Designation: ~~D1105 – 96 (Reapproved 2013)~~ D1105 – 21

~~Technical Association of Pulp and Paper Industry~~
~~Standard Method T 12 os-75~~

Standard Test Method for Preparation of Extractive-Free Wood[1,2]

This standard is issued under the fixed designation D1105; 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 preparation of extractive-free wood and is applicable to all North American woods. Extractives in wood consist of materials that are soluble in neutral solvents and that are not a part of the 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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in 4.2.

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Significance and Use

2.1 Extractives are materials soluble in neutral solvents. They are not generally considered part of the wood polymer structure. These materials should be removed before any chemical analysis of the wood. Ethanol-benzene extracts waxes, fats, some resins, and portions of wood gums. Hot water extracts tannins, gums, sugars, starches, and coloring matter.

3. Apparatus

3.1 *Soxhlet Extraction Apparatus*—A glass Soxhlet extraction apparatus of suitable size for containing the sample, and fritted-glass filters, or cellulose, or Alundum extraction thimbles of medium to coarse ~~porosity, will be required.~~ porosity are recommended. Bags of cotton cloth of fine weave and thoroughly ~~washed,~~ washed with extraction solvents, of a suitable size to fit within the body of the extractor, are also satisfactory in place of the thimbles. Alternatively, a small wad of cotton or a wire screen may be placed in the discharge tube of the extractor and the entire body of the extractor filled with the wood sample. A thin wire screen disk placed over the top of the material will prevent channeling by the dripping condensate.

4. Reagents

4.1 ~~*Ethyl Alcohol*~~ *Ethanol* (*95 %*).

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

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

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[2] This standard was originally based upon TAPPI Standard Method T12 os-75, which has been replaced by T 264 cm-07.

5. Sample

5.1 The sample shall consist of air-dry sawdust or coarsely milled wood that has been reduced by means of a Wiley mill so as to pass through a 250-µm (60 mesh) sieve and be retained on a 180-µm ~~sieve.~~(80 mesh) sieve. The milled sample should then be air-dried.

6. Procedure

6.1 Place a suitable quantity of the sample in the extraction thimble, being certain that the wood does not extend above the level of the top of the siphon tube. Extract for 4 h with ethanol-toluene mixture in the Soxhlet extraction apparatus. Extraction with each solvent should be carried out at a rate of not less than four siphonings per hour. Transfer the wood to a Büchner funnel, remove the excess solvent with suction, and wash the thimble and wood with ~~alcohol~~ethanol to remove the toluene. If the thimble is nearly full, a Gooch crucible of suitable size may be placed in the rim of the thimble to keep the sample together. Extraction with each solvent should be carried out at a rate of not less than four siphonings per hour. Return the wood to the extractor and continue the extraction with ethanol for 4 h, or longer if necessary, until the ethanol siphons over colorless.

6.2 ~~If the thimble is nearly full, a Gooch crucible of suitable size may be placed in the rim of the thimble to keep the sample together. Extraction with each solvent should be carried out at a rate of not less than four siphonings per hour.~~ Remove the wood from the thimble, spread it out in a thin layer, and allow it to dry in the air until free of ~~alcohol.~~ethanol. Transfer the material to a 7.5-L Erlenmeyer or Florence flask and extract successively with three 1-L portions of distilled water, heating the flask with each change of water for 1 h in a hot-water bath at 100°C. The water should be at boiling temperature before the addition of the wood and the flask in the bath should be entirely surrounded by the boiling water. After the third extraction with water is complete, filter on a Büchner funnel, wash with 500 mL of boiling distilled water, and allow the extracted material to become thoroughly dry in the air.

7. Precision and Bias

7.1 Statements of precision and bias are not applicable to this method.

8. Keywords

8.1 extractive-free wood; wood