Designation: D 1106 – 96 (Reapproved 2001)

Technical Association of Pulp and Paper Industry
Standard Method T 222-om-83

Standard Test Method for Acid-Insoluble Lignin in Wood[1]

This standard is issued under the fixed designation D 1106; 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[2] covers the determination of the acid-insoluble lignin content of wood.

1.2 *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 precautionary statements are given in 6.1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1107 Test Method for Ethanol-Toluene Solubility of Wood[3]

3. Principle of Method

3.1 When wood is treated with strong acids the carbohydrates are hydrolyzed, leaving an insoluble residue which is determined as lignin. Since some of the wood extractives (oils, resins, fats, waxes, tannins, gums, and starch) would remain insoluble with the lignin, these are first removed by proper solvents. The 72 % sulfuric acid method for lignin contains two and sometimes three preliminary extractive treatments, namely: (*1*) with alcohol, to remove the catechol tannins; (*2*) with alcohol-benzene solution, to remove the resins, oils, fats and waxes; and (*3*) with hot water, to remove the remaining water-soluble materials.

3.2 The alcohol extraction is necessary in analysis of woods high in tannin; that is, oak, chestnut, redwood, etc. It has not been shown necessary in the more common pulpwoods, such as the various species of spruce, pine, fir, hemlock, poplar, birch, beech, and maple. It is recommended that for these woods the alcohol extraction be omitted unless it is desirable for a special purpose. In analysis of woods not listed, the desirabilty of the alcohol extraction depends upon the purpose of the analysis and the report should state whether or not alcohol extraction was used.

4. Significance and Use

4.1 Wood contains approximately 20 to 30 % lignin. Removal of the lignin is the primary objective of pulping and bleaching procedures. Determination of the lignin content provides information for the evaluation and application of these processes.

5. Apparatus

5.1 *Extraction Apparatus*—A compact form of Soxhlet extraction apparatus, with ground-glass joints, is preferable. The apparatus shall consist of the following items:

5.1.1 *Soxhlet Extraction Flask*, having a capacity of 250 mL.

5.1.2 *Soxhlet Extraction Tube*, 45 to 50 mm in inside diameter, having a capacity to the top of the siphon of approximately 100 mL and a siphon tube approximately 55 mm in height. Extraction tubes of these dimensions siphon more rapidly than extractors with higher siphon tubes.

5.1.3 *Condenser*, of the Hopkins inner-cooled type.

5.1.4 *Extraction Crucibles*, of Alundum or fritted glass and of medium or fine porosity.

5.2 *Filtering Crucibles*—Alundum, porous porcelain, or fritted-glass crucibles (all of fine porosity), or Gooch crucibles with a glass-fiber mat, are recommended for filtering the separated lignin. Glass crucibles cannot be used if the lignin is to be ashed.

6. Reagents

6.1 *Ethylene-Toluene Solution*—Mix 1.0 L absolute ethanol and 427 mL toluene. (**Warning—**Avoid inhalation of vapors and contact with skin.)

6.2 *Sulfuric Acid (72 %)*—Carefully pour 665 mL of H_2SO_4 (sp gr 1.84) into about 300 mL of water, with vigorous stirring, and after cooling, dilute to 1 L. Standardize against standard NaOH solution, using methyl orange indicator. Adjust the H_2SO_4 to a strength of 72 ± 0.1 % by addition of water or H_2SO_4 (sp gr 1.84) as may be found necessary. If desired, the

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[1] This test method is under the jurisdiction of ASTM Committee D7 on Wood and is the direct responsibility of Subcommittee D07.01 on Fundamental Test Methods and Properties.

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[2] For further information on this test method the following references may be consulted:

Bray, M. W., "Methods Used at the Forest Products Laboratory for the Chemical Analysis of Pulps and Pulpwoods," *Paper Trade Journal*, Vol 87, No. 25, December 20, 1928, p. 29.

Ritter, G. J., Seborg, R. M., Mitchell, R. L., *Industrial and Engineering Chemistry*, Analytical Edition, Vol 4, 1932, p. 202.

