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.
- 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:
- D 1107 Test Method for Alcohol-Benzene Solubility of Wood²
- E 1756 Test Method for Determination of Total Solids in Biomass³
- E 1757 Practice for Preparation of Biomass for Compositional Analysis³

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 Extractives are any materials found in biomass that are

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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-µm 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.

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_{st} . 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 1—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.

² Annual Book of ASTM Standards, Vol 04.10.

³ Annual Book of ASTM Standards, Vol 11.05.