



Standard Test Methods for Polyurethane Raw Materials: Determination of Unsaturation of Polyols¹

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1. Scope *

1.1 These test methods measure unsaturation in polyether polyols. (See Note 1.)

1.1.1 *Test Method A, High-Volume Reagent Method*—Uses about 50 mL of 0.1 M mercuric acetate reagent in methanol and 15 g or more of sample. This test method uses an indicator for colorimetric determination of an end point. It is recommended for polyols with low values (below 0.01 millequivalents per gram (meq/g)) of unsaturation where large sample sizes are required.

1.1.2 *Test Method B, Low-Volume Reagent Method*—Uses 2 mL of ca. 0.05 M mercuric acetate reagent in methanol and about 1 g of sample or less. This test method uses a potentiometric determination of an end point.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—There is no equivalent ISO standard.

2. Referenced Documents

2.1 ASTM Standards:

D 883 Terminology Relating to Plastics²

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals³

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, see Terminology D 883.

¹ These test methods are under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.22 on Cellular Plastics. Test Method A was recommended to ASTM by the Society of the Plastics Industry Polyurethane Raw Materials Analysis Committee.

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² *Annual Book of ASTM Standards*, Vol 08.01.

³ *Annual Book of ASTM Standards*, Vol 15.05.

4. Summary of Test Method

4.1 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.⁴ The amount of acetic acid released in this equimolar reaction, which is determined by titration with standard alcoholic potassium hydroxide, is a measure of the unsaturation originally present. Because the acid cannot be titrated in the presence of excess mercuric acetate, due to the formation of insoluble mercuric oxide, sodium bromide is added to convert the mercuric acetate to the bromide, which does not interfere. Inasmuch as this test method is based on an acidimetric titration, a suitable correction must be applied if the sample is not neutral to phenolphthalein indicator. Take care to exclude carbon dioxide, which titrates as an acid and gives erroneous results.

5. Significance and Use

5.1 These test methods are suitable for quality control, as specification tests, and for research.

5.2 Side reactions that form unsaturated compounds in polypropylene oxides produce small amounts of polymers with only one hydroxyl group per chain. These unsaturated polymers lower functionality and molecular weight, while broadening the overall molecular-weight distribution.

6. Interferences

6.1 These test methods do not apply to compounds in which the unsaturation is conjugated with carbonyl, carboxyl, or nitrile groups. Because water presumably hydrolyzes the reaction products to form basic mercuric salts, quantitative results are obtained only when the system is essentially anhydrous. Acetone in low concentrations does not interfere significantly, although its presence may be detrimental to the end point. Inorganic salts, especially halides, must be absent from the

⁴ Sigia, S. and Hanna, J.G., “Quantitative Organic Analysis via Functional Groups,” John Wiley and Sons, New York, 1979.

*A Summary of Changes section appears at the end of this standard.