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

Plastiques — Polyols pour la production de polyuréthannes — Détermination de la basicité

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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).

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 14899:2001), of which it constitutes a minor revision. The changes are as follows:

— the title has been changed to plural form to read: "Plastics — Polyols for use in the production of polyurethanes — Determination of basicity".

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 controlled polymerization rate (CPR) analysis, has become an accepted industry practice, a version of which has been published as part of JIS K 1557.

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

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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 prior to the application of this standard.

1 Scope

This document 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 can be reported as CPR (controlled polymerization rate) units.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

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

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

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
polyurethane
polymer prepared by the reaction of an organic di- or polyisocyanate with compounds containing two or more hydroxyl groups

3.2
CPR value
controlled polymerization rate value
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 can interfere with the analysis by obscuring the titration end point. Some samples can contain traces of several different compounds which can 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, all reagents shall be in accordance with 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.

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.

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 can be seen. Use the last end point.

10 Expression of results

10.1 Calculate the CPR value using Formula (1):

$$C = (V_S - V_B)M \times 1\ 000 \times \frac{30}{m}$$

where

C is the controlled polymerization rate (CPR) value;

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 Formula (2):

$$N = \left(V_S - V_B\right)M \times 1\ 000 \times \frac{56,1}{m}$$

where N is the basicity, µg KOH/g.

11 Precision and bias

11.1 Precision

11.1.1 Use the criteria for repeatability and reproducibility shown in 11.1.2 and 11.1.3 to judge the acceptability of results.

NOTE Precision data were determined by a round-robin test conducted by eight laboratories using five different commercially available polyol samples covering a range of 0,3 CPR units to 1,2 CPR units. Data can be obtained from ASTM Committee D-20 or from the PURMAC committee of the American Plastics Council.

11.1.2 Repeatability (single analyst): Duplicate results obtained by the same analyst using the same equipment on the same day should only be considered suspect if they differ by more than 0,2 CPR units. This repeatability was calculated from pooled experimental data following ASTM E 180.

11.1.3 Reproducibility (multilaboratory): Results, each the mean of duplicates, obtained on identical test materials in separate laboratories should only be considered different if they differ from that of another laboratory by more than 0,6 CPR units. This reproducibility was calculated from pooled experimental data following ASTM E 180.

11.2 Bias

Bias is the difference between the expectation of the test results and an accepted reference value. The bias of this test has not been determined.

12 Test report

The test report shall include the following:

a) a reference to this document, i.e. ISO 14899:2022;

b) all details necessary to identify the product analysed;

c) the results obtained, expressed as CPR units to the nearest 0,1 CPR unit, or as µg KOH/g;

d) any incident or detail not stipulated in this document which might have influenced the result;

e) the date of the analysis.