



Standard Test Method for Ash in Pulp, Paper, and Paper Products¹

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1. Scope

1.1 This test method describes the determination of the ash content of pulp, paper, and paper products by ignition at two different temperatures:

1.1.1 *Method A*—Ash content upon ignition at 525°C.

1.1.2 *Method B*—Ash content upon ignition at 900°C.

1.2 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 585 Practice of Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, or Related Product²

D 644 Test Method for Moisture Content of Paper and Paperboard by Oven Drying²

D 686 Test Methods of Qualitative Examination of Mineral Filler and Mineral Coating of Paper²

D 1968 Terminology Relating to Paper and Paper Products²

3. Terminology

3.1 Definitions:

3.1.1 Definitions shall be in accordance with Terminology D 1968 and the *Dictionary of Paper*.³

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *ash at 525°C*—the ash content of the sample when the ignition temperature is 525°C.

3.2.2 *ash at 900°C*—the ash content of the sample when the ignition temperature is 900°C.

¹ This test method is under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and is the direct responsibility of Subcommittee D06.92 on Test Methods.

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² *Annual Book of ASTM Standards*, Vol 15.09.

³ Available from the Technical Association of the Pulp and Paper Industry, P.O. Box 105113, Atlanta, GA 30348.

4. Summary of Test Method

4.1 A test specimen of paper or paperboard is ignited in a muffle furnace at 525 or 900°C. A separate test specimen is analyzed for the percent moisture. The resulting weight of ash and moisture level in the sample are used to calculate the percent ash present at either of the specified temperatures on a moisture-free sample basis.

5. Significance and Use

5.1 The ash content of the sample may consist of various residues from chemicals used in its manufacture; metallic matter from piping and machinery; mineral matter in the pulp from which the paper was made; and filling, coating, pigmenting or other added materials. The amount and composition of the ash is a function of the presence or absence of any of these materials or others singly or in combination. No specific qualitative meaning is attached to the term “ash” as used in this test method. Where a further qualitative examination of the ash is desired, this method may be used in combination with Test Methods D 686, and major components of the ash identified.

5.2 Volatile decomposition products form from cellulose that is exposed to air at about 300°C. For papers or pulp containing no added fillers or coatings, ignition at *either* 525 or 900°C will yield essentially identical results of a few tenths percent ash or less. Examples of such papers include “ashless” filter papers manufactured for chemical analysis, or dissolving grade pulps.

5.3 Residue from cellulose products that contain oxides of silicon or titanium in fillers, coatings, or pigments may undergo negligible changes in weight when ignited at either 525 or 900°C. Where other fillers, pigments or coatings are known to be absent and where only silicon or titanium oxides are present, ignition at either temperature may be taken as a semi-quantitative measure of the percent of such material present in the sample.

5.4 In most cases, the ash content of paper and paperboard will contain inorganic residues from the pulp, inorganic residues from paper-making chemicals, and loading or filling materials deliberately added. In such cases, the significance of



the ash level determined will vary, depending upon which ashing temperature is used and the identity of the materials added.

5.5 For papers containing only cellulose and calcium carbonate, ignition at 525°C will remove cellulose and moisture, but will leave as ash the calcium carbonate essentially intact. Ignition at 900°C will convert the calcium carbonate to calcium oxide. In such cases, these methods may be used in conjunction to provide a good estimate of added calcium carbonate levels.

5.6 For papers containing cellulose and clays, or materials having variable chemical composition, variable thermal decomposition behavior, or both, ash level may require significant confirmation regarding the materials added, qualitative analysis of the ash as described in Test Methods D 686, and even then, care in determining data significance will be required.

5.7 The user of this test method must determine the correct ashing method to use and the significance of results based on an understanding of the composition of the sample ash and the information desired.

6. Apparatus

6.1 *Crucible*—A platinum crucible or dish with lid or cover is recommended; however, porcelain or silica crucibles may be used provided their weight does not change under the ignition conditions.

6.2 *Analytical Balance*, having a sensitivity of 0.1 mg.

6.3 *Electric Muffle Furnace*, controlled to maintain a temperature of

525 ± 25°C (977 ± 45°F) [Method A]

or

900 ± 25°C (1652 ± 45°F) [Method B]

7. Sampling

7.1 Sample the material in accordance with Practice D 585.

8. Test Specimen

8.1 Select small pieces of the sample to provide a representative specimen weighing at least 1 g moisture free. Increase the sample size, if necessary, to a size sufficient to yield at least 10 mg ash, and preferably greater than 20 mg ash.

8.2 Prepare sufficient test specimens to perform the procedure, 10.2.1 or 10.2.2, or both, at least twice for each test unit.

8.3 Weigh the sample on an analytical balance to the nearest 1.0 mg.

9. Determination of Moisture

9.1 At the same time the test specimen is being weighed (Section 8), weigh the sample for determination of moisture content as described in Test Method D 644.

10. Procedure

10.1 *Moisture Determination*—Continue and complete moisture determination as described in Test Method D 644.

10.2 *Ash Determination—Method A—Ignition at 525°C:*

10.2.1 Carefully clean the empty crucible and ignite in a muffle furnace at 525 ± 25°C for 30 to 60 min. After ignition, cool slightly and then place in a desiccator, containing

indicating-grade anhydrous alumina. When cooled to room temperature, weigh the ignited crucible on the analytical balance to the nearest 0.1 mg.

