



Designation: D 2641 - 89

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St., Philadelphia, Pa. 19103

Reprinted from the Annual Book of ASTM Standards, Copyright ASTM
If not listed in the current combined index, will appear in the next edition.

Standard Test Method for Chlorine in Cellulose¹

This standard is issued under the fixed designation D 2641; 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 method² covers the determination of chlorine in cellulose. Procedures for total chlorine, water insoluble chlorine, and alcohol-benzene insoluble chlorine are described. The range is 5 to 1000 ppm on cellulose. Total chlorine determinations (including ashing) can be performed at a rate of one determination every 30 min.

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 1794 Test Method for Alcohol-Benzene Soluble Matter in Cellulose³

3. Summary of Test Method

3.1 The total chlorine determination is a measure of the water soluble chlorides as well as the chlorine bound in the pulp.

3.2 This test method makes use of the oxygen flask technique for the combustion of the pulp sample. After combustion, the chloride ions are absorbed in dilute sodium hydroxide, the solution is evaporated to dryness, and the residue is redissolved in a small amount of dilute nitric acid. The solution is diluted with acetone and titrated with 0.0025 M silver nitrate solution using a potentiometric titrator.

4. Significance and Use

4.1 Measurement of total chlorine levels gives the sum of water soluble chlorides and the organically bound chlorine compounds present in a pulp sample. Inorganic chlorine is known to be detrimental to the performance of many types of cellulose derivatives such as in viscose manufacturing.

4.2 Organic chlorine compounds are of interest from the environmental standpoint. The levels of organic chlorine compounds in pulp mills and in pulp products is under investigation in light of the suspected toxicity of these compounds to aquatic life.

5. Apparatus

5.1 *Potentiometric Titrator*, preferably equipped with a recorder. The use of a recorder is considered essential at low levels of chlorine.⁴

5.2 *Electrodes*, glass or mercurous sulfate reference electrode and silver-silver chloride indicator electrode.

5.3 *Combustion Flasks*, 1000-mL conical, borosilicate glass, with 35/25 ball and joint stopper and 5000-mL round bottom, borosilicate glass, with 65/40 ball and joint stopper. The flasks are equipped with perforated platinum sample carriers sealed into extended stem of flask stopper.

5.4 *Igniter*, suitable for igniting the sample in the flask. An "infrared igniter" is preferred.⁵

5.5 *Specimen Wrappers*, black paper.

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.⁶ 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 *Water*—Deionized and chloride-free. (It is essential that only this type of water be used in this test method.)

6.3 *Silver Nitrate, Standard Solution (0.0025 M)*—Dissolve 0.4247 g of silver nitrate (AgNO_3) in water, add 3 mL of concentrated nitric acid (HNO_3 , sp gr 1.42), and dilute to 1000 mL. Standardize daily against sodium chloride solution. 1 mL of 0.0025 M AgNO_3 solution is equivalent to 0.0886 mg of chlorine.

6.4 *Sodium Chloride, Standard Solution (0.0025 M)*—Dissolve 0.1462 g of sodium chloride (NaCl) in water and dilute to 1000 mL.

6.5 *Nitric Acid (1+100)*—Dilute 10 mL of concentrated nitric acid (HNO_3 , sp gr 1.42) to 1000 mL with water. Store in a glass-stoppered bottle.

6.6 *Sodium Hydroxide, Standard Solution (0.1 N)*—Transfer the contents of a standard concentrated volumetric ampoule into a polyethylene bottle and dilute to 1000 mL.

6.7 *Acetone*.

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

Current edition approved Oct. 27, 1989. Published December 1989. Originally published as D 2641 - 67. Last previous edition D 2641 - 70(1985)¹.

² See Rioux, J. P., and Hurtubise, F. G., "Determination of Chloride Ions in Pulp and Paper," *TAPPI*, Vol 48, January 1965, pp. 11-14.

³ *Annual Book of ASTM Standards*, Vol 15.04.

⁴ One suitable titrator-recorder is the Radiometer TTT-1 titrator equipped with the Radiometer SBU-1 titration assembly, manufactured by Radiometer America Inc., 811 Sharon Drive, Westlake, OH 44145.

⁵ The Thomas-Ogg apparatus manufactured by the Arthur H. Thomas Co., Philadelphia, PA, has been found satisfactory for this purpose.

⁶ "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."