



Designation: D6099 – 13

Standard Test Method for Polyurethane Raw Materials: Determination of Acidity in Moderate to High Acidity Aromatic Isocyanates¹

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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—This test method is equivalent to ISO 14898, Test Method A.

2. Referenced Documents

2.1 *ASTM Standards*:²

D883 Terminology Relating to Plastics

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)³

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO Standards*:

ISO 14898 Plastics—Aromatic isocyanates for use in the production of polyurethane—Determination of acidity⁴

3. Terminology

3.1 *Definitions*—Terms used in this test method are in accordance with Terminology D883.

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 methanol and a cosolvent. Additional acid is released into the solvent system

¹ This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.22 on Cellular Materials - Plastics and Elastomers.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

during urethane formation. The acid 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*, capable of inflection detection and stirring the sample while, titrating, such as:

6.4.1 *Commerically-available Automatic Titration Apparatus*,

6.4.2 *Reference Electrode*, with saturated LiCl/ethanol solution in both chambers.

6.4.3 *pH Glass Electrode*, (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. Usual sampling

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