



Designation: D3516 – 89 (Reapproved 2019)<sup>ε1</sup>

## Standard Test Methods for Ashing Cellulose<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

<sup>ε1</sup> 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:*<sup>2</sup>

D1193 Specification for Reagent Water

<sup>1</sup> 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.

Current edition approved Dec. 1, 2019. Published December 2019. Originally approved in 1976. Last previous edition approved in 2011 as D3516 – 89 (2011). DOI: 10.1520/D3516-89R19E01.

<sup>2</sup> 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)<sup>3</sup>

### 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 ( $850^\circ\text{C}$ ) 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  $850^\circ\text{C}$ ) results in loss of carbon dioxide from metal carbonates, decomposition of magnesium sulfate, and loss of sodium salts. Ashing at lower temperatures ( $575^\circ\text{C}$ ) 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  $575^\circ\text{C}$  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

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

products are dissolved in an aqueous medium, thus avoiding the possibility of physical loss of sample such as can occur in dry ashing (Test Methods A and B). A limitation is the relatively small sample of 1 g, which can be ashed without excessively large apparatus in which to carry out the oxidation step. Small specimens may not be adequately representative where a particulate contaminate is involved.

**3.4 Test Method D, Wet Ashing of Cellulose for Inorganics**—This test method is recommended for ashing cellulose for subsequent analysis of inorganic constituents. The procedure employs saturation of the cellulose with a concentrated solution of hydrogen peroxide followed by incremental addition to a small volume of concentrated sulfuric acid. It has the advantage over Test Method C of not requiring special apparatus and can be readily applied to samples of 5 to 10 g. The test method becomes tedious and less convenient for use where larger specimens such as 50 or 100 g are required. Specimens this large, although not generally required, may be necessary for the determination of trace levels of constituents such as manganese or silica. Smaller samples may not be adequately representative where a particulate contaminate is involved.

#### 4. Significance and Use

4.1 Ash content gives an estimation of the inorganic content of cellulose samples. The presence of high levels of ash can be expected to be detrimental to the process of making cellulose derivatives. It also provides a rough estimate of silica content which can have a significant effect on the performance of filters in cellulose derivative manufacturing facilities.

#### 5. Purity of Reagents

5.1 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.<sup>4</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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined in Specification **D1193**.

#### TEST METHOD A—ASH IN CELLULOSE AT 575°C

#### 6. Apparatus

6.1 *Balance*, with an accuracy to 0.05 g is required for weighing cellulose samples.

6.2 *Analytical Balance*, with a sensitivity of 0.1 mg is required. It should be checked with Class S weights.

6.3 *Drying Oven*, with the capability of maintaining a temperature of  $105 \pm 2^\circ\text{C}$ .

6.4 *Desiccator*, such as silica gel, indicating drierite, or magnesium perchlorate are suitable.

6.5 *Muffle Furnace*, electric, capable of maintaining a temperature of  $575 \pm 25^\circ\text{C}$ , is recommended.

NOTE 3—Back to front temperatures in muffle furnaces frequently exceed the  $\pm 25^\circ\text{C}$  range, so that operating limits should be established for a given furnace.

6.6 *Dish*, platinum, 100-mL capacity or larger. Porcelain crucibles are not recommended.

6.7 *Tongs*, approximately 0.5 m (20 in.) long, with nickel-chromium or platinum tips.

#### 7. Test Specimen

7.1 The amount of cellulose selected for each ash determination depends on the ash content of the test specimen and should be varied so that the weight of the ash will be not less than 10 mg and preferably over 20 mg. **Table 1** gives suggested sample sizes.

7.2 Selection of the specimen shall be in such a manner as to be representative of the lot being tested.

NOTE 4—No set sampling procedure has been established to be applicable to all samples. It will suffice to specify that the specimen shall be representative of the lot being sampled.

#### 8. Procedure

8.1 Weigh out, to the nearest 0.05 g, sufficient cellulose to give the correct range of ash weight (see **Table 1**). At the same time, a separate specimen (3 to 5 g) should be weighed and dried at  $105^\circ\text{C}$  for a moisture determination.

NOTE 5—Test Methods **D1348**, Method B is suitable. However, accuracy of the moisture test is not critical for the ash determination and any method capable of obtaining the percent oven dry within 0.5 % of the true value is adequate.

8.2 Heat the platinum dish in the muffle furnace at  $575^\circ\text{C}$  for 15 min, cool and weigh to the nearest 0.1 mg. Place the weighed cellulose in the platinum dish and place the dish on the open door of the muffle furnace, previously set and allowed to reach equilibrium at  $575^\circ\text{C}$ . Allow the cellulose to char without flame by gradually moving the dish into the muffle, and continue ashing with the door closed for 1 h.

NOTE 6—With some pulps, the character of the ash inhibits complete carbon removal and gray or black specks persist after 3 h of ignition. The addition of a few drops of distilled water to the ash after **8.3** followed by drying and reignition at  $575^\circ\text{C}$  for 1 h or more may be required. Extreme cases may require a second treatment with water.

