

Designation: E1721 - 01(Reapproved 2009)

Standard Test Method for Determination of Acid-Insoluble Residue in Biomass¹

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INTRODUCTION

Biomass is composed largely of the following: cellulose, a polymer of glucose; hemicellulose, a complex polymer, the main chain of which consists of xylans or glucomannans; and lignin, a complex phenolic polymer. The lignin is mostly insoluble in mineral acids, unlike the other cell wall components of biomass. For this reason, lignin can be analyzed gravimetrically after hydrolyzing the cellulose and hemicellulose fractions with sulfuric acid.

1. Scope

- 1.1 This test method covers determination of the acid-insoluble residue 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 ovendried weight of the sample.
- 1.2 The residue collected contains the acid-insoluble lignin and any condensed proteins from the original sample. An independent nitrogen analysis would be required to determine the acid-insoluble lignin content separate from the condensed protein fraction and is outside the scope of this test method.
- 1.3 A portion of the lignin in some biomass samples will remain soluble during this procedure. The total lignin in a biomass sample includes both acid-soluble lignin and lignin in the acid insoluble residue.
- 1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.5 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. Specific hazards statements are given in Section 8 and Note 2 and Note 4.

2. Referenced Documents

2.1 ASTM Standards:²

E1690 Test Method for Determination of Ethanol Extractives in Biomass

E1756 Test Method for Determination of Total Solids in Biomass

E1757 Practice for Preparation of Biomass for Compositional Analysis

3. Terminology

- 3.1 Definitions:
- 3.1.1 acid-insoluble residue—the solid residue, corrected for acid-insoluble ash, retained on a medium-porosity filter crucible after the primary 72 % and secondary 4 % $\rm H_2SO_4$ hydrolysis described in this test method. This material is primarily acid-insoluble lignin and any condensed proteins.
- 3.1.2 *prepared biomass*—material that has been treated in accordance with Practice E1757 in order to raise the total solids content above 85 %, based on an oven-dried solids weight.

4. Significance and Use

4.1 The acid-insoluble residue content is used in conjunction with other assays to determine the total composition of biomass samples.

5. Interferences

5.1 The results of acid-insoluble residue analysis are affected by the incomplete hydrolysis of biomass. The results will be biased high unless the sample is hydrolyzed completely.

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.