Designation: E1756 – 08 (Reapproved 2015)

Standard Test Method for Determination of Total Solids in Biomass[1]

This standard is issued under the fixed designation E1756; 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.

INTRODUCTION

The total solids content is used to adjust the mass of the biomass so that all analytical results may be reported on a moisture-free basis. Total solids content may be determined by overnight drying at 105°C in a convection oven or with a loss-on-drying moisture analyzer.

1. Scope

1.1 This test method covers the determination of the amount of total solids remaining after drying a sample. Materials suitable for this procedure include samples prepared in accordance with Practice E1757 and extractive-free material prepared in accordance with Test Method E1690. For particulate wood fuels, Test Method E871 should be used.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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]

E871 Test Method for Moisture Analysis of Particulate Wood Fuels

E1690 Test Method for Determination of Ethanol Extractives 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 *oven-dried solids*—the solids remaining after heating the prepared biomass at 105°C to constant mass. For the purposes of this procedure, the moisture content of a biomass sample is considered to be the amount of mass lost during the drying of the sample at 105°C to constant mass. An inherent error of this and any oven-drying procedure is that volatile substances other than water are removed from the sample during drying.

3.1.2 *prepared biomass*—the biomass that has been processed according to Practice E1757.

4. Significance and Use

4.1 Moisture is a ubiquitous and variable component of any biomass sample. Moisture is not considered a structural component of biomass and can change with storage and handling of biomass samples. The determination of the total solids content allows for the correction of biomass samples to an oven-dried solids mass that is constant for a particular sample.

4.2 This procedure is not suitable for biomass samples that visibly change on heating to 105°C, for example, unwashed acid-pretreated biomass still containing free acid.

4.3 Some materials that contain large amount of free sugars or proteins will caramelize or brown under direct infrared heating elements used in Test Method B. Total solids in these materials should be done by Test Method A.

5. Apparatus and Materials

5.1 *Analytical Balance,* sensitive to 0.1 mg.

5.2 *Drying Oven,* 105 ± 3°C (Test Method A only).

5.3 *Desiccator,* containing anhydrous calcium sulfate (Test Method A only).

[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.

Current edition approved June 1, 2015. Published July 2015. Originally approved in 1995. Last previous edition approved in 2008 as E1756-08. DOI: 10.1520/E1756-08R15.

[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.

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

Test Conditions	Number of Observations in Each Average	Single Operator Precision
Test Method A, hybrid poplar	1	0.55
	2	0.39
Test Method A, fermentation residue	1	1.35
	2	0.95
Test Method B, hybrid poplar	1	0.56
	2	0.40
Test Method B, switchgrass	1	0.89
	2	0.63

[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.

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

Test Conditions	Number of Observations in Each Average	Single Operator Precision
Test Method A, hybrid poplar	1	0.39
	2	0.28
Test Method A, fermentation residue	1	0.95
	2	0.67
Test Method B, hybrid poplar	1	0.40
	2	0.28
Test Method B, switchgrass	1	0.63
	2	0.45

[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.

5.4 *Moisture Analyzer,*[3] infrared heated, 20 g capacity, 1 mg resolution (Test Method B only).

5.5 *Drying Pans,* disposable, aluminum, 10 cm diameter, suitable for moisture analyzer (Test Method B only).

6. Sampling

6.1 The sample is material prepared according to Practice E1757 or extractives-free material prepared according to Test Method E1690.

7. Procedure: Test Method A

7.1 This test method is suitable for either prepared biomass samples or extractives-free material and employs drying the sample at 105 ± 3°C in a drying oven.

7.2 Uniquely mark a suitable container, such as disposable aluminum weighing pan or 50 mL beaker, for each sample and place in the drying oven at 105°C for at least one hour. Cool the containers to room temperature in the desiccator.

