



Standard Test Method for Polyurethane Raw Materials: Determination of Acidity In Toluene Diisocyanate¹

This standard is issued under the fixed designation D 4667; 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 This test method determines the acidity of toluene-2,4-diisocyanate and toluene-2,6-diisocyanate or mixtures of the two. (See Note 1.)

1.2 *This standard does not purport to address all of the safety problems, 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. Specific hazards statements are given in Note 2.*

NOTE 1—There is no equivalent ISO standard.

2. Referenced Documents

2.1 ASTM Standards:

D 883 Terminology Relating to Plastics²

D 1193 Specification for Reagent Water³

3. Terminology

3.1 **Definitions:** For definitions of terms used in this test method see Terminology D 883.

4. Summary of Test Method

4.1 The isocyanates react with excess *n*-propanol to form urethanes. The acidic components released are titrated potentiometrically with methanolic potassium hydroxide.

5. Significance and Use

5.1 This test method can be used for research or for quality control to characterize toluene diisocyanates (TDI). Acidity correlates with performance in some polyurethane systems.

6. Apparatus

6.1 *Potentiometric Titrator*, or pH meter.

6.2 *Calomel Electrode*.

6.3 *Glass Electrode*.

6.4 *Buret*, 10-mL, graduated in 0.05-mL divisions.

6.5 *Volumetric Pipets*, 10-mL and 100-mL.

6.6 Magnetic Stirrer and Stirring Bar.

7. Reagents and Materials

7.1 **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.⁴ Other grade 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 **Hydrochloric Acid (HCl), (0.01 N)**—Standardize frequently enough to detect changes of 0.0005 N.

7.4 *n*-propanol.⁵

7.5 **Potassium Hydroxide (KOH), Methanolic Solution (0.01 N)**—Standardize frequently enough to detect changes of 0.0005 N.

8. Sampling

8.1 Since organic isocyanates react with atmospheric moisture, take special precautions in sampling (**Warning: Precaution**—Note 2). 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 2—**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.

9. Test Conditions

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

⁴ *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.

⁵ Kodak Chemicals No. 848 or its equivalent are satisfactory for this purpose. Kodak Chemicals No. 848 are available from Kodak Laboratory and Research Products, Eastman Kodak Company, 343 State Street, Rochester, NY 14650.

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D 20.22 on Cellular Plastics.

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² *Annual Book of ASTM Standards*, Vol 08.01.

³ *Annual Book of ASTM Standards*, Vol 11.01.

10. Procedure

10.1 Pipet 100 mL of *n*-propanol into clean 250-mL beaker containing a stirring bar. With stirring, add by pipet 10 mL of TDI sample (Note 3). Cover the beaker with a watch glass and stir for exactly 10 min.

NOTE 3—If the acidity of the sample is less than 0.005 % HCl, use a 20-mL sample and 200 mL of *n*-propanol.

10.2 Titrate the solution potentiometrically with 0.01 *N* methanolic KOH solution using a glass-calomel electrode system.

10.3 Run a blank exactly as described in 10.2 but omit the TDI sample. Titrate with either 0.01 *N* methanol KOH or HCl to the apparent pH of the end point observed with the sample present (Note 4).

NOTE 4—If HCl is used to obtain the blank, the volume must be added to that of the sample titration.

11. Calculation

11.1 Calculate the acidity of HCl as follows:

$$\text{HCl, \%} = 2.99(A - B)N/C \quad (1)$$

where:

- A* = KOH solution required for titration of the sample, mL,
B = KOH solution required for titration of the blank, mL, (Note 4)

C = sample used mL,
N = normality of the KOH solution, and
 2.99 = constant combining the equivalent weight of HCl (36.5) in g/equiv., conversion of L to 1000 mL, and the density of TDI (1.22), g/mL, and conversion to 100 %.

$$2.99 = (36.5)(100)/(1000)(1.22) \quad (2)$$

12. Precision and Bias

12.1 *Precision*—Attempts to develop a precision and bias statement for this test method have not been successful. For this reason, data on precision and bias cannot be given. Because this test method does not contain a numerical precision and bias statement, it shall not be used as a referee test method in case of dispute. Anyone wishing to participate in the development of precision and bias data should contact the Chairman, Subcommittee D20.22 (Section D20.22.01), ASTM, 1916 Race Street, Philadelphia, PA 19103.

12.1.1 It is estimated that duplicate results by the same analyst should be considered suspect if they differ by more than 0.0005 % HCl.

12.2 *Bias*—The bias for this test method has not yet been determined.

13. Keywords

13.1 acidity; isocyanates; polyurethane raw materials; TDI

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