



Designation: D 4666 – 87^{ε1}

An American National Standard

Standard Test Method for Polyurethane Raw Materials: Determination of Amine Equivalent Crude or Modified Isocyanates¹

This standard is issued under the fixed designation D 4666; 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.

^{ε1} NOTE—Sections 3.1 and 11.2 were corrected editorially in March 1992.

1. Scope

1.1 This test method determines the amine equivalent of crude or modified isocyanates derived from toluene diisocyanate, methylene-bis-(4-phenylisocyanate), and polymethylene polyphenylisocyanate.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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 Note 1.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
 D 841 Specification for Nitration Grade Toluene²
 D 1193 Specification for Reagent Water³

3. Summary of Test Method

3.1 The isocyanate groups present react with an excess of di-n-butylamine to form ureas. The excess amine is titrated potentiometrically with standard hydrochloric acid. The amine equivalent is defined as the weight of sample that will combine with 1.0-g equivalent weight of dibutylamine. The percent NCO can also be calculated as shown in 11.1.

4. Significance and Use

4.1 This test method can be used for research or for quality control to characterize isocyanates used in polyurethane products.

5. Interferences

5.1 Phosgene, the carbamyl chloride of the isocyanate, hydrogen chloride, and any other acidic or basic compounds will interfere. While some crude or modified isocyanates contain acidities of up to about 0.3 %, the NCO or amine equivalent is not normally corrected.

6. Apparatus

- 6.1 *Potentiometric Titrator*, or pH meter.
 6.2 *Calomel Electrode*.
 6.3 *Glass Electrode*.
 6.4 *Lunge Pipet*, or any weighing device suitable for weighing a liquid sample by difference to the nearest 0.001 g.
 6.5 *Magnetic Stirrer*.
 6.6 *Thermometer*, -10 to 100°C range.

7. Reagents and Materials

7.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁴ Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I of Specification D 1193.

7.3 *Dibutylamine Solution* (260 g/L)—Dilute 260 g dry dibutylamine to 1 L with dry toluene.

7.4 *Hydrochloric Acid* (1 N)—Prepare 1 N hydrochloric acid (HCl) and standardize frequently enough to detect changes of 0.001.

7.5 *Isopropyl Alcohol*, 99 % minimum purity.

7.6 *Toluene*, dry, in accordance with Specification D 841, and dried by a drying agent.

8. Sampling

8.1 Since organic isocyanates react with atmospheric moisture, take special precautions in sampling (**Warning: Precaution**—Note 1). 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.

NOTE 1: **Warning**—Organic isocyanates are toxic when they are absorbed through the skin, or when the vapors are breathed. **Precau-**

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.

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² *Annual Book of ASTM Standards*, Vol 06.03.

³ *Annual Book of ASTM Standards*, Vols 02.05, 09.01, 10.02, 11.01 and 11.03.

⁴ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."