Designation: D8032 – 16

Standard Test Method for Acid Number of Terephthalic Acid by Color-Indicator Titration[1]

This standard is issued under the fixed designation D8032; 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 covers the determination of acid number of terephthalic acid (TA) by color-indicator titration. Acid number of TA product is usually within 674 to 676 mg KOH/g.

1.2 In determining the conformance of the test results using this method, results shall be rounded off in accordance with the rounding-off method of Practice E29.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D974 Test Method for Acid and Base Number by Color-Indicator Titration

D1193 Specification for Reagent Water

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E300 Practice for Sampling Industrial Chemicals

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

2.2 *Other Document:*[3]

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200

3. Terminology

3.1 *Definitions:*

3.1.1 *acid number, n*—the quantity of base, expressed in milligrams of potassium hydroxide per gram of sample that is required to titrate a sample in a specified solvent to a specified end point.

4. Summary of Test Method

4.1 A TA sample is dissolved in dimethyl sulfoxide and titrated with standard sodium hydroxide solution to the end point indicated by the color change of the added phenolphtalein solution (colorless in acid and pink in base). The acid number is calculated as milligrams of KOH per gram of TA sample. Its theoretical value of TA sample is 675.5 mg KOH/g.

5. Significance and Use

5.1 An estimate of TA purity can be determined by titrating with KOH. As an index of TA purity, the acid number can be used as a guide in the quality control of TA production.

6. Apparatus

6.1 *Analytical Balance,* capable of weighing ±0.0001 g.

6.2 *Burets,* 50-mL with 0.1-mL graduations.

7. Reagents

7.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents shall conform to the reagent grade specification of the Analytical Reagents of the American

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.02 on Oxygenated Aromatics.

Current edition approved March 1, 2016. Published June 2016. DOI: 10.1520/D8032-16.

[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 U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.access.gpo.gov.

Chemical Society,[4] where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the performance or accuracy of the determination. Reagent chemicals shall be used for all tests.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type III of Specification D1193. Boil the water gently for 5 to 10 min to remove any CO_2 and cool the water to room temperature.

7.3 *Dimethyl Sulfoxide*—(**Warning**—Flammable and harmful if inhaled, swallowed or absorbed through the skin.).

7.4 *Sodium Hydroxide Solution* (0.5 M)—Weigh 20 g of sodium hydroxide (NaOH) (**Warning**—Highly corrosive to all body tissue.) in a beaker. Add 100 mL water (boiling gently for 5 to 10 min and cooling to room temperature, free of CO_2) to dissolve and cool the solution. Then quantitatively transfer the solution into a 1000 mL volumetric flask and dilute to volume with the above water. The NaOH solution should be stored in a plastic bottle and stopper must be stressed.

NOTE 1—After standardization, the NaOH solution should not be stored in a glass container because it will be slowly neutralized from exposure to a glass container. It will also "cement" a glass stopper into a glass container.

7.5 *Ethanol.*

7.6 *Phenolphthalein Indicator Solution* (1 g/L)—Dissolve 0.1 g solid phenolphthalein in 100 mL ethanol.

7.7 *Potassium Hydrogen Phthalate.*

8. Hazards

8.1 Consult current federal regulations, supplier's Safety Data Sheets, and local regulations for all materials used in this test method.

9. Sampling, Test Specimens, and Test Units

9.1 Use only representative samples obtained as described in Practice E300, unless otherwise specified.

10. Standardization of Titrant

10.1 Place 10 to 20 g of primary standard potassium hydrogen phthalate in a weighing bottle and dry at 120°C for 2 h. Close the weighing bottle and cool in a desiccator.

10.2 Weigh, to the nearest 0.0001 g, 4.5 to 5.0 g of the dried potassium hydrogen phthalate and transfer to a 250-mL Erlenmeyer flask. Add 100 mL of CO_2-free water and stir gently to dissolve the sample.

