



Designation: E1690-01 ~~Designation: E 1690 – 08~~

Standard Test Method for Determination of Ethanol Extractives in Biomass¹

This standard is issued under the fixed designation E 1690; 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 ethanol soluble extractives, expressed as a percentage of the oven-dried biomass, of hard and soft woods, herbaceous materials (such as switchgrass and sericea), agricultural residues (such as corn stover, wheat straw, and bagasse), and wastepaper (such as office waste, boxboard, and newsprint). All results are reported relative to the oven-dried weight of the sample.

NOTE 1—This test method may not be suitable for hard and soft wood samples that include bark or those with more than 10% extractable materials.

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

D 1107 Test Method for ~~Alcohol-Benzene~~ Ethanol-Toluene Solubility of Wood

E 1756 Test Method for Determination of Total Solids in Biomass

E 1757 Practice for Preparation of Biomass for Compositional Analysis

3. Terminology

3.1 *Definitions:*

3.1.1 *ethanol extractives*—the residue remaining after evaporation of the ethanol solvent. These are materials that are removed from the biomass by extraction with 190 proof ethanol.

3.1.2 *oven-dried weight*—the moisture-free corrected weight of the prepared biomass sample. The correction factor is determined in accordance with Test Method E 1756.

3.1.3 *prepared biomass*—the biomass that has been prepared in accordance with Practice E 1757.

4. Significance and Use

4.1 Ethanol extractives are any materials found in biomass that are soluble in ethanol. They are not considered to be part of the structural components of biomass and should be removed prior to any chemical analysis of the sample. The prolonged extraction removes nonstructural materials that can include waxes, fats, resins, tannins, gums, sugars, starches, and pigments. Removing hydrophobic materials from the biomass makes it easier to wet the material for the analysis of structural components in the biomass.

4.2 This test method yields results comparable to Test Method D 1107.

5. Apparatus

5.1 *Soxhlet Extraction Apparatus*, glass, of a size (100 mL) suitable for containing the sample and a 250-mL collection flask.

5.2 *Alundum Extraction Thimbles*, medium porosity (10 to 15- μ m pore), sized to fit the Soxhlet extractor.

5.3 *Analytical Balance*, sensitive to 0.1 mg.

5.4 *Rotary Evaporator with Vacuum and Water Bath*, configured with a “bump” or safety trap, condenser, and receiving vessel. A water aspirator will generally provide sufficient vacuum (<150 torr).

5.5 *Vacuum Oven*, controllable to $40 \pm 1^\circ\text{C}$ and with vacuum between 75 and 100 torr.

¹ This test method is under the jurisdiction of ASTM Committee E48 on Biotechnology and is the direct responsibility of Subcommittee E48.05 on Biomass Conversion. Current edition approved November 10, 2001. Published February 2002. Originally published as E 1690-95. Last previous edition E 1690-95. Current edition approved May 1, 2008. Published May 2008. Originally approved in 1995. Last previous edition approved in 2001 as E 1690-01.

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.

6. Reagents and Materials

6.1 *Ethyl Alcohol*, 190 proof, USP grade.

7. Sampling

7.1 The sample is milled material prepared in accordance with Practice E 1757.

8. Procedure

8.1 Dry the Soxhlet extraction thimble in a drying oven at 105°C overnight. Remove it and allow it to cool to room temperature in a desiccator.

8.2 Weigh the Soxhlet extraction thimble to the nearest 0.1 mg, and record the weight as W_t . Add a suitable quantity of sample to the extraction thimble. Do not over fill the thimble; the largest sample should still leave approximately 1 cm of free space in the top of the thimble. Weigh to the nearest 0.1 mg, and record this weight as W_{st} .

NOTE 2—The precision for quantitation of the extractives weight is based on a nominal 7-g prepared sample weight. Smaller samples will introduce a larger relative error.

NOTE 3—The samples for total solids determination should be weighed out at the same time as those for extractives determination. If this is performed at a later time, it can introduce an error in the calculation because ground biomass can gain or lose moisture rapidly when exposed to the atmosphere.

8.3 Place several boiling chips into a 250-mL flask. Weigh the flask, with chips, to the nearest 0.1 mg, and record this weight as the tare weight of the flask, W_f .

8.4 Assemble the Soxhlet apparatus using at least 160 mL of 190 proof ethanol. Insert the thimble, and heat at reflux for 24 h. Check the reflux rate periodically, and adjust the heating rate to provide four to five solvent exchanges per hour in the Soxhlet thimble. Approximately 100 to 120 solvent exchanges are required during the 24-h period.

8.5 Remove the thimble when the extraction time is complete, and transfer the sample carefully to a Buchner funnel. Remove any residual solvent by vacuum filtration, and wash the sample thoroughly with 190 proof ethanol, collecting all of the filtrate. Allow the biomass to air dry in the Buchner funnel while it is still attached to the vacuum system.

NOTE 4—If the extractives-free sample is to be analyzed further, store the air-dried extractives-free sample in a freezer at -10°C until needed.

8.6 Combine the filtrate from the previous step and any solvent from the upper section of the Soxhlet apparatus with the solvent in the 250-mL flask. Place the flask on the rotary evaporator, and remove the solvent under vacuum (75 to 100 torr). Use a water bath temperature of $45 \pm 5^\circ\text{C}$ to heat the flask during evaporation.

8.7 Place the flask in a vacuum oven (75 to 100 torr) at $40 \pm 1^\circ\text{C}$ for 24 ± 1 h after all of the visible solvent is removed by the rotary evaporator. Remove the flask at this time, and allow it to cool to room temperature in a desiccator. Weigh the flask to the nearest 0.1 mg. Record this weight as W_{ef} .

9. Calculation

9.1 Calculate the weight of oven-dried biomass used for the extraction:

$$W_s = (W_{st} - W_t) \times (T/100 \%) \quad (1)$$

where:

W_s = weight of moisture-free solids in the prepared sample, g,

W_{st} = weight of sample and thimble (8.2), g,

W_t = tare weight of thimble (8.2), g, and

T = percentage of total solids in the sample, as determined in accordance with Test Method E 1756.

9.2 Calculate the percent of extractives on a 105°C oven-dried weight basis:

$$\% \text{ extractives} = [(W_{ef} - W_f)/W_s] \times 100 \% \quad (2)$$

where:

% extractives = percentage of extractives based on the oven-dried weight,

W_{ef} = weight of extractives, flask, and boiling stones (8.7), g,

W_f = tare weight of flask with boiling stones (8.3), g, and

W_s = weight of moisture-free solids in the prepared sample (9.1), g.

10. Report

10.1 Report the percent extractives based on the oven-dried weight of the sample.

11. Precision and Bias

11.1 *Precision:*

11.1.1 Data obtained by replicate testing of a hybrid poplar sample in one laboratory yielded a standard deviation in extractive content of 0.15 % and a CV % of 7.6 %. Replicate testing of a National Institute of Standards and Technology (NIST) No. 8494 wheat straw yielded a standard deviation of 0.20 % and a CV % of 1.6 %, and NIST No. 8493 *Pinus radiata* (Monterey Pine) yielded a standard deviation of 0.20 % and a CV % of 8.0 %.