7.3 Weigh each container on the analytical balance to the nearest 0.1 mg. Record this as the tare mass, m_t.

7.4 Weigh a nominal 0.5 g of sample into the marked, tared container to the nearest 0.1 mg. Record the mass of the biomass plus container as the initial mass, $m_{i\,1}$.

7.5 Place the sample in the drying oven at 105 ± 3°C for at least 3 h but not longer than 72 h. Allow the samples to cool to room temperature in a desiccator. Weigh each sample to the nearest 0.1 mg and record this mass. After weighing, return the samples to the drying oven at 105°C for 1 h, cool again in the desiccator, and weigh again. Repeat this step until the mass of the samples varies by less than 0.3 mg from the previous weighing. Record this mass as the final mass, m_{f1}.

8. Calculation: Test Method A

8.1 Calculate the mass percent of the total solids obtained by drying at 105°C as

$$\%T_{105} = (m_{f1} - m_t)/(m_{i1} - m_t) \times 100\,\% \quad (1)$$

where:

$\%T_{105}$ = mass percent of total solids based on 105°C dry mass,
m_t = tare mass of dried container,
$m_{i\,1}$ = initial mass of container and biomass, and
m_{f1} = final mass of container and biomass after drying at 105°C.

9. Procedure: Test Method B

9.1 This test method is suitable for either prepared biomass samples or extractives-free material and employs an automated moisture analyzer. This test method is not suitable for materials with bulk densities of less than 0.1 g/cm^3.

9.2 Set the standby temperature to 60°C and allow the instrument to warm-up for 30 min. Set the drying temperature to 105°C. The drying program should be set to end when the sample mass changes less than 0.05 % in mass per minute.

9.3 Tare a disposable aluminum drying pan on the analyzer's balance according to the manufacturer instructions.

9.4 Measure 2.0 ± 0.2 g of sample onto the drying pan. The sample must be spread in a thin, even layer that completely covers the bottom of the pan. Record the initial mass of the prepared biomass, m_{i2}, when the balance stabilizes.

9.5 Start the drying program according to the manufacturer's instructions. Upon termination of the drying cycle, record the final moisture-free solids mass, m_{f2}.

10. Calculation: Test Method B

10.1 Calculate the mass percent of the total solids obtained by drying at 105°C as:

$$\%T_{105} = (m_{f2}/m_{i2}) \times 100\,\% \quad (2)$$

where:

$\%T_{105}$ = mass percent total solids based on 105°C dry mass,
m_{i2} = initial mass of sample, and

[3] The sole source of supply of the Denver Instruments, Model IR-120 known to the committee at this time is Denver Instrument Company, 1401 17th St. Suite 750, Denver, CO 80202. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

m_{f2} = final mass of sample.

11. Precision and Bias

11.1 *Summary*—In comparing two single observations from the analysis of hybrid poplar using Test Method A, the difference should not exceed 0.55 % 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.[4] When analyzing washed, lyophilized fermentation residues by Test Method A, the difference should not exceed 1.35 %. In comparing two single observations from the analysis of hybrid poplar and switchgrass using Test Method B, the difference should not exceed 0.56 and 0.89 %, respectively. The true total solids value can only be defined in terms of this procedure. Within this limitation, this test method has no known bias.

11.2 *Critical Differences*—The observed total solids values should be considered significantly different at the 95 % probability level if the difference equals or exceeds the critical differences given in Table 1.

11.3 *Confidence Limits*—Single averages of observed values have the 95 % confidence limits given in Table 2.

NOTE 1—The values of the critical differences and confidence limits should be considered to be a general statement.

11.4 *Bias*—The procedure in this test method has no bias because the value of the total solids is defined in terms of the test method. Changes in the drying parameters may vary the observed total solids content.

12. Keywords

12.1 agricultural residue; biomass; fermentation residue; herbaceous; moisture; total solids; wastepaper; wood

[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 Biochemistry and Biotechnology* , Vol 45/46, 1994, pp. 611-626.