



Designation: D3516 – 89 (Reapproved 2019)^{ε1}

Standard Test Methods for Ashing Cellulose¹

This standard is issued under the fixed designation D3516; 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.

^{ε1} NOTE—Editorial updates made to Section 2 and Footnote 4 in December 2019.

1. Scope

1.1 These test methods cover four ashing methods for cellulose. These are intended for use on unbleached and bleached cellulose in sheeted or bulk fiber form. Each one of the test methods has advantages, so that preference applications exist for all four.

1.2 The test methods appear as follows:

	Sections
Test Method A—Ash in Cellulose at 575°C	6 to 11
Test Method B—Sulfated Ash in Cellulose at 575°C	12 to 17
Test Method C—Ashing Cellulose by Schoniger Oxidation	18 to 22
Test Method D—Wet Ashing of Cellulose for Inorganics	23 to 29

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see 20.6.1.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

¹ These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.36 on Cellulose and Cellulose Derivatives.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

D1348 Test Methods for Moisture in Cellulose (Withdrawn 2017)³

3. Summary of Test Methods

3.1 *Test Method A, Ash in Cellulose at 575°C*—This test method measures the ash content of cellulose, which is defined for this test as the residue remaining after ignition at $575 \pm 25^\circ\text{C}$ until all carbon has been burned off. It is the simplest of the four test methods for the determination of ash content only, and it should not be considered as a standard preparative procedure for elemental analysis.

NOTE 1—The ash content at this ignition temperature is a reasonable measure of the mineral salts and inorganic foreign matter in the cellulose. The weight of ash obtained varies with the temperature of ignition. Higher temperatures (850°C) will convert calcium carbonate and other carbonates to the oxides and thus give lower values for ash. The composition of ash may vary with the pulping process employed for manufacture, which limits the significance of the ash determination in absolute terms.

3.2 *Test Method B, Sulfated Ash in Cellulose at 575°C*—In this ashing procedure the metal salts are converted to sulfates by treatment with sulfuric acid and ignition at $575 \pm 25^\circ\text{C}$ to overcome limitations of Test Method A. It should not be considered as a standard preparative test method for elemental analysis.

NOTE 2—Conventional dry ashing at high temperature (over 850°C) results in loss of carbon dioxide from metal carbonates, decomposition of magnesium sulfate, and loss of sodium salts. Ashing at lower temperatures (575°C) gives a better estimate of the inorganic impurities, but has the disadvantage of requiring a long ignition time and also the heat produced by the burning pulp can raise the temperature far above 575°C for a short period of time, converting calcium carbonate to the oxide in varying amounts. The sulfated ash method overcomes these objectionable features and has additional advantages. It is relatively insensitive to ignition time. Ash weight remains constant during weighing, and because the ash does not melt it will not attack the crucible.

3.3 *Test Method C, Ashing Cellulose by Schoniger Oxidation*—This test method is generally applicable to ashing cellulose for subsequent analysis of inorganic constituents. The procedure utilizes the Schoniger technique in which the cellulose is burned in a combustion flask and all combustion

³ The last approved version of this historical standard is referenced on www.astm.org.