Designation: D3643 – 15

Standard Test Method for Acid Number of Certain Alkali-Soluble Resins[1]

This standard is issued under the fixed designation D3643; 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 certain alkali-soluble resins.

1.2 This test method is not suitable for styrene-maleic anhydride resins.

1.3 The resin manufacturer should specify whether or not this test method may be used for his product(s).

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

1.5 *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]

D362 Specification for Industrial Grade Toluene (Withdrawn 1989)[3]

D1152 Specification for Methanol (Methyl Alcohol)

D1193 Specification for Reagent Water

D3644 Test Method for Acid Number of Styrene-Maleic Anhydride Resins

3. Terminology

3.1 *Definitions:*

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 not appropriate for alkali-soluble resins whose acid functionality is due to incorporated anhydrides. Variations in manufacture, storage, and possible contamination of anhydride functional resins may cause partial hydrolysis, or esterification, which will invalidate data from this test method. Anhydride functional resins should be characterized by Test Method D3644.

4.2 This test method is used to measure a property of acid functionalized resins. Acid number determines the utility of resins as well as being a significant quality control measure.

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 D1193.

5.3 *Neutral Solvent Mixture*—Mix equal parts of denatured alcohol and industrial toluene conforming respectively to the requirements described for SDA. 3A (200 proof) and Specification D362. Neutralize the mixture using 0.1 *N* KOH solution and phenolphthalein indicator solution, until the faint pink color persists for 1 min.

5.4 *Phenolphthalein Indicator Solution* (10 g/L)—Dissolve 1.0 g of phenolphthalein in 100 mL of denatured alcohol (SDA 3A, 200 proof).

5.5 *Potassium Hydroxide, Methyl Alcohol Solution* (1 mL = 5.6 mg KOH)—Dissolve 6.6 g of potassium hydroxide

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

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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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[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.

(KOH) in 1 L of methyl alcohol conforming to the requirements described in Specification D1152. 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 milligrams of KOH per L of solution.

6. Procedure

6.1 Weigh or transfer into a 250-mL Erlenmeyer flask 0.5 g of crushed resin to the nearest 0.001 g.

6.2 Add 100 mL of neutral solvent. Mix until all material is dissolved. Add 3 to 5 drops of phenolphthalein indicator solution and titrate to the end point, a faint pink color which persists for 1 min.

6.3 Run a blank titration in the procedure of 6.1 and 6.2 without including test material.

6.4 For each test material run three sample titrations and one Blank titration.

7. Calculation

7.1 Calculate the acid number as follows:

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

where:

A = millilitres of alkali solution required for titration of the sample,
B = millilitres of alkali solution required for titration of the Blank sample,
N = normality of the alkali solution, and
C = grams of resin used.

8. Report

8.1 Report the acid number of 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 ±5 units.

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; alkali-soluble resins; free acid; polish; resins; titration