Designation: D4659 – 14

Standard Test Methods for Polyurethane Raw Materials: Determination of Specific Gravity of Isocyanates [1]

This standard is issued under the fixed designation D4659; 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 These test methods determine the specific gravity of toluenediisocyanate and crude methylene-bis-(4-phenylisocyanate). These test methods also are applicable to many other liquids. (See Note 1.)

1.1.1 *Test Method A*—Specific gravity by pycnometer, for high-accuracy determination.

1.1.2 *Test Method B*—Specific gravity by hydrometer, for a less accurate, but rapid, determination.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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. For specific warning and precautionary statements see Section 7.*

NOTE 1—There is no known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics

D891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals

D1193 Specification for Reagent Water

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

E100 Specification for ASTM Hydrometers

E202 Test Methods for Analysis of Ethylene Glycols and Propylene Glycols

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

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 can be used for research or for quality control to characterize isocyanates used in polyurethane products.

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

TEST METHOD A—SPECIFIC GRAVITY BY PYCNOMETER

5. Apparatus

5.1 *Pycnometer,* of 25 or 50-mL capacity, conical shape with a capillary side arm overflow tube having a standard-taper 5/12 ground-glass joint and a ground-glass vented cap. A thermometer graduated from 12 to 38°C in 0.2° divisions attached to the neck of the flask by a standard-taper 10/18 ground-glass joint. This thermometer is to be calibrated using the ASTM thermometer specified in 5.3.

5.2 *Water Bath*—A water bath maintained at 25 ± 0.05°C.

5.3 *Thermometer*—An ASTM low-softening point thermometer, calibrated from −2 to +80°C, which meets the requirements for Thermometer S15C in Specification E2251.

5.4 *Analytical Balance*—A balance having a sensitivity of at least 0.1 mg.

[1] These test methods are 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 March 1, 2014. Published March 2014. Originally approved in 1987. Last previous edition approved in 2009 as D4659 - 09. DOI: 10.1520/D4659-14.

[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**

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6. Reagents and Materials

6.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[3] Other grades can be used, provided it is ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV or better of Specification D1193.

6.3 *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 Since organic isocyanates react with atmospheric moisture, take special precautions in sampling. Usual sampling methods, even when conducted rapidly, can cause contamination of the sample with insoluble urea. Therefore, blanket the sample with dry air or nitrogen at all times. (**Warning—**Many diisocyanates are known or suspected sensitizers. Over-exposure to diisocyanates can lead to adverse health effects, which may include the development of occupational asthma and other respiratory, skin, and eye effects. Engineering controls, or personal protective equipment, or both, including respiratory, skin, and eye protection, are to be used when there is a potential for over-exposure to diisocyanates. The product suppliers' Material Safety Data Sheet (MSDS) provides more detailed information about potential adverse health effects and other important safety and handling information. Always follow the specific instructions provided on the MSDS.)

8. Test Conditions

8.1 Since isocyanates react with moisture, keep laboratory humidity low, preferably around 50 % relative humidity.

9. Procedure

9.1 Clean the pycnometer by filling it with chromic acid cleaning solution and by allowing it to stand for a few hours. Empty the pycnometer and rinse well with distilled water.

9.2 Fill the pycnometer with freshly boiled distilled water that has been cooled to 22 to 24°C. Insert the thermometer into the pycnometer without trapping air bubbles. Place the pycnometer in a water bath at 25 ± 0.05°C and allow it and its contents to equilibrate for at least 30 min. Wipe the overflow from the side-arm capillary and cover it with the vented cap. Remove the pycnometer from the bath, wipe dry, and weigh.

9.3 Empty the pycnometer and rinse it with alcohol, then with ether. Remove the ether and dry the pycnometer under vacuum for 15 min. Weigh the pycnometer. Determine the weight, *W,* of the water at 25.0°C in air by subtracting the weight of the empty pycnometer from the weight of the pycnometer filled with water.

NOTE 2—Other drying techniques can be used if ether is to be avoided. It must be established that for any alternative drying method, a clean and dry pycnometer results.

9.4 Carry out the rest of the procedure in a ventilated hood. The sample for testing must be completely liquid. If the sample contains solid toluene diisocyanate, warm it in its original container until it becomes liquid. Rapidly cool the liquid to 22 to 24°C and fill the pycnometer, while minimizing exposure of the sample to air.