Ritter, G. J., and Barbour, J. H., *Industrial and Engineering Chemistry*, Analytical Edition, Vol 7, 1935, p. 238.

[3] *Annual Book of ASTM Standards*, Vol 04.10.

solution may be standardized by an accurate determination of its specific gravity. For 72 % H_2SO_4 the specific gravity at 20/4°C is 1.6338; for use of this specific gravity method appropriate tables should be consulted.

7. Test Specimen

7.1 The test specimen shall consist of 1 g of wood that has been ground to pass a 425-µm (40 mesh) sieve and thoroughly air-dried.

8. Procedure

8.1 Weigh two 1-g test specimens in tared glass-stoppered weighing bottles. Dry in an oven for 2 h at 100 to 105°C, replace and stopper, and cool in a desiccator. Loosen the stopper to equalize the pressure and weigh. Continue the drying for 1-h periods until the weight is constant. Calculate the percentage of moisture-free wood.

8.2 Weigh in the extraction crucibles two additional 1-g test specimens for the lignin determination in duplicate. Place the extraction crucible containing the specimen in a Soxhlet extraction apparatus. Extract with 95 % alcohol for 4 h, unless the wood is known not to contain catechol tannins, in which case this extraction with alcohol will not be required. Then extract the test specimen with ethanol-toluene solution as described in Test Method D 1107. Remove as much of the solvent by suction as possible and wash by suction with 50 mL of ethanol to remove the toluene. Remove the excess ethanol, transfer to a beaker, and digest with 400 mL of hot water in a steam or hot-water bath at approximately 100°C for 3 h. Filter, wash with 100 mL of hot water, and finally with 50 mL of ethanol to facilitate the removal of the test specimen from the crucible. After these preliminary extractions, let the specimen dry in the air.

8.3 Transfer all of the air-dried test specimen to a glass-stoppered weighing bottle or a small beaker with a glass cover and add slowly, while stirring, 15 mL of cold (12 to 15°C) $H_2 SO_4$ (72 %). Mix the specimen well with the acid by stirring constantly for at least 1 min. Allow to stand for 2 h, with frequent stirring, at a temperature of 18 to 20°C. A water bath may be necessary to keep the temperature within these limits. Wash the material into a 1-L beaker or Erlenmeyer flask, dilute to a 3 % concentration of $H_2 SO_4$ by adding 560 mL of distilled water, and boil for 4 h, either under a reflux condenser or in the nearly constant volume condition maintained by the occasional addition of hot water to the flask.

8.4 After allowing the insoluble material to settle, filter into a filtering crucible that has been dried at 100 to 105°C and weighed in a glass-stoppered weighing bottle. Wash the residue free of acid with 500 mL of hot water and dry the crucible and contents in an oven for 2 h at 100 to 105°C. Place in the weighing bottle, cool in a desiccator, loosen the stopper of the bottle, and weigh the contents of the crucible as lignin. Repeat the drying and weighing until the weight is constant.

8.5 If a correction for ash is desired, transfer the lignin to a tared platinum crucible and determine the ash by igniting at 900°C. If the lignin cannot be quantitatively transferred, it may be ashed in the filtering crucible, provided the latter has been ignited to constant weight before filtration of the lignin residue. Ignition cannot be performed in fritted-glass crucibles.

9. Report

9.1 Report the results as percentage by weight of lignin in moisture-free unextracted wood. If the wood was extracted with alcohol, or if the lignin was corrected for ash, state this in the report.

10. Precision and Bias [4]

10.1 An interlaboratory study conducted by nine laboratories on six woods indicates that the precision both within and between laboratories is approximately constant throughout the range of lignin content. A range of content from 19 to 30 % gave a repeatability of 0.34 and a reproducibility of 0.79.

11. Keywords

11.1 acid-insoluble lignin; wood

[4] Data in this section obtained form the Technical Association of the Pulp and Paper Industry, P.O. Box 105113, Atlanta, GA 30348.