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## Standard Test Method for Chromatographic Analysis of Chemically Refined Cellulose<sup>1</sup>

This standard is issued under the fixed designation D 1915; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

<sup>ε1</sup> NOTE—Editorial changes were made throughout, including the title, in October 1989.

### 1. Scope

1.1 This test method covers the determination of the composition of chemically refined cellulose by chromatographic analysis. This test method is suitable for rapid and routine testing of large numbers of samples with high accuracy and precision.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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 Document

2.1 *ASTM Standard:*  
D 1193 Specification for Reagent Water<sup>2</sup>

### 3. Summary of Test Method

3.1 Quantitative paper chromatographic analysis of polysaccharides by the chemical method involves the following operations: (1) total hydrolysis, (2) neutralization, (3) concentration, (4) chromatographic separation, (5) elution, (6) analysis of separated sugars, and (7) calibration and calculation.

### 4. Significance and Use

4.1 While this test method involves a chemical determination of separated sugars, it is recognized that direct photometry of the chromatogram can also be capable of good precision. All of the steps involved, up to the actual determination, are the same for both techniques. Direct chemical measurement is of more general applicability in a chromatographic laboratory, however, since it involves no special instrumentation and it is adaptable to the determination of a variety of materials.

4.2 The carbohydrate composition of a cellulosic material may be expressed either on the basis of the total initial sample or on the basis of the carbohydrate portion of the sample. The former establishes composition on an absolute basis. It requires precise information concerning all components present in the starting material, or lacking this, it

requires quantitative handling of the carbohydrate portion throughout the entire sequence of chromatographic operations. In the case of purified celluloses, where the carbohydrate fraction comprises all but a minor part of a given sample, it is convenient to express composition on the basis of the ratios of the carbohydrate constituents; that is, the quantity of each isolated sugar is expressed as a percent of the combined sugar content of the sample. Quantitative handling is thus not required until application of the hydrolyzate to the paper. Proper correction for conversion of polysaccharide to monosaccharide and for decomposition are of course required in any method of analysis. These may conveniently be accounted for by calibration.

### 5. Purity of Reagents

5.1 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.<sup>3</sup> 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 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

### 6. Sampling

6.1 Take samples from not less than 10 % of each lot in the shipment. Then composite these samples to represent each lot or batch.

### TOTAL HYDROLYSIS

### 7. Summary of Procedure

7.1 The total hydrolysis of a cellulose requires a primary hydrolysis with strong mineral acid followed by a secondary hydrolysis in dilute acid. The primary hydrolysis results in the formation of a mixture of oligosaccharides. It is the function of the secondary hydrolysis to complete the conversion to monomeric sugars (1).<sup>4</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-23 on Cellulose and Cellulose Derivatives and is the direct responsibility of Subcommittee D23.20 on Cellulose.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 06.03.

<sup>3</sup> "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY and the "United States Pharmacopeia."

<sup>4</sup> The boldface numbers in parentheses refer to the list of references at the end of this test method.