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Standard Test Method for
Ash in Biomass[1]

This standard is issued under the fixed designation E1755; 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 method covers the determination of ash, expressed as the mass percent of residue remaining after dry oxidation (oxidation at 575 ± 25°C), of hard and soft woods, herbaceous materials (such as switchgrass and sericea), agricultural residues (such as corn stover, wheat straw, and bagasse), wastepaper (such as office waste, boxboard, and newsprint), acid and alkaline pretreated biomass, and the solid fraction of fermentation residues. All results are reported relative to the 105°C oven-dried mass of the sample. For particulate wood fuels, Test Method E1534 should be used.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *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:*[2]

E1534 Test Method for Determination of Ash Content of Particulate Wood Fuels

E1756 Test Method for Determination of Total Solids in Biomass

E1757 Practice for Preparation of Biomass for Compositional Analysis

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *ash*—the inorganic residue left after ignition at 575 ± 25°C.

4. Significance and Use

4.1 The ash content is an approximate measure of the mineral content and other inorganic matter in biomass.

4.2 The ash content is used in conjunction with other assays to determine the total composition of biomass samples.

5. Apparatus

5.1 *Ashing Crucibles, 50 mL,* platinum crucibles are preferred, but silica or porcelain crucibles may be used.

5.2 *Muffle Furnace,* an electric furnace is recommended for igniting the sample. The furnace should be fitted with an indicating pyrometer or thermocouple, so that the required temperature of 575 ± 25°C can be maintained.

5.3 *Analytical Balance,* sensitive to 0.1 mg.

5.4 *Desiccator,* using anhydrous calcium sulfate.

5.5 *Drying Oven,* with temperature control of 105 ± 2°C.

6. Test Specimen

6.1 Test specimens suitable for analysis by this test method are as follows:

6.1.1 Prepared biomass samples that have been prepared according to Practice E1757.

6.1.2 Biomass feedstocks, dried at 105°C according to Test Method E1756,

6.1.3 Pretreated biomass, dried at 105°C according to Test Method E1756, and

6.1.4 The solids fraction of fermentation residues, dried at 105°C according to Test Method E1756.

6.2 The test specimen shall consist of approximately 0.5 to 1.0 g of sample obtained in such a manner to ensure that it is representative of the entire lot of material being tested.

NOTE 1—All samples should be ground or milled to reduce the size of the large pieces to less than 1 mm in diameter. The sample is then redried at 105°C prior to testing. Prepared biomass can be used in place of 105°C dried material, but the mass of the material must be corrected for its moisture content by using Test Method E1756, prior to calculating the ash.

7. Procedure

7.1 Mark a pan or crucible with a unique identification using a porcelain marker, place it in the muffle furnace at 575 ± 25°C. Remove the pan or crucible from the furnace, cool to room temperature in a desiccator, and weigh to the nearest 0.1

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[1] This test method is under the jurisdiction of ASTM Committee E48 on Bioenergy and Industrial Chemicals from Biomass and is the direct responsibility of Subcommittee E48.05 on Biomass Conversion.

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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.

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mg. For an aluminum pan, two hours of heating at 575 ± 25°C will be sufficient to bring the pan to constant mass. With a crucible, however, the following procedure is used: place the crucible in the furnace at 575 ± 25°C for 3 h. Remove the crucible and place in a desiccator. Allow the crucible to cool to room temperature and then weigh the crucible to the nearest 0.1 mg. Record this mass. After weighing, return the crucible to the furnace for 1 h at 575 ± 25°C, cool again in the desiccator, and reweigh. Repeat this step until the mass of the crucible varies by less than 0.3 mg from the previous weighing. Record this final mass as the crucible tare mass, m_{cont}. Keep the pan or crucible in a desiccator until used.

7.2 Weigh approximately 0.5 to 1.0 g, to the nearest 0.1 mg, of a test specimen into the tared pan or crucible. If the sample being analyzed is a test specimen dried at 105°C, the sample should be stored in a desiccator until use. For a 105°C dried test sample, record the mass (container plus sample mass) as the initial mass, m_{od}. For prepared biomass samples prepped according to Practice E1757, record the mass (container plus sample mass) as the initial mass, m_{ar}.

NOTE 2—For prepared biomass samples, it is recommend that samples for moisture determination should be weighed out at the same time as the samples for the ash determination. If this is done at a later time it can introduce an error in the calculation because ground biomass can rapidly gain or lose moisture when exposed to the atmosphere.

7.3 Place the container and contents in the muffle furnace and ignite at 575 ± 25°C for a minimum of 3 h, or until all the carbon is eliminated. To avoid flaming, initially heat the sample to 250°C at a rate of 10 K/min and hold for 30 min before increasing the temperature to 575 ± 25°C. Avoid heating above the maximum stated temperature.

NOTE 3—For test specimens containing high amounts of ash (greater than 5 % by mass), it will be necessary to increase the time in the furnace to overnight to ensure complete elimination of the carbon. This ignition time period should not exceed 24 h.

7.4 Taking care to protect the test container from strong drafts to avoid mechanical loss of test specimen, remove the pan or crucible with its contents to a desiccator, cool to room temperature, weigh to the nearest 0.1 mg, and record this mass. Repeat the heating for one hour periods until the mass after cooling is constant to within 0.3 mg. Record the final mass of the ash, m_{ash}, as the container plus ash mass.

