Designation: D4659 – 19

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, polymeric (methylene phenylisocyanate), and liquid methylene di(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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

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

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]

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

E456 Terminology Relating to Quality and Statistics

E202 Test Methods for Analysis of Ethylene Glycols and Propylene Glycols

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

E2935 Practice for Conducting Equivalence Testing in Laboratory Applications

3. Terminology

3.1 *Definitions*—Terms used in this standard are defined in accordance with Terminology D883, unless otherwise specified. For terms relating to precision and bias and associated issues, the terms used in this standard are defined in accordance with Terminology E456.

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.

[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.

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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.

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

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.

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 are acceptable, 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—** Diisocyanates are eye, skin and respiratory irritants at concentrations above the occupational exposure limit (TLV or PEL). Diisocyanates can cause skin and respiratory sensitization (asthma) in some people. Once sensitized, it is essential to limit further exposure to diisocyanates. Use a combination of engineering controls and personal protective equipment, including respiratory, skin and eye protection, to prevent overexposure to diisocyanates. Consult the product suppliers' Safety Data Sheet (SDS) for more detailed information about potential health effects and other specific safety and handling instructions for the product.)

8. Test Conditions

8.1 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 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. (See Note 2.) 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 are acceptable if ether is to be avoided. It must be established that for any alternative drying method, a clean and dry pycnometer results.

9.4 The isocyanate sample for testing must be completely liquid, that is, free of any solid material. Fill the pycnometer, while minimizing exposure of the sample to air.

9.5 Insert the thermometer into the pycnometer without trapping air bubbles and cleanly wipe any sample off the outside of the pycnometer. Allow the pycnometer to equilibrate in the water bath for at least 30 min. Wipe any overflow from the side arm capillary and cover it with the vented cap. 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 the maximum expected difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory due solely to the method is 0.0002 units.

11.2.2 *Reproducibility*—It has been estimated that the maximum expected difference between two test results for the same

[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.