10.3 Add 3 drops of phenolphthalein indicator solution and titrate with 0.5 M NaOH solution (7.4) until one drop of NaOH changes the color of the solution from colorless to pink.

10.4 Perform a blank titration by repeating the above steps without adding potassium hydrogen phthalate.

10.5 Calculate the molarity of the NaOH solution as follows:

$$C = \frac{m \times 1000}{M \times (V - V_0)} \quad (1)$$

where:

C = molarity of NaOH solution, mol/L,
V = NaOH solution required for titration of the potassium hydrogen phthalate (10.3), mL,
V_0 = NaOH solution required for titration of the potassium hydrogen phthalate (10.4), mL,
M = 204.23 g/mol, molar mass of the potassium hydrogen phthalate (7.7), and
m = mass of potassium hydrogen phthalate titrated, g.

11. Procedure

11.1 Weigh, to the nearest 0.0001 g, 0.8 to 1.5 g of TA sample, into a 250-mL flask, and add 20 mL of dimethyl sulfoxide with swirling to dissolve TA completely.

11.2 Add 20 mL of CO_2-free water, and 0.1 mL of the phenolphthalein indicator solution into the flask.

11.3 Titrate using the standard NaOH solution and swirl flask contents gently during titration to a 15-s pink end point. Record the amount of titrant required.

11.4 Perform a blank titration by repeating the above steps without adding the TA sample.

12. Calculation

12.1 Calculate the acid number as follows:

$$\text{Acid number, mg of KOH / g} = [(A - B) \times C \times M] / W \quad (2)$$

where:

A = NaOH solution required for titration of the TA sample (11.3), mL,
B = NaOH solution required for titration of the blank (11.4), mL,
C = molarity of the NaOH solution (10.5), mol/L,
M = 56.11 g/mol, molar mass of the KOH, and
W = mass of TA sample titrated, g.

13. Report

13.1 Report the value of acid number in mg KOH/g, to the nearest 0.1 unit.

13.2 Report the following information in the report:

13.2.1 The complete identification of the sample tested.

13.2.2 Any deviation from the procedure specified (for example operating conditions).

13.2.3 Results of the test.

13.2.4 Any abnormal situations observed during the test.

14. Precision and Bias

14.1 The precision of this method is based on an intralaboratory study of Test Method D8032 conducted in 2015. One laboratory tested one TA sample for acid number by color-indicator titration. Every test result represents an individual determination. The laboratory reported 20 replicate results for each analysis/material combination in order to estimate the

[4] *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.

repeatability limits of the standard. Practice E691 was followed for the design and analysis of the repeatability data; the details are given in Research Report RR:D16-1058.[5]

14.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

14.1.1.1 Repeatability limits are listed in Table 1.

TABLE 1 Repeatability Limits (TA)

Color-Indicator Titration	Average (mg KOH/g) $\bar{X}$	Repeatability Standard Deviation Sr	Repeatability Limit r
acid number	675.2	0.49	1.36

14.1.2 Reproducibility has not been determined and will be determined within five years.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D16-1058. Contact ASTM Customer Service at service@astm.org.

14.2 *Bias*—At the time of the study, the test specimens chosen for analysis were not accepted reference materials suitable for determining the bias for this test method, therefore no statement on bias is being made.

15. Quality Guidelines

15.1 Laboratories shall have a quality control system in place.

15.1.1 Confirm the performance of the test instrument or test method by analyzing a quality control sample following the guidelines of standard statistical quality control practices.

15.1.2 A quality control sample is a stable material isolated from the production process and representative of the sample being analyzed.

15.1.3 When QA/QC protocols are already established in the testing facility, these protocols are acceptable when they confirm the validity of test results.

15.1.4 When there are no QA/QC protocols established in the testing facility, use the guidelines described in Guide D6809 or similar statistical quality control practices.

16. Keywords

16.1 acid number; color-indicator titration; terephthalic acid (TA)