Designation: ~~D4662 – 15~~ D4662 – 20

Standard Test Methods for Polyurethane Raw Materials: Determination of Acid and Alkalinity Numbers of Polyols [1]

This standard is issued under the fixed designation D4662; 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 These test methods measure the acidic and basic constituents in polyols and other materials of high acidity or alkalinity that are soluble in mixtures of toluene and ethyl alcohol. These test methods do not apply to polyethers. (See Note 1.)

1.2 *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

~~NOTE 1—There is no known ISO equivalent to this standard.~~
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1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D883 Terminology Relating to Plastics
D1193 Specification for Reagent Water
E456 Terminology Relating to Quality and Statistics
E2935 Practice for Conducting Equivalence Testing in Laboratory Applications

3. Terminology

3.1 *Definitions—~~For definitions of terms used in these test methods see~~* Terms used in this standard are defined in accordance with Terminology D883~~.~~, unless otherwise specified. For terms relating to precision and bias and associated issues, the terms used in this standard are defined in accordance with Terminology E456.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *acid ~~number—~~number, n—*the quantity of base, expressed in milligrams of potassium hydroxide, that is required to titrate acidic constituents present in 1 g of sample.

3.2.2 *alkalinity ~~number—~~number, n—*the quantity of base, expressed as milligrams of potassium hydroxide, present in 1 g of sample.

4. Summary of Test Method

4.1 The sample is dissolved in a mixture of toluene and ethyl alcohol. The resulting single-phase solution is titrated at room temperature with alcoholic potassium hydroxide solution, to the end point indicated by the color change of added phenolphthalein. Alkalinity numbers are determined by back-titration after adding excess hydrochloric acid. The endpoint of these titrations also can be determined potentiometrically.

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[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are 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.

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

5. Significance and Use

5.1 These test methods are suitable for quality control, as specification tests, and for research. The acid and alkalinity numbers indicate the extent of a reaction with acids. The results are measures of batch-to-batch uniformity and are typically used as correction factors in calculating hydroxyl number.

6. Reagents and Materials

6.1 *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.[3] Other grades are allowed, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I of Specification D1193.

6.3 *Ethyl Alcohol,* ~~95 %.~~95 % (minimum purity).

6.4 *Hydrochloric Acid* (0.1 *N*)—Prepare a 0.1 *N* solution of hydrochloric acid (HCl). Standardization is unnecessary.

6.5 *Phenolphthalein Indicator Solution*—Dissolve 0.5 g of phenolphthalein in 100 mL of a mixture of equal volumes of water and ethyl alcohol. Add a slight excess of 0.1 *N* NaOH solution (pink color) and then just neutralize (colorless) with 0.1 *N* HCl.

6.6 *Potassium Hydroxide, Standard Alcoholic Solution* (0.1 *N*)—Dissolve 5.61 g of potassium hydroxide (KOH) in 10 mL of carbon dioxide-free water and dilute to 1 L with ethyl alcohol. Store the solution in a chemical-resistant dispensing bottle protected by a guard tube containing soda-lime or ascarite. Standardize frequently enough to detect changes of 0.0005 *N*, preferably against pure potassium acid phthalate ($KHC_8H_4O_4$, 0.8 to 0.9 g) in about 100 mL of carbon dioxide-free water, using phenolphthalein to detect the end point.

6.7 *Sodium Hydroxide, Standard Solution* (0.1 *N*)—Prepare and standardize a 0.1 *N* solution of sodium hydroxide (NaOH). Follow 6.6 for instructions on preparing and standardizing solution.

6.8 *Titration Solvent*—Mix equal volumes of toluene and ethyl alcohol.

7. Sampling

7.1 Polyesters usually contain molecules covering an appreciable range of molecular weights. These have a tendency to fractionate during solidification. Unless the material is a finely ground solid, it is necessary to melt (using as low a temperature as necessary) and mix the resin well before removing a sample for analysis. Because many polyols are hygroscopic, one must take care to provide minimum exposure to atmospheric moisture during the sampling.

