



Designation: **D4659 – 14 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 and crude methylene-bis-(4-phenylisocyanate):~~ 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. For specific warning and precautionary statements see Section 7.*

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

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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*—~~For definitions of terms used in these test methods see~~ 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.

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

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 $25 \pm 0.05^\circ\text{C}$.

5.3 *Thermometer*—An ASTM low-softening point thermometer, calibrated from -2 to $+80^\circ\text{C}$, 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 ~~can be used, 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—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 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, or both, including respiratory, skin, skin and eye protection, are to be used when there is a potential for to prevent over-exposure to diisocyanates. The Consult the product suppliers' Material Safety Data Sheet (MSDS) provides(SDS) for more detailed information about potential adverse health effects and other important specific safety and handling information. Always follow the specific instructions provided on the MSDS:instructions for the product.~~)

8. Test Conditions

8.1 ~~Since isocyanates react with moisture, keep~~ 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 $25 \pm 0.05^\circ\text{C}$ 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.

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.