INTERNATIONAL STANDARD

ISO 14899

First edition
2001-02-15

Plastics — Polyols for use in the production of polyurethane — Determination of basicity

Plastiques — Polyols pour la production du polyuréthanne — Détermination de la basicité

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Reference number
ISO 14899:2001(E)

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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 14899 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

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Introduction

This method is for the determination of trace amounts of basicity in polyether polyols, which are used in the preparation of polyurethane prepolymers and polyurethane products. Knowledge of this value is important to prevent gelation during prepolymer production and to control reaction rates during polyurethane preparation. The method, known as the CPR (controlled polymerization rate) analysis, has become an accepted industry practice, a version of which has been published as part of JIS K 1557.

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Plastics — Polyols for use in the production of polyurethane — Determination of basicity

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.

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1 Scope

This International Standard specifies a method for the measurement of trace amounts of basic materials present in polyether polyols used in the production of polyurethanes. It is important to know the trace amount of basicity in a polyol to prevent gelation of the reaction mass during the production of polyurethane prepolymers. It is also useful to control the basicity in polyols used for polyurethane production to assure consistent and reproducible reaction behaviour. This method is suitable for quality control, as a specification test and for research.

The applicable range is 0 µg to 50 µg/g, expressed as KOH. The method is not applicable to amine-based polyols. The values may be reported as CPR (controlled polymerization rate) units.

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

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

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3.2
CPR value
controlled polymerization rate value
the number of microequivalents of base in a 30 g test portion of polyol (i.e. meq of base in 30 kg of polyol)

4 Principle

A 30 g test portion of polyol is diluted with methanol and titrated with aqueous 0,01 mol/l HCl. The results are compared with a blank titration of the methanol.

5 Sampling

Draw samples from a well-mixed vessel into a thoroughly cleaned and dry borosilicate glass container (soft glass containers are not acceptable). If sampling from a line or valve, flush the line thoroughly with the product before starting to draw the sample. Seal the sample until analysis.

6 Interferences

Any acidic or basic materials inadvertently introduced into the sample will cause errors in the analysis. Any material capable of serving as a buffer may interfere with the analysis by obscuring the titration end point. Some samples may contain traces of several different compounds which may have the effect of causing multiple breaks in the titration curve, making interpretation difficult. This analysis is not applicable to amine-based polyols.

7 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.

7.1 Hydrochloric acid, standard solution, 0,01 mol/l.

Prepare and standardize in accordance with good practice, using potassium acid phthalate (7.2) as a primary standard. Standardize to detect changes of 0,000 1 mol/l.

7.2 Potassium acid phthalate.

Use a certified primary standard.

7.3 Methanol, reagent grade, conforming to ISO 6353-2.

8 Apparatus

8.1 Autotitrator, capable of determining multiple end points, equipped with a pair of electrodes or a combination glass calomel electrode, a 5 ml burette and a recorder.

8.2 Burette, or other automatic dispensing device, capable of dispensing 50 ml ± 0,1 ml.

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8.3 **Balance**, capable of weighing 30 g test portions to ±1 mg.

8.4 **Titration flask**, 100 ml, or other suitable titration vessel.

8.5 **Magnetic stirrer**, equipped with an inert stirrer bar, or equivalent.

9 Procedure

9.1 Set up the titrator (8.1) for titrations having a maximum titrant volume of 5 ml.

9.2 Add 50 ml of methanol (7.3) to a 100 ml titration flask (8.4) for use as a solvent blank.

NOTE Some popular automatic titrimeters are equipped with 100 ml titration vessels. With other titrimeters, an acceptable variation of the method is to use 100 ml of methanol in a 150 ml titration vessel, as is done in JIS K 1557.

9.3 Titrate the solvent blank with 0,01 mol/l HCl (7.1) and record the volume of titrant used. The end point is taken as the point of inflection of the last end point on the titration curve. The blank should consume less than 0,2 ml of 0,01 mol/l HCl.

9.4 Into a 100 ml titration flask, weigh about 30 g of sample to the nearest 1 mg. Add 50 ml of methanol and stir until well mixed. (See the note to 9.2.)

9.5 Titrate with 0,01 mol/l HCl and record the volume of titrant up to the last end point.

NOTE Depending on the sample being analysed, as many as three inflection points may be seen. Use the last end point.

10 Expression of results

10.1 Calculate the CPR value using the following equation:

$$\text{CPR value} = (V_S - V_B)M \times 1000 \times \frac{30}{m}$$

where

V_S is the volume of HCl needed to titrate the test portion, in ml;

V_B is the volume of HCl needed to titrate the blank, in ml;

M is the molarity of the HCl, in mol/l;

m is the mass of the test portion, in g.

10.2 The basicity may also be calculated as µg KOH/g of sample using the following equation:

$$\text{µg KOH/g of sample} = (V_S - V_B)M \times 1000 \times \frac{56{,}1}{m}$$

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