document are improved versions of imidazole-catalysed procedures specifically tailored to the determination of the hydroxyl number of many types of polyol, including those used in the production of polyurethanes. Method A is especially suited to the determination of the hydroxyl number of polyether polyols that may have steric hindrance, or are otherwise difficult to determine by phthalation. Method B is a general method based on phthalation and is applicable to a wide range of polyol types. Both of these methods are similar to procedures found in ASTM D 4274.

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WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies two methods for the measurement of the hydroxyl number of polyols used as polyurethane raw materials. It is necessary to know the hydroxyl content of polyols to properly formulate polyurethane systems. Method A is primarily applicable to readily esterified polyether polyols. It is also recommended for polyols which have significant steric hindrance, such as those based on sugars. Method B is recommended for polyether polyols, polymer polyols and amine-initiated polyols, but may give low results for sterically hindered polyols. Other polyols may be analysed by these methods if precautions are taken to verify applicability. These methods can be used for research and for quality control and specification purposes.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 760:1978, *Determination of water — Karl Fischer method (General method).*

ISO 835-1:1981, *Laboratory glassware — Graduated pipettes — Part 1: General requirements.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 4788:1980, *Laboratory glassware — Graduated measuring cylinders.*

ISO 6353-1:1982, *Reagents for chemical analysis — Part 1: General test methods.*

ISO 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series.*

ISO 6353-3:1987, *Reagents for chemical analysis — Part 3: Specifications — Second series.*

3 Terms and definitions

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

3.1

polyurethane

a polymer prepared by the reaction of an organic di- or polyisocyanate with compounds containing two or more hydroxyl groups

3.2

hydroxyl number

hydroxyl value

the number of milligrams of potassium hydroxide equivalent to the hydroxyl content of 1 g of a test portion

4 Principle

4.1 Method A: A test portion is refluxed in a solution of acetic anhydride in pyridine to acetylate the hydroxyl groups present; the reaction is catalysed by imidazole. The excess reagent is hydrolysed with water and the resulting acetic acid is titrated with standardized sodium hydroxide solution. The hydroxyl content is calculated from the difference in titration of the test portion and a blank solution.

CAUTION — Acetic anhydride and pyridine are toxic and flammable. In addition, acetic anhydride is corrosive. Take proper precautions when handling these reagents.

4.2 Method B: The hydroxyl groups in a test portion are esterified by refluxing with a solution of phthalic anhydride in pyridine; the reaction is catalysed by imidazole. The excess anhydride is hydrolysed with water and the phthalic acid formed is titrated with standardized sodium hydroxide solution. The hydroxyl content is calculated from the difference in titration of the test portion and a blank solution.

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5 Interferences

5.1 Excess water will interfere by destroying part of the esterification reagent. If the sample contains more than 0,2 % water, dry the sample with a reagent that will not add acidity or basicity to the sample.

5.2 Primary and secondary amines and long-chain fatty acids react with the reagent to form stable compounds that will be included in the result.

6 Reagents

Reagent-grade chemicals shall be used in all determinations. Unless otherwise indicated, it is intended that all reagents conform to the specifications of ISO 6353-1, ISO 6353-2 and ISO 6353-3, although other grades may be used provided that it is first determined that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Unless otherwise indicated, references to water shall be understood to mean grade 2 reagent water as defined by ISO 3696:1987.

6.1 Acetylation reagent (for method A).

Mix 127 ml of acetic anhydride with 1 000 ml of dry pyridine (6.7). Add 16 g of imidazole (6.3) and swirl carefully to dissolve. Prepare the reagent fresh daily and keep it in a dark bottle. Do not use it if it is darker than pale yellow.

NOTE Some laboratories have reported that dark, resinous solids have formed when the mixture of pyridine and esterification reagent is heated. In that event, it has been found that preparing the acetylation reagent with the addition of 0,4 %

water in the pyridine (6.8) will correct the problem. However, care has to be taken that sufficient reagent is present to complete the reaction quantitatively. See also 8.1.2.

CAUTION — Acetic anhydride and pyridine are eye, skin, and respiratory irritants. Avoid bodily contact with these reagents and use only in a well-ventilated area.

6.2 Phthalation reagent (for method B).

Weigh 116 g of phthalic anhydride into a 1 litre brown bottle. Add 700 ml of pyridine (6.7) and shake vigorously until dissolved. Add 16 g of imidazole and swirl carefully to dissolve. The reagent shall stand overnight before use. Avoid prolonged exposure of the reagent to moisture in the air. Discard reagent that develops a colour. In the blank titration as described in the procedure for method B, exactly 25 ml of this reagent shall consume between 95 ml and 100 ml of 0,500 mol/l sodium hydroxide.

6.3 Imidazole, reagent grade or equivalent.

6.4 Hydrochloric acid, standard solution, 0,1 mol/l.

Prepare and standardize to four significant figures in accordance with good practice, using potassium acid phthalate (6.6) as a primary standard. Determine and record the temperature at which the standardization was performed. The concentration of the solution shall be corrected to the temperature at which the determination is performed, as described in 6.9. This solution is required only if a correction is to be applied for the presence of strong base in the sample being analysed.

6.5 Phenolphthalein indicator solution, 10 g/l.

Prepare a solution of 1 g of phenolphthalein in 100 ml of pyridine (6.7).

6.6 Potassium acid phthalate.

Use a certified primary standard.

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6.7 Pyridine, reagent grade, containing less than 0,1 % water.

If purification is required, distil from phthalic anhydride, discarding the fraction boiling below 114 °C to 115 °C.

6.8 Pyridine, containing from 0,30 % to 0,45 % water (used for special cases: see the note to 6.1).

Determine the water content of the pyridine using ISO 760. Add the required amount of water. The required volume of water to add per litre of pyridine may be calculated as follows:

$$\text{Water to add, in ml} = 4,0 - 9A$$

where A is the percent water already in the pyridine.

6.9 Sodium hydroxide, standard solution, 0,5 mol/l.

Prepare and standardize to four significant figures in accordance with good practice. Determine and record the temperature at which the standardization was performed. The factor for thermal expansion of this solution is 0,000 14. For calculation of the hydroxyl content, the concentration of the solution shall be corrected to the temperature at which the determination was performed by the following:

$$c_{\theta_2} = c_{\theta_1} + F(\theta_1 - \theta_2)$$

where

c_{θ_1} is the concentration of the solution when standardized, in mol/l;

c_{θ_2} is the concentration during analysis of samples, in mol/l;