



## Standard Test Method for Silica in Cellulose<sup>1</sup>

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### 1. Scope

1.1 This test method covers the determination of silica in cellulose and is applicable to all pulps. It is designed to measure up to 200  $\mu\text{g}$  of silica ( $\text{SiO}_2$ ) in 50 mL of solution.

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 1348 Test Methods for Moisture in Cellulose<sup>2</sup>

### 3. Summary of Test Method

3.1 The cellulose is ashed and hydrochloric acid (HCl)-soluble compounds are extracted. The ash is then fused with sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) to form soluble silicates. The salt is dissolved, acidified, and converted to a yellow-colored complex of silicomolybdic acid. The complex is reduced to silicomolybdenum blue by treatment with aminonaphthol sulfonic acid plus sodium sulfite ( $\text{Na}_2\text{SO}_3$ ). The concentration of silica is determined spectrophotometrically. When compared with gravimetric procedures for silica, the spectrophotometric method has better specificity, sensitivity, and precision.

### 4. Significance and Use

4.1 Low-silica specifications are required by viscose rayon processors. The general claims are that (1) silica may affect the filterability of viscose by forming insoluble silicates, (2) silica and the formation of insoluble silicates can contribute to spinneret crater formation during spinning (deposits which build up around spinneret holes to deform yarn cross section and ultimately block the holes; these deposits occur on the acid-wet face), and (3) particulate silica can blind spinneret holes or pass through and cause partial or complete discontinuity in yarn.

### 5. Apparatus

5.1 *Dish*, platinum.

5.2 *Stirring Rod*, plastic.

5.3 *Spectrophotometer*, for measuring absorbance at 700 nm.

### 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.<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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water.

6.3 In some cases, it may be necessary to select specific lots of chemicals to avoid high blank corrections. Minimize the use of glassware; particularly avoid holding alkaline solutions in glassware. Store all reagents in plastic bottles.

6.4 *1-Amino-2-Naphthol-4-Sulfonic Acid Solution*—Dissolve 7 g of anhydrous sodium sulfite ( $\text{Na}_2\text{SO}_3$ ) in 100 mL of water. Add 1.5 g of 1-amino-2-naphthol-4-sulfonic acid. Dissolve 90 g of sodium bisulfite ( $\text{NaHSO}_3$ ) in 800 mL of water. Blend the two solutions and filter through a double thickness of an acid-washed general purpose filter paper into a 1000-mL volumetric flask. Dilute to volume.

NOTE 1—Some batches of commercial aminonaphtholsulfonic acid are incompletely soluble or produce dark-colored solutions. These are not satisfactory for silica analysis. The reagent solution should always be discarded if a dark color appears with age. Stability of the solution is improved by refrigeration and protection from light.

6.5 *Ammonium Molybdate Solution*—Dissolve 100 g of ammonium heptamolybdate ( $(\text{NH}_4)_6\text{Mo}_7\text{O}_{23} \cdot 4\text{H}_2\text{O}$ ) in approximately 800 mL of water. Adjust to a pH of 8 by adding sodium hydroxide (NaOH). Dilute to 1000 mL.

NOTE 2—If necessary, filter the ammonium molybdate solution. With the pH adjusted to 8, the reagent is stable indefinitely. If the pH adjustment is not made, a precipitate will form on standing and the solution must be discarded. The added NaOH can be a significant source of  $\text{SiO}_2$  contamination. Use a 50 % NaOH solution that has not been in glassware. About 35 mL of this solution are required for 1000 mL of molybdate solution.

6.6 *Hydrochloric Acid Solution A*—Dilute 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 1 volume of water. Determine the volume of this acid that is required to adjust the pH of a solution of 1 g of sodium

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 15.04.

<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."