Designation: D4669 – 07 (Reapproved 2013)ε1

Standard Test Method for Polyurethane Raw Materials: Determination of Specific Gravity of Polyols [1]

This standard is issued under the fixed designation D4669; 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.

ε1 NOTE—Reapproved with editorial changes in April 2013.

1. Scope*

1.1 These test methods measure the specific gravity of polyols. (See Note 1.)

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 known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

E202 Test Methods for Analysis of Ethylene Glycols and Propylene Glycols

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods see Terminology D883.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *specific gravity*—the ratio of the weight in air of a given volume of the material at a stated temperature to the weight in air of an equal volume of water at a stated temperature. It shall be expressed as specific gravity, 25/25°C, indicating that the sample and reference water were both measured at 25°C.

4. Significance and Use

4.1 These test methods are suitable for quality control, specification testing, and research. The specific gravity is necessary when converting kinematic viscosity to absolute viscosity.

TEST METHOD A—SPECIFIC GRAVITY USING A PYCNOMETER

5. Apparatus

5.1 *Pycnometer*, of 25 or 50-mL capacity, conical shape with a capillary side arm overflow tube complete with a standard-taper 5/12 ground-glass joint to receive a ground-glass vented cap. A thermometer with a scale graduated from 12 to 38°C in 0.2-degree divisions joins the neck of the flask with a standard-taper 10/18 ground-glass joint. The thermometer contained in the pycnometer shall be calibrated in accordance with Specification E2251.

5.2 *Water Bath*, capable of maintaining a temperature of 25.0 ± 0.05°C during the test.

5.3 *Thermometer*, an ASTM Low Softening Point Thermometer having a range from -2 to +80°C and conforming to the requirements for Thermometer S15C as prescribed in Specification E2251.

5.4 *Analytical Balance*, sensitive to 0.1 mg.

6. Reagents

6.1 *Chromic Acid Cleaning Solution* —Prepare a saturated solution of chromic acid (CrO_3) in concentrated sulfuric acid (H_2SO_4, sp gr 1.84).

6.2 *Acetone,* reagent grade

7. Sampling

7.1 High molecular weight polyester and polyether polyols contain molecules covering an appreciable range of molecular weights. These have a tendency to fractionate during solidification. Unless the material is a finely ground solid it is necessary to melt (using no higher temperature than necessary) and mix the polyol well before removing a sample for analysis.

[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.22 on Cellular Materials - Plastics and Elastomers. It was recommended to ASTM by the Center for the Polyurethane Industry of the American Chemistry Council.

Current edition approved April 1, 2013. Published April 2013. Originally approved in 1987. Last previous edition approved in 2007 as D4669 – 07. DOI: 10.1520/D4669-07R13E01.

[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.

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

Since many polyols are hygroscopic, take care to provide minimum exposure to atmospheric moisture during the sampling.

8. Procedure

8.1 Clean the pycnometer by filling it with a chromic acid cleaning solution. Allow it to stand for a few hours, empty, and rinse well with distilled water.

8.2 Fill the pycnometer with freshly boiled distilled water cooled to 22 to 24°C, and set the pycnometer thermometer in place carefully, avoiding trapping of air. Place the pycnometer in the water bath that has been maintained at 25.0 ± 0.05°C for at least 30 min and allow it to equilibrate to 25.0°C. Wipe the overflow from the side-arm capillary and cover with the vented cap, remove from the bath, wipe dry, and weigh.

8.3 Empty the pycnometer, rinse well with acetone and dry under vacuum for 15 min. Weigh the pycnometer and subtract the weight of the empty pycnometer from the weight when filled with water in order to obtain the weight, W, of the contained water at 25°C in air.

8.4 The sample for test must be completely liquid. If the sample contains solid polyol, warm the entire sample in the original container until it becomes liquid. Then cool the sample to 22 to 24°C and quickly fill the pycnometer with it, allowing it minimal exposure to the atmosphere.

8.5 Insert the thermometer carefully, avoiding trapping of air. Place the pycnometer in the water bath that has been maintained at 25.0 ± 0.05°C for at least 30 min and allow it to equilibrate to 25.0°C. Cover the side arm with the vented cap, remove from the bath, wipe dry, and weigh. Subtract the weight of the empty pycnometer from the weight when filled with the sample in order to obtain the weight, S, of the contained sample at 25.0°C.

9. Calculation

9.1 Calculate the specific gravity at 25/25°C as follows:

$$S/W = \textit{Specific gravity}, 25/25°C$$

where:

S = sample used, g, and
W = water in the pycnometer, g.

10. Precision and Bias

10.1 Attempts to develop a precision and bias statement for this test method have not been successful. Data on precision and bias cannot be given for this reason. Anyone wishing to participate in the development of precision and bias data should contact the Chairman, Subcommittee D20.22 (Section D20.22.01), ASTM 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

10.2 Test Methods E202 is similar and does include precision data. Precision statements from Test Methods E202 are restated below as an estimate of the precision for this test method.

10.2.1 *Repeatability*—It has been estimated that duplicate results by the same analyst on different days should be considered suspect if they differ by 0.0002 units based on a 95 % confidence interval.

10.2.2 *Reproducibility*—It has been estimated that results reported by different laboratories should be considered suspect if they differ by 0.0005 units based on a 95 % confidence interval.

10.3 *Bias*—There are no recognized standards by which to estimate the bias of this test method.

TEST METHOD B—SPECIFIC GRAVITY USING A DIGITAL DENSITY METER

11. General

11.1 A general test method for specific gravity using a digital density meter, which applies to polyols as well as other liquids is published in Test Method D4052.

12. Keywords

12.1 density; polyols; polyurethane raw materials; specific gravity

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D4669 – 07) that may impact the use of this standard. (April 1, 2013)

(1) In 2.1, changed reference from ASTM E1 to E2251 to allow for use of non-mercury thermometers.
(2) In 4.1, changed grammar to reflect multiple methods and clarify statement.
(3) In 5.1, updated reference to ASTM E2251.
(4) In 5.3, updated thermometer name and reference.
(5) In 6.2, added the solvent to the list of reagents.
(6) In 7.1, made editorial changes for clarity.
(7) In 8.2, expanded explanation of procedure for clarity.
(8) In 8.3, replaced ether and alcohol solvents with acetone.
(9) In 8.4, expanded explanation of procedure for clarity.
(10) In 10.2.1, expanded statement for clarity.
(11) In 10.2.2, corrected a typographical error and expanded statement for clarity.