



Designation: D 5629 – 99

## Standard Test Method for Polyurethane Raw Materials: Determination of Acidity in Low-Acidity Aromatic Isocyanates and Polyurethane Prepolymers<sup>1</sup>

This standard is issued under the fixed designation D 5629; 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 This test method covers the acidity, expressed as ppm of hydrochloric acid, in aromatic isocyanate or polyurethane prepolymer samples of below 100 ppm acidity. The test method is applicable to products derived from toluene diisocyanate and methylene-bis-(4-phenylisocyanate) (see Note 1).

NOTE 1—There are no equivalent ISO standards.

1.2 The values stated in SI units are to be regarded as the 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.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 883 Terminology Relating to Plastics<sup>2</sup>

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals<sup>3</sup>

### 3. Terminology

3.1 *Definitions*—The terminology in this test method follows the standard terminology defined in Terminology D 883.

#### 3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *acidity, n*—the acid strength of a sample expressed in ppm HCl.

### 4. Summary of Test Method

4.1 The isocyanate is mixed with an excess of n-propanol, a cosolvent, and a known amount of HCl. Additional acid is released into the solvent system during urethane formation. The acid is then titrated potentiometrically with methanolic KOH. The same procedure is performed with a blank solution,

and the difference in titer is used to calculate the acidity present in the isocyanate sample.

### 5. Significance and Use

5.1 This test method can be used for research or for quality control to characterize aromatic isocyanates and low-acidity prepolymers. Acidity correlates with performance in some polyurethane systems.

### 6. Apparatus

6.1 *Disposable Beakers*, 250 mL.

6.2 *Repipet*, 50 mL.

6.3 *Pipet*, 100 mL, class A volumetric; or a 50-mL buret dosimat unit, and a dosimat; or a 100-mL repipet, class A volumetric.

6.4 *Automatic Titration Equipment*, such as the following:

6.4.1 *Titroprocessor*.<sup>4</sup>

6.4.2 *Dosimat*, with magnetic stirrer.<sup>4</sup>

6.4.3 *Reference Electrode* (Brinkman Catalogue No. 020-94-400-5 or equivalent: bridge electrolyte (double junction), sleeve-type diaphragm), having saturated LiCl/ethanol solution in both chambers.

6.4.4 *pH Glass Electrode* (Brinkman Catalogue No. 020-91-012-7 or equivalent) (see Note 2).

NOTE 2—A combination pH electrode with internal reference may also be used.

6.5 *Magnetic Stirrer*.

6.6 *Polytetrafluoroethylene-Coated Stir Bars*.

6.7 *Watch Glasses*.

6.8 *Analytical Balance*, capable of weighing to the nearest 0.1 mg.

### 7. Reagents and Materials

7.1 *KOH in Methanol*, 0.01 N: 0.66 g 87.7 % KOH/1000 mL methanol, standardized with potassium hydrogen phthalate (KHP).

<sup>1</sup> 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.

Current edition approved Nov. 10, 1999. Published February 2000. Originally published as D 5629 – 94. Last previous edition D 5629 – 94.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 08.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 15.05.

<sup>4</sup> Instruments similar to and including the Metrohn 686 Titroprocessor with a Metrohn 665 Dosimat/magnetic stirrer as supplied by Brinkman Instruments Company, Division of Sybron Corporation, Cantiague Road, Westbury, NY 11590-9974, have been found to be satisfactory for this analysis.

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