



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

This standard is issued under the fixed designation E 1721; 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

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 oven-dried 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 The values stated in SI units are to be regarded as the standard.

1.4 *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:

E 1690 Test Method for Determination of Ethanol Extractives in Biomass²

E 1756 Test Method for Determination of Total Solids in Biomass²

E 1757 Practice for Preparation of Biomass for Compositional Analysis²

¹ This test method is under the jurisdiction of ASTM Committee E-48 on Biotechnology and is the direct responsibility of Subcommittee E48.05 on Biomass Conversion.

Current edition approved Sept. 10, 1995. Published November 1995.

² *Annual Book of ASTM Standards*, Vol 11.05.

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 % H₂SO₄ 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 E 1757 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. Take care to mix the acid/biomass slurry thoroughly during the concentrated acid hydrolysis.

5.2 The results of acid-insoluble residue analysis are affected by the timing of the acid digestion steps. The insoluble residue will dissolve slowly into solution in an irreproducible fashion. The timing within this test method must be followed closely.

6. Apparatus

6.1 *Analytical Balance*, readable to 0.1 mg.

6.2 *Convection Oven*, with a temperature control of 105 ± 3°C.

6.3 *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