NOTE 7—If the sample is too large, either separate ashings are required

**TABLE 1 Suggested Sample Sizes**

Ash, %	Moisture-Free Cellulose, g
Over 0.5	5
0.20 to 0.5	10
0.12 to 0.2	20
0.08 to 0.12	30
0.04 to 0.08	40
Less than 0.04	50

<sup>4</sup> ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

or successive additions of cellulose are made with extreme care so as not to disturb the ash. 8.2 is then repeated between each addition.

8.3 Remove the dish from the muffle furnace and allow to cool somewhat. Place it in the desiccator and allow it to cool to room temperature.

NOTE 8—Care must be taken at all times to keep drafts away from the light, fluffy ash.

8.4 Weigh the dish and ash to the nearest 0.1 mg. Reignite for a 15-min period and reweigh. Repeat as required to obtain constant weight.

## 9. Calculation

9.1 Calculate the ash percent of moisture-free cellulose,  $E$ , as follows:

$$E = \frac{(C - B)(100)(100)}{(A)(D)} \quad (1)$$

where:

- $A$  = weight of specimen as is, g,
- $B$  = weight of dish, g,
- $C$  = weight of dish plus ash, g, and
- $D$  = oven dry cellulose in specimen, %

## 10. Report

10.1 Report the results to the nearest 0.01 %. Duplicate determinations should check within approximately 5 %.

## 11. Precision and Bias

11.1 A precision of 8 % relative at the 95 % confidence level is found for specimens at the 0.05 % ash level.

11.2 No statement of bias can be made as no suitable reference material exists for determining bias.

## TEST METHOD B—SULFATED ASH IN CELLULOSE AT 575°C

## 12. Apparatus and Reagent

12.1 *Hot Plate.*

12.2 Additional apparatus shall be in accordance with Section 6.

12.3 *Sulfuric Acid*—Concentrated  $H_2SO_4$ .

## 13. Test Specimen

13.1 The amount of cellulose selected for each ash determination depends upon the sulfated ash content of the test specimen and should be varied so that the weight of the ash will be not less than 10 mg and preferably over 20 mg. Table 1 gives suggested specimen sizes for Test Method A. The table is applicable to sulfated ash also, providing the percent ash column is taken as percent sulfated ash.

NOTE 9—The sulfated ash values will be higher than the ash obtained by ignition of the cellulose without addition of the sulfuric acid. The magnitude of the difference will depend upon the proportions of different inorganic salts present, but based on the following conversion factors for the salts commonly present, will generally be about twice the regular ash:

$$\frac{MgSO_4}{MgO} = 2.99; \frac{CaSO_4}{CaO} = 2.43; \frac{MgSO_4}{MgCO_3} = 1.43 \quad (2)$$

$$\frac{CaSO_4}{CaSO_3} = 1.36; \frac{Na_2SO_4}{Na_2CO_2} = 1.34 \quad (3)$$

13.2 Selection of the specimen shall be in such a manner as to be representative of the lot being tested (see Note 4).

## 14. Procedure

14.1 Weigh out to the nearest 0.05 g sufficient cellulose to give the correct range of ash weight (see Table 1). At the same time, weigh a separate specimen (3 to 5 g) and dry to constant weight at  $105 \pm 2^\circ C$  for a moisture determination (see Note 5).

14.2 See 8.2.

14.3 Remove the dish and allow it to cool to room temperature (see Note 7 and Note 8).

14.4 Moisten the ash with 1 to 2 mL of concentrated  $H_2SO_4$  and heat on a hot plate until all the dense white fumes of sulfur trioxide ( $SO_3$ ) are liberated.

14.5 Place in the muffle furnace and ignite at  $575^\circ C$  for 1 h. If carbon persists repeat 14.3, 14.4, and 14.5.

14.6 After the last trace of carbon has disappeared, ignite in the muffle furnace for 1 h at  $575^\circ C$ .

14.7 Remove the dish and allow it to cool somewhat before placing it in desiccator and cooling to room temperature.

14.8 Weigh the dish and sulfated ash to the nearest 0.1 mg. Reignite for a 15-min period and reweigh. Repeat as required to obtain constant weight.

NOTE 10—To remove the ash from the dish, clean with boiling hydrochloric acid.

NOTE 11—On igniting sulfated ash at  $575^\circ C$ , there should be no fusion with the dishes, even when ashing cellulose that gives an alkaline ash.

14.9 Prepare a reagent blank for the  $H_2SO_4$  corresponding to the number of millilitres of  $H_2SO_4$  used in 14.4 and 14.5, by the addition of the acid to a platinum dish after ignition and true weight was determined, and carrying through 14.4 – 14.8 as they apply.

## 15. Calculation

15.1 Calculate the sulfated ash,  $S$ , as percent of moisture-free cellulose, as follows:

$$S = \frac{(C - B - R)(100)(100)}{(A)(D)} \quad (4)$$

where:

- $A$  = weight of specimen, g,
- $B$  = weight of dish, g,
- $C$  = weight of dish plus sulfated ash, g,
- $R$  = weight of  $H_2SO_4$  reagent blank residue, g, and
- $D$  = oven-dry cellulose in specimen, %.

## 16. Report

16.1 Report the results to the nearest 0.01 %. Duplicate determinations should check within approximately 5 %.

## 17. Precision and Bias

17.1 A precision of 15 % relative is found for specimens in the 0.1 to 0.3 % ash range.