Designation: ~~D4672 – 12~~ D4672 – 18

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

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

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

~~E180~~E691 Practice for ~~Determining~~ Conducting an Interlaboratory Study to Determine the Precision of ~~ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals~~a Test Method ~~(Withdrawn 2009)~~

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:*

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

3.1.1.1 *Discussion*—

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

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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.

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[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] ~~The last approved version of this historical standard is referenced on www.astm.org.~~Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[4] ~~Instruments similar to and including the following types have been found suitable for determining water content of polyols, based on round-robin studies: Metrohm models 633, 652, 658, 665, 684, 701, 720, 737, and 758 (available from Brinkmann Instruments, Inc. at www.brinkmann.com) and Mettler Toledo models DL 18, 31, 37, and 38 (www.mt.com).~~

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

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 alcohol (ROH) and pyridine (C_5H_5N) or a similar amine (R'N) are present to react with the sulfur trioxide (SO_3) and hydriodic acid (HI) produced 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 by buret (volumetry) or 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 analysis, including a list of interferences, can be found in Test Method E203.

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.

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 ~~can be purchased~~ 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 hygroscopic and errors from this source are particularly significant in the determination of the small amount of water usually present. Use almost-filled, tightly capped containers and limit as much as possible contact of the sample with air when transferring the sample to the titration vessel. Avoid intermediate sample containers, if possible. If several different analyses are to be performed on the same sample, determine the water first and do not open the sample prior to the actual analysis.

8.1.2 In particularly humid environments, the autotitrator can be set up in a dry air or nitrogen purge box so that sample handling and the titration can be carried out in a low humidity environment.

9. Standardization of Reagent

9.1 Since different autotitrators ~~may~~ vary in standardization procedures, consult the operating manual for the autotitrator in use. Water is an excellent primary standard. In addition, stable, prepackaged, primary standards are also available for establishing the standardization factor.

10. Procedure

10.1 Refer to the operating manual for the autotitrator in use. Basically, after preneutralization of the reagent in the titrator vessel, the sample is introduced, and the volumetric titration (or coulometric generation of titrant) proceeds automatically to the end point.

NOTE 2—In choosing the appropriate sample size for use with specific autotitrators, use the manufacturer's recommendations. If no instructions are available, use the guidelines listed in Table 1 and Table 2.