Designation: D 3644 – 98

Standard Test Method for Acid Number of Styrene-Maleic Anhydride Resins[1]

This standard is issued under the fixed designation D 3644; 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 measurement of the free acidity present in styrene-maleic anhydride resins.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 329 Specification for Acetone[2]

D 1193 Specification for Reagent Water[3]

3. Terminology

3.1 *Definition:*

3.1.1 *acid number*—the number of milligrams of potassium hydroxide (KOH) required to neutralize the alkali-reactive groups in 1 g of material under the conditions of test.

4. Significance and Use

4.1 This test method is used to determine the property of styrene-maleic anhydride resins functionality. Acid functionality determines the utility of resin as well as being a significant quality control test.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

5.3 *Acetone*, conforming to the requirements described in Specification D 329.

5.4 *Phenolphthalein Indicator Solution* (20 g/L)—Dissolve 10 g of phenolphthalein in 100 mL of acetone.

5.5 *Sodium Hydroxide, Standard Solution* (0.1 N)—Dissolve 7 g of sodium hydroxide (NaOH) in 7 mL of water, and filter the resulting solution through an asbestos mat in a Gooch crucible with the aid of suction. Do not wash the residue. Dilute two thirds of the clear filtrate to 1 L with freshly boiled water. Standardize against National Institute of Standards and Technology standard sample of acid potassium phthalate No. 84, using phenolphthalein as the indicator. Do not adjust the concentration of the solution, but calculate the normality.

5.6 *Sulfuric Acid, Standard Solution* (0.1 N)—Measure out 3 mL of concentrated sulfuric acid (H_2SO_4) (d = 1.84) and pour it slowly, and with constant stirring, into about 100 mL of water. Cool to room temperature, mix thoroughly, and dilute to 1 L. Standardize against 0.1 *N* NaOH solution, as prepared in 4.5, using phenolphthalein as the indicator. Do not adjust the concentration of the solution, but calculate the normality.

6. Procedure

6.1 Weigh 0.1 g of the sample to the nearest 0.0001 g into a 250-mL Erlenmeyer flask.

6.2 Add 150 ± 2 mL of acetone to the flask, stopper it, and swirl it until the sample is dissolved. All 0.2 mL of phenolphthalein indicator solution and titrate with NaOH solution to the end point, a faint pink color which persists for 30 s. Then add 2.0 mL of additional NaOH solution, stopper the flask, and allow it to stand for exactly 10 min.

NOTE 1—If a precipitate forms during the titration, discard the sample and repeat the test using a smaller sample or a greater volume of acetone, or both. A slight haze can be tolerated and does not interfere with the test.

6.3 Back titrate with 0.1 *N* H_2SO_4 to the end point, at which the sample turns colorless and remains so for 30 s.

6.4 Using the same procedure, titrate a blank composed of 150 mL of acetone and 5 mL of water.

7. Calculation

7.1 Calculate the acid number as follows:

$$\text{Blank} = (V_B N_B) - (V_A N_A)$$

[1] This test method is under the jurisdiction of ASTM Committee D-21 on Polishes and is the direct responsibility of Subcommittee D21.02 on Raw Materials.

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[2] *Annual Book of ASTM Standards*, Vol 06.04.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[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.

$$\text{Acid number} = \frac{(VBNB - (VANA + \text{blank})) \times 56.1}{C}$$

V_A = millilitres of H_2SO_4,
N_A = normality of H_2SO_4,
V_B = millilitres of NaOH solution,
N_B = normality of NaOH solution, and
C = weight of sample.

8. Report

8.1 Report the acid number of the resin tested, to the nearest whole number.

9. Precision and Bias

9.1 *Precision*—Duplicate results by the same operator shall not be considered suspect unless they differ by more than a standard deviation of 0.8.

9.2 *Bias*—This test has no bias because the values produced are defined only in terms of this test method.

10. Keywords

10.1 acid number; free acid; polish; resins; SMA resins; styrene-maletic anhydrid resin; titration