9.5 Insert the thermometer into the pycnometer without trapping air bubbles. Cover the side arm with the vented cap and allow the pycnometer to equilibrate in the water bath for at least 30 min. Wipe the overflow from the side arm capillary and cover it with the cap vented. Remove the pycnometer from the bath, wipe dry, and weigh. To obtain the weight, *S,* of the sample at 25.0°C, subtract the weight of the empty pycnometer from the weight when filled with sample.

10. Calculation

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

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

where:

S = sample used, g (see 9.5), and
W = water in the pycnometer, g (see 9.3).

11. Precision and Bias

11.1 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 can contact the Chairman, Subcommittee D20.22 (Section D20.22.01), ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

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

11.2.1 *Repeatability*—It has been estimated that duplicate results by the same analyst are to be considered suspect if they differ by 0.0002 units.

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

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

[3] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

TEST METHOD B—SPECIFIC GRAVITY BY HYDROMETER

12. Apparatus

12.1 *Hydrometer*—An ASTM hydrometer with a specific gravity range from 1.195 to 1.255 at 15.6/15.6°C conforming to the requirements for Hydrometer 115H in Specification E100.

12.2 *Hydrometer Cylinder*—A clear glass cylinder with an inside diameter at least 25.4 mm [1 in.] larger than the hydrometer and tall enough so that there is at least 1 in. between the bottom of the cylinder and the bottom of the hydrometer after equilibration at 25°C.

12.3 *Thermometer*—See 5.3.

12.4 *Water Bath*—See 5.2.

13. Sampling

13.1 Since organic isocyanates react with atmospheric moisture, take special precautions in sampling. Usual sampling methods, even when conducted rapidly, can cause contamination of the sample with insoluble urea. Therefore, blanket the sample with dry air or nitrogen at all times. (**Warning—**Many diisocyanates are known or suspected sensitizers. Over-exposure to diisocyanates can lead to adverse health effects, which may include the development of occupational asthma and other respiratory, skin, and eye effects. Engineering controls, or personal protective equipment, or both, including respiratory, skin, and eye protection, are to be used when there is a potential for over-exposure to diisocyanates. The product suppliers' Material Safety Data Sheet (MSDS) provides more detailed information about potential adverse health effects and other important safety and handling information. Always follow the specific instructions provided on the MSDS.)

14. Test Conditions

14.1 Since isocyanates react with moisture, keep laboratory humidity low, preferably around 50 % relative humidity

15. Procedure

15.1 The sample for test must be completely liquid. If the sample is turbid, warm it in its original container until it becomes clear, then cool it to 22 to 24°C. Carry out this entire procedure in a well-ventilated hood.

15.2 To avoid forming air bubbles, slowly pour the sample into the dry hydrometer cylinder. Remove any air bubbles adhering to the surface by touching them with a piece of clean filter paper. Place the cylinder in the water bath. Stir the contents of the cylinder, being careful to avoid formation of air bubbles. When the temperature of the sample is 24.8°C, slowly and carefully lower the hydrometer into the sample liquid to a level two small scale divisions below that at which it will float and then release the hydrometer. Allow the hydrometer to come to rest and make sure it does not touch the walls of the cylinder. When the temperature of the sample reaches 25.0°C, read the gravity by sighting below the level of the liquid and slowly raising up until the surface of the sample, first seen as a distorted ellipse, seems to become a straight line cutting the hydrometer scale.

15.3 Determine the temperature of the sample just before and just after reading the hydrometer.

16. Calculation

16.1 Calculate the specific gravity at 25/25°C as follows (Note 3):

$$\text{Specific gravity, } 25/25°C = \text{observed value} + 0.0021$$

NOTE 3—The correction factor +0.0021 is applied to correct the reading to 25/25°C because the hydrometer was standardized at 15.6/15.6°C.

17. Precision and Bias

17.1 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 can contact the Chairman, Subcommittee D20.22 (Section D20.22.01), ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

17.2 Test Method D891 is similar and does include precision data. Precision statements from D891 are restated below as an estimate of the precision for this test method.

17.2.1 *Repeatability*—It has been estimated that duplicate results by the same analyst are to be considered suspect if they differ by 0.0005 units.

17.2.2 *Reproducibility*—It has been estimated that results reported by different laboratories are to be considered suspect if they differ by 0.0021 units.

17.3 There are no recognized standards by which to estimate the bias of this test method.

18. Keywords

18.1 density; hydrometer; isocyanates; polyurethane raw materials; pycnometer; specific gravity

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SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D4659 - 09) that may impact the use of this standard. (March 1, 2014)

(1) Added Test Method D4052 under 2.1.
(2) Added 4.2 indicating the existence of Test Method D4052.
(3) Updated method for use of non-mercury thermometers in 5.3.

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