8. Calculation

8.1 For 105°C dried materials, calculate the mass percent of ash, as follows:

$$\%\ ash = [(m_{ash} - m_{cont})/(m_{od} - m_{cont})] \times 100 \qquad (1)$$

where:

$\%\ ash$ = mass percent of ash, based on 105°C oven-dried mass of the sample,
m_{ash} = mass of ash and container, g,
m_{cont} = tare mass of container, g, and
m_{od} = initial mass of 105°C dried sample and container, g.

8.2 For a prepared biomass sample, prepped according to Practice E1757, the following calculation may be used to report the mass percent of ash on a 105°C dried mass basis, provided that the test specimen has already been analyzed for mass percent total solids at 105°C using Test Method E1756.

$$m_s = (m_{ar} - m_{cont}) \times T/100 \qquad (2)$$

where:

m_s = mass of moisture-free solids in the prepared biomass sample, g,
m_{ar} = initial mass of prepared biomass sample and container, g,
m_{cont} = tare mass of container, g, and
T = mass percent of total solids in sample, as determined by Test Method E1756.

$$\%\ ash = [(m_{ash} - m_{cont})/m_s] \times 100 \qquad (3)$$

where:

$\%\ ash$ = mass percent of ash, based on 105°C oven-dried mass of the sample,
m_{ash} = mass of ash and container, g,
m_{cont} = tare mass of container, g, and
m_s = mass of moisture-free solids in the prepared biomass sample, g.

9. Report

9.1 Report the result to two decimal places, as a mass percent of the sample's 105°C dried mass, and cite the basis used in the calculation.

10. Precision and Bias

10.1 *Summary*—In comparing two single observations (Eq 1 only), the difference should not exceed 10.4 % of the average of the two observations in 95 out of 100 cases when both observations are taken by the same well-trained operator using this procedure and specimens randomly drawn from the same sample of material.[3,4] The true value of ash can only be defined in terms of this procedure. Within this limitation, this test method has no known bias.

10.2 *Interlaboratory Test Data*[5]—In interlaboratory testing run in 1993, randomly drawn samples of hybrid poplar were tested in each of five different laboratories. An operator in each laboratory tested duplicate specimens of the sample. The components of variance for ash results expressed as coefficients of variation were calculated to be:

Single-operator component	2.67 % of the average
Between-laboratory component	8.93 % of the average

NOTE 4—The square roots of the components of variance are being reported to express the variability in the appropriate units of measure rather than as the squares of those units of measure.

10.3 *Critical Differences*—For the components of variance reported in 10.2, two averages of observed values of the ash content should be considered significantly different at the 95 %

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[3] Technical Association of the Pulp and Paper Industry (TAPPI) Standard Methods, *Method T 211 om-85, Ash in Wood and Pulp*, 1991.

[4] Vinzant, T.B., Ponfick, L., Nagle, N.J., Ehrman, C.I., Reynolds, J.B., and Himmel, M.E., "SSF Comparison of Selected Woods From Southern Sawmills," *Applied Biochemical Biotechnology*, 45/46, 1994, pp. 611–626.

[5] A copy of the supporting data for the interlaboratory test is available from the Ethanol Project Chemical Analysis and Testing Task, National Renewable Research Laboratory, 1617 Cole Boulevard, Golden, CO 80401-3393.

probability level if the difference equals or exceeds the critical differences given in Table 1.

TABLE 1 Critical Difference, Percent of Grand Average, For the Conditions Noted[A,B]

Number of Observations in Each Average	Single Operator Precision	Between-Laboratory Precision
1	10.4	26.8
2	7.4	25.8

[A] The critical differences were calculated with $z = 1.960$.

[B] To convert the values of the critical differences to units of measure, multiply the critical differences by the average of the two specific sets of data being compared and divide by 100.

10.4 *Confidence Limits*—For the components of variance reported in 10.2, single averages of observed values have the 95 % confidence limits given in Table 2.

NOTE 5—The values of the critical differences and confidence limits should be considered to be a general statement, particularly with respect to between-laboratory precision. Before a meaningful statement can be made about two specific laboratories, the amount of statistical bias, if any, between them must be established, with each comparison being based on recent data obtained on specimens taken from a lot of material of the type being evaluated so as to be as nearly homogenous as possible and then randomly assigned to each of the laboratories.

TABLE 2 Width of 95 % Confidence Limits, Percent of Grand Average, For the Conditions Noted[A,B]

Number of Observations in Each Average	Single Operator Precision	Between-Laboratory Precision
1	±7.4	±19.0
2	±5.2	±18.2

[A] The critical differences were calculated with $z = 1.960$.

[B] To convert the values of the critical differences to units of measure, multiply the critical differences by the average of the two specific sets of data being compared and divide by 100.

10.5 *Bias*—This procedure for measuring ash has no bias because the value of ash is only defined in terms of this test method. Changes in ashing parameters may vary the mass of ash recovered. Prolonged heating of the ash beyond the times specified in 7.3 may cause the volatilization of alkali elements and cause a lower ash content.

11. Keywords

11.1 agricultural residue; ash; biomass; fermentation residue; herbaceous; waste paper; wood

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