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## Standard Test Methods for Polyurethane Raw Materials: Determination of Specific Gravity of Isocyanates <sup>1</sup>

This standard is issued under the fixed designation D 4659; 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 ( $\epsilon$ ) 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 the standard. ~~The values given in brackets~~ No other units of measurement are for information only, 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 ~~Notes 2 and 3.~~ Section 7.*

NOTE 1—There is no equivalent ISO standard.

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D 883 [Terminology Relating to Plastics](#)

D 891 [Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals](#)

D 1193 [Specification for Reagent Water](#)

E 1 [Specification for ASTM Liquid-in-Glass Thermometers](#)

E 100 [Specification for ASTM Hydrometers](#)

E 202 [Test Methods for Analysis of Ethylene Glycols and Propylene Glycols](#)

### 3. Terminology

3.1 ~~Definitions~~ Definitions:

3.1.1 ~~For~~ For definitions of terms used in these test methods see Terminology D 883. [94eb11b4519/astm-d4659-09](#)

3.2 ~~Description~~ 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.

#### 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 ~~should~~ is to be calibrated using the ASTM thermometer specified in 5.3.

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto: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.

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 15C in Specification E 1.

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.<sup>3</sup> Other grades may 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 D 1193.

6.3 *Chromic Acid Cleaning Solution*—Prepare a saturated solution of chromic acid ( $\text{CrO}_3$ ) in concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ , sp gr 1.84).

## 7. Sampling

~~7.1 Since organic isocyanates react with atmospheric moisture, take special precautions in sampling.~~ **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—Organic isocyanates are toxic when they are absorbed through the skin, or when the vapors are breathed. Precaution—Provide adequate ventilation and wear protective gloves and eyeglasses.. Usual sampling methods (for example, sampling an open drum with a thief, even when carried out rapidly, can cause contamination of the sample with insoluble urea. Therefore, blanket the sample with dry air or nitrogen at all times. 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  $24^\circ\text{C}$ . 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.

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  $25.0^\circ\text{C}$  in air by subtracting the weight of the empty pycnometer from the weight of the pycnometer filled with water.

NOTE 2—Other drying techniques may 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  $24^\circ\text{C}$  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  $25.0^\circ\text{C}$ , subtract the weight of the empty pycnometer from the weight when filled with sample.

## 10. Calculation

10.1 Calculate the specific gravity at  $25/25^\circ\text{C}$  as follows:

<sup>3</sup> *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.