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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Acid and Base Milliequivalent Content of Electrocoat Bath¹

This standard is issued under the fixed designation D 4370; 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 cover the determination of acid and base milliequivalent contents of anodic and cathodic electrocoat baths and their ultrafiltrates.

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 1193 Specification For Reagent Water²

3. Summary of Test Methods

3.1 Specimens are titrated with standard acid and alkali solutions respectively. Alternative procedures are given for determining acid and base concentrations potentiometrically or using a pH meter.

4. Significance and Use

4.1 The acid and base concentrations are a measurement of the titratable acidic and alkaline components in the electrocoat baths. These measurements are used for research, production or electrocoat bath process control.

5. Apparatus

- 5.1 *Automatic Potentiometric Titrator with Stirrer and Recorder*, any model.
5.2 *Analytical Balance*, with sensitivity of 0.1 mg.
5.3 *pH Meter*, any model.
5.4 *Glass and Saturated Calomel Electrodes*.
5.5 *Syringes*, 5-mL disposable.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paint and Paint Materials.

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² *Annual Book of ASTM Standards*, Vol 11.01.

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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

6. Reagents

6.1 *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.³

Other grades may be used, provided it is 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*—References to water shall be understood to mean water conforming to Type II of Specification D 1193.

6.3 *Potassium Hydroxide Solution in Methanol*, 0.1 N—Prepare by dissolving 5.6 g of potassium hydroxide (KOH) pellets in 1 L of methanol. Standardize against NIST standard reference material of acid potassium phthalate No. 84 using an automatic potentiometric titrator⁴ to a given end point or, alternatively, to a phenolphthalein end point.

6.4 *Hydrochloric Acid Solution*, 0.1 N—Prepare by mixing about 8.50 mL of concentrated hydrochloric acid (HCl) (1.19 sp gr) into a mixture of 600 mL water and 400 mL methanol. Standardize against 0.1 N potassium hydroxide solution (see 6.3).

6.5 *1,3-Propanediol* (Propylene Glycol) (PG).

6.6 *Tetrahydrofuran* (THF).

6.7 *Reference pH Standard Solutions*—Commercial standards of pH 4.0, 7.0, and 10.0.

7. Sampling and Sample Preparation

7.1 The sample should be obtained while the electrocoat bath is under proper circulation such that a uniform material is obtained. In case of an ultrafiltrate, the material should be thoroughly mixed or stirred prior to sampling to assure uniformity.

7.2 After sampling and prior to removing a test specimen, it is mandatory that the samples be shaken or stirred until they are homogeneous and free of any settled material. This is particularly important if there is a delay between sampling the bath and performing the test. The absence of settled material can be ascertained visually (in a transparent container) or by inserting a spatula, scraping the bottom of the container, and making sure that there is no settled matter. The shaking or stirring of

³ Svehla, G., *Automatic Potentiometric Titration*, Pergamon Press, 1978, p. 187.