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ISO 17710

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Plastics — Polyols for use in the production of polyurethane — Determination of degree of unsaturation by microtitration

Plastiques — Polyols pour la production du polyuréthanne — Détermination du degré de non-saturation par microtitrage

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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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Contents

Page

iTeh STANDARD PREVIEW
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ISO 17710:2002
https://standards.iteh.ai/catalog/standards/sist/381dc6c6-d8ec-40c5-9519-c73fb46a5980/iso-17710-2002

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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 17710 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

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Introduction

Standards have been published which deal with the measurement of the degree of unsaturation in polyols used for the production of polyurethane plastics (ASTM D 4671, JIS K 1557, part 6.7). These standards are based on the reaction of mercuric acetate with the unsaturation present in the molecule. The method described in this International Standard relies on the same chemistry, but is a microtitration method which uses less reagent and therefore reduces the disposal problems associated with mercury compounds. It is based primarily on ASTM D 4671.

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Plastics — Polyols for use in the production of polyurethane — Determination of degree of unsaturation by microtitration

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 of this standard to establish appropriate safety and health practices, and to ensure compliance with any national regulatory conditions prior to use.

1 Scope

This International Standard specifies a microtitration method to measure the degree of unsaturation in polyether polyols used in the production of polyurethanes. It is based on the reaction of mercuric acetate with double bonds in the polyol. It does not apply to compounds in which the unsaturation is conjugated with carbonyl, carboxyl or nitrile groups. The product being measured must be essentially dry and free of inorganic salts, especially halides.

2 Normative references

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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.

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

ISO 4787:1984, *Laboratory glassware — Volumetric glassware — Methods for use and testing of capacity*

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
unsaturation
property of a compound or polymer distinguished by the presence of a carbon-to-carbon double bond

3.2
polyol
organic compound which contains two or more hydroxyl groups capable of reacting with isocyanates to form polyurethanes

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3.3
polyurethane
polymer prepared from the reaction of an organic di- or polyisocyanate with compounds containing two or more hydroxyl groups

4 Principle

Carbon-to-carbon unsaturated compounds in the sample are reacted with mercuric acetate and methanol in a methanolic solution to produce acetoxymercuricmethoxy compounds and acetic acid. The amount of acetic acid released is determined by microtitration with standard alcoholic potassium hydroxide and the result used to calculate the amount of unsaturation originally present. Because the acid cannot be titrated in the presence of excess mercuric acetate, sodium bromide is added to convert the mercuric acetate to the corresponding bromide, which does not interfere with the titration. A suitable correction must be applied if the sample is not neutral to phenolphthalein indicator. Carbon dioxide must be excluded from the reaction.

5 Application

Side reactions in polymerizations based on propylene oxide produce small amounts of polymers with only one hydroxyl group per chain. These unsaturated polymers lower functionality and molecular mass, thus changing the overall molecular mass distribution. This test method is suitable for quality control, as a specification test, and for research.

6 Interferences

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This test method does not apply to compounds in which the unsaturation is conjugated with carbonyl, carboxyl or nitrile groups. The system must be essentially free of water and inorganic salts, especially halides. Acetone in low concentrations does not interfere significantly, although its presence may make the end point less distinct.

ISO 17710:2002
https://standards.iteh.ai/catalog/standards/sist/381dc6c6-d8ec-40c5-9519-c73fb46a5980/iso-17710-2002

7 Reagents

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of ISO 6353-1, ISO 6353-2 and ISO 6353-3. 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 Methanolic mercuric acetate solution, $c = 0{,}05$ mol/l.

Dissolve 16 g of mercuric acetate [$Hg(C_2H_3O_2)_2$] into 1 litre of reagent grade methanol and add sufficient glacial acetic acid to require a blank titration of 0,5 ml to 1 ml of 0,05 mol/l methanolic KOH for a 2 ml aliquot. Usually several drops of acid are required. Prepare the reagent fresh weekly and filter before using.

WARNING — Mercury compounds are highly toxic. Handle all mercury-containing reagents and waste solutions with care. Handle waste solutions as hazardous materials, and dispose of wastes in accordance with good laboratory practice and in accordance with applicable regulations.

7.2 Methanolic potassium hydroxide solution, $c = 0{,}05$ mol/l.

Prepare and standardize in accordance with good practice, using potassium acid phthalate as a primary standard.

7.3 Methanolic hydrochloric acid solution, $c = 0{,}05$ mol/l.

Prepare by successively diluting concentrated hydrochloric acid with methanol. This will introduce less than 0,5 % water into the titration reagent. Standardize by titrating against 0,05 mol/l methanolic KOH (7.2).

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7.4 **Sodium bromide** (NaBr).

7.5 **Methanol**.

8 Apparatus

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

8.2 **Analytical balance**, capable of weighing samples to 0,1 mg.

8.3 **Pipette**, 2 ml capacity, conforming to ISO 4787.

8.4 **Titration vessel**, 50 ml to 100 ml capacity.

9 Sampling

Samples shall be drawn from a well-mixed vessel into a thoroughly cleaned and dry borosilicate glass container (soft-glass containers are not acceptable). If drawing from a line or valve, flush the line thoroughly with the product before starting to draw the sample. Seal the sample until taking a test portion for analysis. Care shall be taken to exclude excess moisture and carbon dioxide.

10 Procedure

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10.1 This method requires at least a two-fold molar excess of mercury reagent for quantitative reaction of the unsaturated species. If the test portion is too large, the method will give inaccurate (low) results as well as reduced precision. Use no more than 0,033 milli-equivalents (meq) of unsaturated species for the analysis therefore. For samples having 0,033 meq or less of unsaturation per gram of sample, weigh approximately 1 g of sample (weighed to 0,1 mg) into a 100 ml titration flask. If the unsaturation value is not known, determine an approximate value by using a 1 g test portion. Use this approximate value to calculate a correct test portion size that will contain no more than 0,033 meq of unsaturation from the following relationship:

ISO 17710:2002
https://standards.iteh.ai/catalog/standards/sist/581dc0c6-d8ec-40c5-9519-c73ab4ba5980/iso-17710-2002

$$\text{Test portion size (in grams)} = \frac{0{,}033}{\text{Approx. sample unsaturation}}$$

10.2 Add 2 ml of mercuric acetate solution (7.1) and swirl to dissolve the test portion completely. Cover with a watch glass and allow the solution to stand for thirty minutes. Add 50 ml of methanol (7.5) followed by 0,25 g of sodium bromide crystals (7.4).

10.3 Titrate using 0,05 mol/l methanolic KOH (7.2) to the end point using an automatic titrator (8.1).

10.4 Titrate a blank using the same procedure, but without adding a test portion.

10.5 To determine the acidity or basicity of the polyol in order to correct the results, prepare a test portion exactly as above, but omit the mercuric acetate. Titrate, as above, with 0,05 mol/l methanolic KOH to the potentiometric end point. If the solution is determined to be already past the end point, repeat this procedure, but titrate with 0,05 mol/l methanolic HCl (7.3).

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