Designation: D7253 – 22

Standard Test Method for Polyurethane Raw Materials: Determination of Acidity as Acid Number for Polyether Polyols[1]

This standard is issued under the fixed designation D7253; 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 This test method measures the acidic constituents in polyether polyols and reports the results as acid number. The typical acid number range is 0-0.1 mg KOH/g sample. (See Note 1.)

1.2 The values stated in SI units are to be regarded as 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—There is no known ISO equivalent to this standard.

1.4 *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

E456 Terminology Relating to Quality and Statistics

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

E2935 Practice for Evaluating Equivalence of Two Testing Processes

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method see Terminology D883, unless otherwise specified. For terms relating to precision and bias and associated issues, the term 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*—the quantity of base, expressed in milligrams of potassium hydroxide, that is required to titrate acidic constituents in 1 g of sample.

4. Summary of Test Method

4.1 The sample is dissolved in 2-propanol. The resulting single-phase solution is titrated at room temperature with 0.02 *N* methanolic potassium hydroxide solution to an end point indicated by the color change (pink endpoint) of the added phenolphthalein.

NOTE 2—It is permissible to use automatic titrators using a potentiometric endpoint for the titration portion of this test provided the automatic titration method is tested to obtain at least equivalent results to the manual titration.

NOTE 3—Phenolphthalein is the indicator of choice based on published hydroxyl number methods that include an acid number correction. Other indicators are chosen if specific acids are of interest. Bromothymol blue (green endpoint) is used for stronger acids (pKa's < ~4) and thymolphthalein (blue endpoint) is used for weak acids (pKa's > ~7).

5. Significance and Use

5.1 This test method is suitable for quality control, as a specification test, and for research. Acid numbers indicate the extent of any neutralization reaction of the polyol with acids. The results of this method measure batch-to-batch uniformity and are used as correction factors in calculating true hydroxyl numbers.

6. Interferences

6.1 Samples or constituents that are highly-colored will interfere with or prevent the use of this test method. In this case, a potentiometric titration using an autotitrator is recommended, see Note 2.

7. Apparatus

7.1 *Buret, 10 mL,* manual or automatic.

7.2 *Graduated cylinder, 10 mL, maximum.*

7.3 *Balance, analytical with sensitivity of at least ±0.0001 g.*

[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.

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

7.4 *Erlenmeyer flask, at least 250 mL capacity.*

7.5 *Stirring bars or stirring rods.*

8. Reagents and Materials

8.1 *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 acceptable provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Phenolphthalein,* Indicator Grade.

8.3 *Phenolphthalein Indicator Solution*—Dissolve 1 g of phenolphthalein in 100 mL of methanol.

8.4 *Potassium Hydroxide Standard Solution (0.02 N)*—Prepare and standardize a 0.02 *N* methanolic potassium hydroxide solution. (1.32 g KOH pellets (85 % KOH) per 1000 mL methanol, standardized with potassium hydrogen phthalate.) The KOH solution is to remain capped to avoid the formation of carbonates due to the reaction with atmospheric CO_2.

9. Procedure

9.1 Add 100 ± 20 mL of 2-propanol and 1 mL of phenolphthalein indicator solution to an Erlenmeyer flask.

9.2 Titrate the stirred solution with 0.02 *N* KOH solution to the first faint pink endpoint that persists for 30 seconds.

9.3 Weigh 50-60 g of sample into the same Erlenmeyer flask and record the weight of the sample to the nearest 0.1 g.

9.4 Stir or swirl the solution in the flask until the sample is completely dissolved.

9.5 Titrate the clear sample solution with standardized 0.02 *N* KOH solution to the first faint pink endpoint that persists for 30 seconds.

10. Calculation or Interpretation of Results

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

$$Acid\ Number = [A \times N \times 56.1]/W \quad (1)$$

where:

A = volume of KOH solution for the titration of the sample in mL,
N = the normality of the KOH solution, and
W = the weight of the sample used in g.

11. Report

11.1 Report results to the nearest 0.001 mg KOH/g.

[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 Phamacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

TABLE 1 Round Robin Data for Acid Number (mg KOH/g) in Accordance with Practice E691

Material	Average	S_r[A]	S_R[B]	r[C]	R[D]	n[E]
1	0.026	0.001	0.005	0.003	0.013	7
2	0.015	0.001	0.005	0.004	0.014	7
3	0.016	0.002	0.005	0.005	0.013	7
4	0.105	0.001	0.008	0.004	0.022	6

[A] S_r = within-laboratory standard deviation of the replicates.
[B] S_R = between-laboratory standard deviation of the averages.
[C] r = within-laboratory repeatability limit = 2.8*S_r.
[D] R = between-laboratory reproducibility limit = 2.8*S_R.
[E] n = number of laboratories contributing valid data for this material.

12. Precision and Bias[4]

12.1 Table 1 is based on a round robin involving seven laboratories and conducted in 2005 in accordance with Practice E691. All labs used a colorimetric endpoint titration for the generation of the data used in this study. All the samples were prepared at one source, but the individual specimens were prepared at the laboratories that tested them. Each test result was the average of two individual determinations. Each laboratory made duplicate determinations on each material on each of two days. (**Warning—**The following explanations of *r* and *R* (12.2 – 12.2.3) are intended only to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 is not to be rigorously applied to the acceptance or rejection of material, as those data are specific to the round robin and are not representative of other lots, conditions, materials, or laboratories. Users of this test method are to apply the principles outlined in Practice E691 to generate data specific to their laboratory and materials or between specific laboratories. The principles of 12.2 – 12.2.3 are then valid for such data.)

12.2 *Precision*

12.2.1 *Repeatability, (r)*—It has been determined that the maximum expected difference between two test results for the same material, obtained by the same operator using the equipment on the same day in the same laboratory due solely to the method is *r*.

12.2.2 *Reproducibility, (R)*—It has been estimated that the maximum expected difference between two test results for the same material, obtained by different operators using different equipment in different laboratories due solely to the method is *R*.

12.2.3 Any judgment in accordance with 12.2.1 and 12.2.2 has an approximate 95 % (0.95) probability of being correct.

12.3 There are no recognized standards by which to estimate the bias of this test method.

12.4 For information on equivalence, refer to Practice E2935.

13. Keywords

13.1 acidity; acid number; hydroxyl number; polyether polyols

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1244.