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Designation: ~~D4672 – 18~~ D4672 – 24

Standard Test Method for Polyurethane Raw Materials: Determination of Water Content of Polyols [1]

This standard is issued under the fixed designation D4672; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method measures the water content of polyols and many other organic compounds.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

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NOTE 1—This test method is equivalent to ISO 14897.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

ASTM D4672-24
https://standards.iteh.ai/catalog/standards/astm/f9c7576a-e99a-4f58-a2ea-841708ba38ff/astm-d4672-24

2.1 *ASTM Standards:*[2]

~~D1193 Specification for Reagent Water~~
D883 Terminology Relating to Plastics
E203 Test Method for Water Using Volumetric Karl Fischer Titration
E456 Terminology Relating to Quality and Statistics
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E2935 Practice for Evaluating Equivalence of Two Testing Processes

2.2 *ISO Standards:*[3]

ISO 14897 Plastics—Polyols for use in the production of polyurethane—Determination of water content

3. Terminology

3.1 ~~*Definitions:*~~*Definitions*—Terms used in this standard are defined in accordance with Terminology D883, unless otherwise specified. For terms relating to precision and bias and associated issues, the terms used in this standard are defined in accordance with Terminology E456.

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[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.22 on Cellular Materials - Plastics and Elastomers.

Current edition approved ~~April 1, 2018~~Feb. 1, 2024. Published ~~April 2018~~February 2024. Originally approved in 1991. Last previous edition approved in ~~2012~~2018 as ~~D4672 - 12.~~D4672 - 18. DOI: ~~10.1520/D4672-18.~~10.1520/D4672-24.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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

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3.1.1 *polyurethane, n*—a polymer prepared by the reaction of an organic diisocyanate with ~~compounds containing hydroxyl groups.~~polyols.

3.1.1.1 *Discussion*—

Polyurethanes, or urethanes, as they are sometimes called, can be thermosetting, thermoplastic, ~~rigid or soft and~~ rigid, flexible, cellular or solid. (See Terminology D883.)

4. Summary of Test Methods

4.1 This method is based ~~essentially~~ on volumetric or coulometric titrations that follow the reduction of iodine by sulfur dioxide in the presence of water. ~~This reaction proceeds quantitatively when methanol or another~~ An alcohol (ROH) and ~~pyridine (C~~an ~~$_5H_5N$) or a similar~~ amine (R'N) ~~are present to~~ react with ~~the~~ sulfur ~~trioxide~~dioxide (SO_{3}~~$_2$~~) ~~and hydriodic acid (HI) produced~~ to form an intermediate that is then oxidized by the stoichiometric reaction of iodine and water in the presence of the amine according to the following reactions:

$ROH + SO_2 + R'N \rightarrow [R'NH]SO_3R$

$H_2O + I_2 + [R'NH]SO_3R + 2R'N \rightarrow [R'NH]SO_4R + 2[R'NH]I$

4.2 ~~To determine water, Karl Fischer reagent (a solution of iodine, sulfur dioxide, imidazole, and pyridine or a pyridine substitute) is added to a solution of the sample in methanol or other alcohol until all of the water present has been consumed. The titrant is either added~~ Pyridine was historically used but has been displaced by imidazole and other amines. Methanol is a commonly used alcohol, but others are available. In Karl Fischer titrations, iodine is either added as part of the titrant by buret (volumetry) or is generated electrochemically in the titration cell (coulometry). Coulometric titrations eliminate the need for standardizing the reagent. ~~Pyridine is less commonly used recently due to its toxicity. If pyridine is to be used, refer to the SDS for proper precautions.~~

4.3 This method provides details specific to water determinations in polyols. ~~General guidance to the use of Karl-Fischer~~A general procedure for volumetric Karl Fischer analysis, including a list of interferences, can be found in Test Method E203.

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5. Significance and Use

5.1 This test method is suitable for quality control, as a specification test, and for research. The water content of a polyol is important since isocyanates react with water.

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6. Apparatus

6.1 Several commercial Karl Fischer autotitrators are available that employ volumetric or coulometric titrations. These instruments consist of an automated buret assembly, a sealed titration vessel with appropriate electrodes and sensing circuitry, and a vacuum system for removal of solution after analysis. These automated systems provide several advantages and conveniences. Atmospheric moisture contamination can be more closely controlled; calibration is simplified; and the preneutralization step is automatic. Titrations are rapid, and reagent consumption is low. Autotitrators automatically calculate and display or print the water concentration.

7. Reagents

7.1 Commercial reagents and reagent systems of various types are available for use with autotitrators for water determination. Pyridine-free reagents have improved stability and less objectionable odor than the conventional Karl Fischer reagent. Reagents are available in split or composite forms in different concentrations to fit various ranges of water content. A *composite* reagent contains all the components required for a Karl Fischer titration in a single solution. *Split* implies separate solutions of the solvent and titrant.

8. Sampling

8.1 Sampling is conveniently accomplished by use of a tared syringe. The material is drawn into the syringe, weighed, and delivered through the sample port of the autotitrator vessel. The syringe is then reweighed to obtain the sample weight by difference.

8.1.1 It is essential to avoid changes in the water content of the material during sampling operations. Many polyols are quite