TEST METHOD A—ACID NUMBER

8. Procedure

8.1 Into a 250-mL Erlenmeyer flask, introduce a weighed quantity of the sample (Note 2). Add 50 mL of the titration solvent and 0.5 mL of the indicator solution, and swirl until the sample is completely dissolved (heat only if necessary, and do not boil).

NOTE 2—For samples with an acid number of less than 7.0, use 6 to 8 g of sample. If the acid number is expected to exceed 7.0, choose an amount of sample that will contain 0.7 to 0.9 meq of acid. If the sample is not sufficiently soluble to enable use of the above amounts, decrease the sample size as necessary. Weigh samples exceeding 1.0 g to the nearest 1 mg. Weigh smaller samples to the nearest 0.1 mg.

8.2 Titrate immediately with 0.1 *N* KOH solution at a temperature below 30°C, using a 10-mL buret to add the KOH solution and using phenolphthalein as the indicator. Swirl the solution vigorously and add the KOH solution dropwise when approaching the end point. Consider the end point definite if the color change persists for 15 s.

8.3 Make a blank determination on 50 mL of the titration solvent and 0.5 mL of the indicator solution, in the same manner as the sample was titrated. Record the quantity of 0.1 *N* KOH solution required to reach the phenolphthalein end point.

9. Calculation

9.1 Calculate the acid number, in milligrams of KOH/gram of sample, as follows:

$$\text{Acid number} = [(A - B)N \times 56.1]/W$$

where:

A = KOH solution required for titration of the sample, mL,

[3] *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

B = KOH solution required for titration of the blank, mL,
N = normality of the KOH solution, and
W = sample used, g.

10. Report

10.1 For acid and alkalinity numbers below 7.0, report the value to the nearest 0.01.

10.2 For acid or alkalinity numbers of 7.0 or over, report the value to the nearest 0.1.

11. Precision and Bias[4]

11.1 *Precision*—Attempts to develop a precision and bias statement for these test methods have not been successful. For this reason, data on precision and bias cannot be given. Because these test methods do not contain numerical precision and bias statements, they shall not be used as referee test methods 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 International, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

11.1.1 A limited round robin was run with three laboratories. It is estimated that duplicate results by the same analyst should be considered suspect if they differ by more than the following amounts:

Acid or Alkalinity Number	Repeatability
less than 7.0	0.1 number
7.0 and over	1 %

11.2 *Bias*—The bias for these test methods has not yet been determined.

12. Keywords

12.1 acidity; acid number; alkalinity number; polyols

TEST METHOD B—ALKALINITY NUMBER

13. Procedure

13.1 Proceed as directed in Section 8. If the sample solution is alkaline to phenolphthalein, add 0.1 N HCl from a 10-mL buret until the solution is colorless; then add 1.0 mL excess. Back-titrate to the end point with 0.1 N NaOH solution from a 10-mL buret. Titrate a blank containing the same amount of added 0.1 N HCl.

14. Calculation

14.1 Calculate the alkalinity number, in milligrams of KOH/gram of sample, as follows:

$$\text{Alkalinity number} = [(B - A)N \times 56.1]/W$$

where the terms are defined as in Section 9.

15. Report

15.1 For acid and alkalinity numbers below 7.0, report the value to the nearest 0.01.

15.2 For acid or alkalinity numbers of 7.0 or over, report the value to the nearest 0.1.

16. Precision and Bias[4]

16.1 *Precision*—Attempts to develop a precision and bias statement for these test methods have not been successful. For this reason, data on precision and bias cannot be given. Because these test methods do not contain numerical precision and bias statements, they shall not be used as referee test methods 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 International, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

16.1.1 A limited round robin was run with three laboratories. It is estimated that duplicate results by the same analyst should be considered suspect if they differ by more than the following amounts:

[4] Supporting data are available from ASTM Headquarters. Request RR:D20-1089.