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Standard Test Method for Carbohydrate Distribution of Cellulosic Materials¹

This standard is issued under the fixed designation D 5896; 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 determination of the carbohydrate composition of cellulosic materials such as ground wood meal, chemically refined pulp, mechanical pulps, brownstocks, and plant exudates (gums) by ion chromatography. This test method is suitable for rapid, routine testing of large numbers of samples with high accuracy and precision. For a review of this technique, see Lee (1).²
- 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. For hazard statement, see Section 8.

2. Referenced Documents

- 2.1 ASTM Standards:
- D 1193 Specification for Reagent Water³
- D 1695 Terminology of Cellulose and Cellulose Derivatives⁴

3. Terminology

- 3.1 For standard terminology of cellulose and cellulose derivatives, see Terminology D 1695.
 - 3.2 Abbreviations:
 - 3.2.1 IC—ion chromatography,
 - 3.2.2 SPE—solid phase extraction,
 - 3.2.3 PAD—pulsed amperometric detector,
 - 3.2.4 PED—pulsed electrochemical detector,
 - 3.2.5 mM—millimolar.

4. Summary of Test Method

- 4.1 IC analysis of cellulosics requires the following operations:
 - (1) sample preparation,
 - (2) total hydrolysis,
 - (3) dilution,
 - (4) SPE,
- ¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.36on Cellulose and Cellulose Derivatives.
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- ² The boldface numbers in parentheses refer to the list of references at the end of this test method.
 - ³ Annual Book of ASTM Standards, Vol 11.01.
 - ⁴ Annual Book of ASTM Standards, Vol 06.03.

- (5) ion chromatographic analysis, and
- (6) calibration/calculation.

5. Significance and Use

- 5.1 This test method requires total hydrolysis of carbohydrate material to monosaccharides, and is thus applicable to any cellulosic or related material that undergoes substantial hydrolysis, including cellulose derivatives such as cellulose acetate.
- 5.2 The carbohydrate composition of a cellulosic material can be expressed on the basis of the total initial sample, or on the basis of the carbohydrate portion of the sample. The former requires quantitative handling and may require special knowledge of the other components present in order to establish the absolute carbohydrate level or determine individual wood hemicelluloses such as galactoglucomannan, etc. Since the solid portion of purified pulps is almost all carbohydrate (98 + %), the latter basis is often used to express the carbohydrate distribution as a percent.
- 5.3 If heated under alkaline conditions, isomeric sugars may begin to appear in the chromatogram. The major impurity present in purified pulps is saccharinic acids. These acidic components, and other anions such as sulfate, carbonate, and acetate are removed by a strong base anion exchange SPE, and would need to be determined separately to get a more exact carbohydrate distribution.

6. Apparatus

- 6.1 Blender.
- 6.2 Screw Cap Culture Tubes, 25 by 150 mm, outside diameter.
 - 6.3 Refrigerator.
 - 6.4 Pressure Cooker.
 - 6.5 SPE Cartridges.
 - 6.6 Water Bath.
 - 6.7 Ion Chromatograph.
 - 6.8 Moisture Balance.
 - 6.9 Hot Plate.
 - 6.10 Pipets.

TOTAL HYDROLYSIS

7. Reagents and Materials

7.1 Sulfuric Acid (72 \pm 0.1 weight %): To 1 volume of water, add slowly while stirring vigorously 2 volumes of concentrated sulfuric acid (sp gr 1.84). Standardize against an