10.2.2 Transfer the test specimen to the crucible and, with the lid ajar, gently carbonize the specimen in the crucible on the hearth of the furnace or directly over a low flame of a bunsen burner. Alternately, place the crucible, with lid removed, in a furnace at about 100°C. Raise the temperature to 525°C slowly so that the sample becomes carbonized without flaming.

10.2.3 The sample must be charred, not burned, so that the temperature of the sample will not exceed 525°C. If the crucible is too small to hold the entire specimen, gently char the portion added and add more as the sample chars, but in either case protect the contents of the crucible with a lid or cover so that a portion of the ash will not be blown from the crucible or the sample will not burn, or both. When the residue has ceased to char, place the crucible with specimen into the furnace at 525 ± 25°C and remove the lid after the crucible seems to have reached the temperature of the furnace.

10.2.4 When the specimen is completely combusted, as indicated by the absence of black particles, remove the crucible from the furnace, replace the cover, and allow to cool somewhat; then place in a desiccator containing indicating grade anhydrous alumina and cool to room temperature. Weigh the crucible with ash to the nearest 0.1 mg. Repeat the ignition and weighing until the weight of the ash is constant to ± 0.2 mg.

10.3 *Ash Determination—Method B—Ignition at 900°C:*

10.3.1 Carefully clean the empty crucible and ignite in a muffle furnace at 900 ± 25°C for 30 to 60 min. After ignition, cool slightly and then place it in a desiccator, containing indicating-grade anhydrous alumina. When cooled to room temperature, weigh the ignited crucible on the analytical balance to the nearest 0.1 mg.

10.3.2 Transfer the test specimen to the crucible. Ignite the specimen in the crucible on the hearth of the furnace until well carbonized or directly over a low flame of a bunsen burner. If the crucible is too small to hold the entire specimen, gently burn the portion added and add more as the flame subsides, but in either case protect the content of the crucible with a lid or covers so that a portion of the ash will not be blown from the crucible. When the residue has ceased to burn with a flame, place the crucible with specimen into the furnace at 900 ± 25°C and remove the lid after the crucible seems to have reached the temperature of the furnace.

10.4 When the specimen is completely combusted, as indicated by the absence of black particles, remove the crucible from the furnace, replace the cover, and allow to cool somewhat; then place in a desiccator containing indicating grade anhydrous alumina and cool to room temperature. Weigh the crucible with ash to the nearest 0.1 mg. Repeat the ignition and weighing until the weight of the ash is constant to ± 0.2 mg.

11. Calculation

11.1 Calculate the percent of ash, based on the moisture-free weight of the paper as follows:

$$\text{Ash, \%} = \frac{A \times 100}{B} \quad (1)$$

where:

A = weight of ash, g, and

B = moisture-free weight of test specimen, g.

The moisture-free weight, B , g may be determined by multiplying the test specimen weight measured in Section 8 by the difference between 100 and the percent moisture (9.1) divided by 100, as follows:

$$B = \frac{W \times (100 - X)}{100} \quad (2)$$

where:

W = the test specimen weight (Section 8), and

X = % moisture in the sample (Section 9).

12. Report

12.1 Report the ashing temperature and the percent of ash for each test unit as the average of at least two determinations to the nearest 0.05 % for papers containing 5 % ash or less, to the nearest 0.1 % for papers containing 5 to 10 % ash, or to the nearest 0.2 % for papers containing more than 10 % ash.

13. Precision and Bias

13.1 Precision:

13.1.1 The results of duplicate ash determination should be suspected if they differ by more than the following:

Weight of Ash, mg	Maximum Permissible Difference, mg
Over 50	3
20 to 50	2
10 to 20	1

13.1.2 See Table 1 for repeatability of ash content at high temperature combustion.

13.1.3 Repeatability of lower temperature combustion was determined by testing 60 wood and 50 pulp samples in one laboratory by several operators. The ash content in the wood ranged from 0.16 to 0.84 % and had a repeatability of 0.03 %. The ash content in the pulp ranged from 0.24 to 1.60 % and had a repeatability of 0.04 %.

13.2 *Bias*—Results by this test method depend upon ashing condition (Method A, 525°C; Method B, 900°C), and are greatly influenced by the composition of non-cellulosic material present. There are no standard reference materials and bias does not apply.

14. Keywords

14.1 ash; paper; paperboard; pulp; wood

TABLE 1 Repeatability of Ash Content

NOTE 1—These data were obtained in one laboratory where 25 samples of each ash content level were tested by several operators.

Ash content, %	Repeatability, absolute (as ash content, %)	Repeatability, relative (as percent of ash content)
Less than 1	0.03	4.8
1 to 5	0.07	2.8
5 to 10	0.17	2.4
10 to 20	0.32	2.0
Over 20	0.42	1.4

APPENDIX

(Nonmandatory Information)

X1. ADDITIONAL INFORMATION

X1.1 Other published methods may be found where ignition is accomplished at temperatures other than the 525 or 900°C required in this test method. Depending upon the material present in the test specimen, particularly as described

in Section 5, significantly different results may be obtained when conditions other than those specified in this test method are employed.

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