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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Carboxyl Content of Cellulose¹

This standard is issued under the fixed designation D 1926; 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 the carboxyl content, or ion-exchange capacity, of cellulose from any source. Two test methods are described, the sodium chloride-sodium bicarbonate method (1)² and the methylene blue method (2). The test methods must be used within their limitations, and it must be recognized that there is no way of determining the accuracy of any method for the determination of carboxyl. The precision of the sodium chloride-sodium bicarbonate method is low in the lower range of carboxyl values. The methylene blue method can be used over the whole range of carboxyl values; it is especially useful in the low range. It is not applicable to the determination of carboxyl in soluble carbohydrate material. Although these test methods may be used to determine the ion-exchange capacity of unbleached pulps, the residual lignin will cause an undetermined error, especially the sulfonic acid groups in unbleached sulfite pulps (3).

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 Water³

3. Significance and Use

3.1 These test methods measure the amount of carboxyl groups present in wood or cotton linter pulp. Carboxyl groups are indicative of the surface charge of the pulp which is a very important quantity for use in the papermaking industry.

4. Purity of Reagents

4.1 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.

4.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

SODIUM CHLORIDE-SODIUM BICARBONATE METHOD

5. Summary of Test Method

5.1 In the sodium chloride-sodium bicarbonate method the specimen is deashed with hydrochloric acid, washed, soaked in sodium chloride-sodium bicarbonate solution, filtered, and an aliquot of the filtrate titrated with 0.01 *N* hydrochloric acid to a methyl red end point. The difference between the concentration of the filtrate and of the sodium chloride-sodium bicarbonate solution is a measure of the ion-exchange capacity of the cellulose.

6. Reagents

6.1 *Hydrochloric Acid, Standard (0.01 N)*—Prepare and standardize a 0.01 *N* solution of hydrochloric acid (HCl).

6.2 *Hydrochloric Acid (1 + 99)*—Dilute 1 volume of concentrated HCl (sp gr 1.19) with 99 volumes of water.

6.3 *Methyl Red Indicator Solution.*

6.4 *Sodium Chloride-Sodium Bicarbonate Solution*—Dissolve 5.85 g of sodium chloride (NaCl) and 0.84 g of sodium bicarbonate (NaHCO₃) in water and dilute to 1 L.

6.5 *Sodium Hydroxide Solution (0.4 g/L)*—Dissolve 0.4 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

7. Procedure

7.1 Condition the specimen in the atmosphere near the balance for at least 20 min before weighing duplicate portions of 2.5 ± 0.01 g. At the same time, weigh specimens for the determination of moisture. Disintegrate the specimen in water,

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 D01.36 on Cellulose and Cellulose Derivatives.

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² The boldface numbers in parentheses refer to the list of references at the end of these test methods.

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