



Designation: D 6099 – 97

## Standard Test Method for Polyurethane Raw Materials: Determination of Acidity in Moderate to High Acidity Aromatic Isocyanates<sup>1</sup>

This standard is issued under the fixed designation D 6099; 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 determines the acidity, expressed as parts per million (ppm) of HCl, in aromatic isocyanate samples of greater than 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 is no similar or equivalent ISO standard.

### 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>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>4</sup>

### 3. Terminology

3.1 *Definitions*—Terms used in this test method are in accordance with 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 hydrochloric acid.

### 4. Summary of Test Method

4.1 The isocyanate is mixed with an excess of methanol and a cosolvent. Additional acid is released into the solvent system during urethane formation. The acid then is titrated potentiometrically with methanolic KOH, and the acidity present in the isocyanate sample is calculated from the titer.

### 5. Significance and Use

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

### 6. Apparatus

6.1 250-mL Beakers.

6.2 50-mL Pipet or Repipet, Class A volumetric.

6.3 100-mL Pipet or Repipet, Class A volumetric.

6.4 Automatic Titration Equipment, such as:

6.4.1 Titroprocessor,<sup>5</sup> and

6.4.2 Dosimat,<sup>5</sup> with magnetic stirrer.

6.4.3 Reference Electrode,<sup>6</sup> with saturated LiCl/ethanol solution in both chambers.

6.4.4 pH Glass Electrode,<sup>7</sup> (see Note 2).

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

6.5 Magnetic Stirrer.

6.6 Stir Bars.

6.7 Watch Glasses.

6.8 Analytical Balance, capable of weighing to the nearest 1 mg.

### 7. Reagents and Materials

7.1 0.02 N KOH in Methanol—1.32 g KOH pellets (85 % KOH)/1000 mL methanol, standardized with potassium hydrogen phthalate (KHP).

7.2 Toluene or 1,2,4-Trichlorobenzene (TCB), dried for 24 h over molecular sieves.

7.3 Anhydrous Methanol.

### 8. Sampling

8.1 Since organic isocyanates react with atmospheric moisture, take special precautions in sampling (see Note 3: **Warning** and Note 4: **Precaution**). Usual sampling methods, for example, sampling an open drum thief, even when carried out rapidly, can cause contamination of the sample with insoluble urea. Blanket the sample, therefore, with dry air or nitrogen at all times.

<sup>5</sup> Instruments similar to and including the Metrohn 686 Titroprocessor with a Metrohn 665 Dosimate/magnetic stirrer as supplied by Brinkman Instruments Co., Cantiague Road, Westbury, NY 11590-9974, or an equivalent instrument, have been found to be satisfactory for this analysis.

<sup>6</sup> The Brinkman CAT. #020-94-400-5, or its equivalent: bridge electrolyte (double junction), sleeve-type diaphragm, has been found satisfactory for this analysis. See Footnote 5 for the address to Brinkman Instruments Co.

<sup>7</sup> The Brinkman CAT. #020-91-012-7, or its equivalent, has been found satisfactory for this analysis. See Footnote 5 for the address to Brinkman Instruments Co.

<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 Materials—Plastics and Elastomers.

Current edition approved April 10, 1997. Published May 1998.

<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> Annual Book of ASTM Standards, Vol. 14.02.