



Designation: D 4669 – 98

Standard Test Method for Polyurethane Raw Materials: Determination of Specific Gravity of Polyols¹

This standard is issued under the fixed designation D 4669; 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 specific gravity of polyols using a pycnometer. (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 equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics²

E 1 Specification for ASTM Thermometers³

E 202 Test Methods for Analysis of Ethylene Glycols and Propylene Glycols.⁴

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method see Terminology D 883.

3.2 *Description of Term 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 This test method is suitable for quality control, as a specification test, and for research. It is necessary when converting from kinematic to absolute viscosity.

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 E 1.

5.2 *Water Bath*, capable of maintaining a temperature of $25.0 \pm 0.05^\circ\text{C}$ 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 15°C as prescribed in Specification E 1.

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).

7. Sampling

7.1 Polyesters and polyethers usually 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 resin well before removing a sample for analysis. 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 $25.0 \pm 0.05^\circ\text{C}$ for at least 30 min. Wipe the overflow from the side-arm capillary

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.22 on Cellular Plastics. It 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 14.03.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.



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and cover with the vented cap, remove from the bath, wipe dry, and weigh.

8.3 Empty the pycnometer, rinse successively with alcohol and ether, remove the ether vapor, 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. 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:

$$S/W = \text{Specific gravity, 25/25}^\circ\text{C}$$

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 E 202 is similar and does include precision data. Precision statements from Test Methods E 202 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 should be considered suspect if they differ by 0.0002 units.

10.2.2 *Reproducibility*—It has been estimated that results reported by different laboratories should be considered suspect if they differ by 0.0008 units.

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

11. Keywords

11.1 density; polyols; polyurethane raw materials; specific gravity

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D-20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

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(1) *Section 2.1*—Added references to Test Methods E 202.

(2) *Footnotes*—Inserted footnote 4.

(3) *Section 10*—Replaced the precision and bias statement with quote and reference to the precision and bias statement of Test Methods E 202.

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