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ISO TC 61/SC 12/WG 6

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

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Foreword

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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 17710:2002), 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 degree of unsaturation by microtitration"

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.

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

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 mustshall be applied if the sample is not neutral to phenolphthalein indicator. Carbon dioxide mustshall 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

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

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 7.1 Methanolic mercuric acetate solution, $C = 0,05$ mol/l.

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