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Standard Test Method for Pentosans in Cellulose

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1. Scope

1.1 This test method covers the determination of pentosans in cellulose by the orcinol colorimetric method, in the range from less than 1 to about 16 %.

NOTE 1—This test method is an adaptation of the method designated CCA 24:56 by the Analysis Committee of the Cellulose Industry of the Swedish Association of Pulp and Paper Engineers.

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 Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1348 Test Methods for Moisture in Cellulose³

3. Summary of Test Method

3.1 The cellulose is treated with boiling hydrochloric acid (approximately 12 % or 3 + 7) to hydrolyze the pentosans to pentoses, which are then converted to furfural. The furfural is distilled and collected. Hydroxymethyl furfural is also produced during the distillation, but does not interfere in the following colorimetric determination.

3.2 An aliquot of the distillate is treated with orcinol reagent, and after standing for 2 h the absorbance is measured at 630 nm. By reference to a calibration curve prepared from known furfural solutions, the percent pentosans is calculated.

4. Significance and Use

4.1 This test method is used for measuring the level of hemicelluloses in wood pulps. The amount of pentosans, primarily arabinose and xylose, are measures of the purity of wood pulps used in chemical cellulose applications.

5. Apparatus

5.1 *Flask*, two-neck round-bottom, 500-mL, standard-taper 24/40 joint.

5.2 *Dropping Funnel*, 500-mL, with standard-taper 24/40 neck.

5.3 *Distillation Connecting Tube*, with standard-taper 24/40 inner joints to fit the round-bottom flask and the Graham condenser, respectively, with the proper angles to allow for the vertical position of the condenser. If a thermometer is to be used, the adapter should have a standard-taper 10/30 thermometer well.

5.4 *Condenser*, Graham, water-cooled, with standard-taper 24/40 connections.

5.5 *Adapter*, with standard-taper 24/40 joints with gas inlet.

5.6 *Receiving Flask*, 500-mL, with standard-taper 24/40 joint, to collect the distillate.

5.7 *Ice Bath*, for receiving flask to help prevent loss of furfural.

5.8 *Heating Mantle*, for reaction flask, controlled by a variable transformer. A gas burner may be substituted for the heating mantle.

5.9 *Constant-Temperature Bath*, capable of maintaining a temperature within $\pm 0.5^\circ\text{C}$ of the temperature selected for development of the color with orcinol, which temperature should be between 15 and 30°C .

5.10 *Filter Photometer or Spectrophotometer*, suitable for measuring absorbance at 630 nm.

6. Purity of Reagents

6.1 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.

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

6.3 *Ethanol*, 95 %, aldehyde-free.

6.4 *Furfural*—Distill furfural immediately before use. Use only freshly-distilled pale yellow material with a boiling point of 162°C . It is preferable to distill in vacuum.

6.5 *Hydrochloric Acid* (3 + 7)—Mix 3 volumes of concentrated hydrochloric acid (HCl, sp gr 1.19) with 7 volumes of water. The concentration will be approximately 12 %.

6.6 *Orcinol Solution*—Dissolve 0.40 g of orcinol and 0.5 g of ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) in 1 L of 11.0 N HCl. Discard solutions more than 2 weeks old.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.36 on Cellulosics.

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² *Annual Book of ASTM Standards*, Vol 06.03.

³ *Annual Book of ASTM Standards*, Vol 06.02.

⁴ "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. 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."