~~Designation:D5629–05~~ Designation: D5629 – 11

Standard Test Method for Polyurethane Raw Materials: Determination of Acidity in Low-Acidity Aromatic Isocyanates and Polyurethane Prepolymers[1]

This standard is issued under the fixed designation D5629; 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*

~~1.1This~~1.1 This test method measures the acidity, expressed as ppm of hydrochloric acid (HCl), 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)~~methylene di(phenylisocyanate) (see Note 1). Refer to Test Method D6099 ~~for determination of acidity in moderate- to high-acidity aromatic isocyanates.~~

~~NOTE1—ISO 14898, Method B is technically equivalent to this test method.~~ for determination of acidity in moderate- to high-acidity aromatic isocyanates.

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.*

NOTE 1—This standard is equivalent to ISO 14898, Method B.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics

D6099 Test Method for Polyurethane Raw Materials: Determination of Acidity in Moderate to High Acidity Aromatic Isocyanates

E180 ~~Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals~~

~~2.2~~ *Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals*

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

2.2 *ISO Standards:*[3]

ISO 14898 Plastics—Aromatic Isocyanates for Use in the Production of Polyurethane—Determination of Acidity

3. Terminology

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

[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 Plastics.~~

~~Current edition approved March 1, 2005. Published March 2005. Originally approved in 1994. Last previous edition approved in 1999 as D5629-99. DOI: 10.1520/D5629-05.~~on Cellular Materials - Plastics and Elastomers.

Current edition approved Feb. 1, 2011. Published March 2011. Originally approved in 1994. Last previous edition approved in 2005 as D5629 - 05. DOI: 10.1520/D5629-11.

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

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

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 or buret, 50 mL.*

6.3 *Pipet*, 100 mL, class A volumetric; or a 100-mL buret with a ~~dosimat;~~dosing unit; or a 100-mL repipet, class A volumetric.

6.4 ~~*Automatic Titration Equipment*, such as the following:~~ Potentiometric Titrator:

6.4.1 ~~*Titroprocessor.*~~

~~6.4.2~~*Dosimat*~~, with magnetic stirrer.~~[4]

~~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~~*—bridge-type electrolyte (double junction), sleeve-type diaphragm, having saturated LiCl/ethanol solution in both chambers, or equivalent.*

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

NOTE~~2—A combination pH electrode with internal reference can also be used.~~ 2—A combination pH electrode with internal reference is acceptable.

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

7.2 *Toluene or THF* (dried for 24 h over 5 A molecular sieves).

7.3 *n-Propanol*, acidified with 120 µL concentrated hydrochloric acid per 4-L bottle. Allow this solution to stand for at least 24 h before use to allow equilibration. The solution is to be mixed well before dispensing.

8. Sampling

~~8.1Since~~8.1 Since organic isocyanates react with atmospheric moisture, take special precautions in sampling. Usual sampling ~~methods (for example, sampling from an open drum with a thief),~~methods, even when conducted rapidly, can cause contamination of the sample with insoluble urea. Therefore, blanket the sample with dry air or nitrogen at all times. (**Warning—**~~Organic isocyanates are toxic when they are absorbed through the skin or when the vapors are breathed. Provide adequate ventilation, and wear protective gloves and eye glasses.)~~ Many diisocyanates are known or suspected sensitizers. Over-exposure to diisocyanates can lead to adverse health effects which include the development of occupational asthma and other respiratory, skin and eye effects. Engineering controls and/or personal protective equipment, including respiratory, skin and eye protection, are to be used when there is a potential for over-exposure to diisocyanates. The product suppliers' Material Data Safety Sheet (MSDS) provides more detailed information about potential adverse health effects and other important safety and handling information. Always follow the specific instructions provided on the MSDS.)

9. Calibration

9.1 Calibrate the electrodes using pH 4 and 7 aqueous buffers.

10. Test Conditions

~~10.1Since isocyanates react with moisture, keep the laboratory humidity low, preferably at approximately 50% relative humidity.~~

10.1 Since isocyanates react with moisture, keep samples protected against moisture until immediately before testing.

11. Procedure

~~11.1Make~~11.1 Make a blank determination in conjunction with each series of samples. Prepare the blank in the same manner as a sample, only omitting the sample. All samples and blanks are to be made in duplicate.

11.2 Accurately weigh, to the nearest 0.1 mg, 20 g of sample into a 250-mL beaker.

11.3 Add 50 mL of dried toluene (dried over 5-A molecular sieves).

NOTE~~3—Substitute THF for toluene for prepolymers.~~ 3—For prepolymers, THF is an acceptable substitute for toluene.

NOTE~~4—If a sample is difficult to get into solution, it may be gently heated and stirred for a longer period of time to ensure homogeneity.~~ 4—If a sample is difficult to get into solution, it is acceptable to gently heat and stir to ensure homogeneity.