



Designation: D4672 – 12

# Standard Test Method for Polyurethane Raw Materials: Determination of Water Content of Polyols<sup>1</sup>

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 ( $\epsilon$ ) 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 and health practices and determine the applicability of regulatory limitations prior to use.*

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

## 2. Referenced Documents

2.1 *ASTM Standards*:<sup>2</sup>

D1193 [Specification for Reagent Water](#)

D883 [Terminology Relating to Plastics](#)

E180 [Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals](#) (Withdrawn 2009)<sup>3</sup>

## 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 be thermosetting, thermoplastic, rigid or soft and flexible, cellular or solid. (See Terminology D883.)

<sup>1</sup> 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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<sup>2</sup> 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.

<sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

## 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<sub>5</sub>H<sub>5</sub>N) or a similar amine (R'N) are present to react with the sulfur trioxide (SO<sub>3</sub>) and hydriodic acid (HI) produced according to the following reactions:



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 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.

## 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<sup>4</sup> 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.

<sup>4</sup> 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