Preparation of wood for chemical analysis
(Ten-year review of Classical Method T 264 cm-07: Proposed Reconfirmation)

1. Scope

1.1 This method describes a procedure for further preparation of wood that has been sampled for analysis in accordance with TAPPI T 257 “Sampling and Preparing Wood for Analysis.”

1.2 A method for determination of moisture content in wood is described.

2. Summary

Wood is ground to particles and extracted successively with acetone and hot water.
NOTE 1: Due to health hazards associated with the use of benzene, the ethanol-benzene mixture used previously has been replaced with acetone. The acetone extractives content is generally lower than that obtained with the ethanol-benzene mixture. However, subsequent extraction with hot water removes most of the compounds soluble in ethanol-benzene mixture but not in acetone, such as polyphenols and low molecular weight carbohydrates. The type and amount of extractives with successive extraction with acetone and water will, in most cases, be comparable to that removed with ethanol benzene mixture and water.

3. Significance

3.1 Classification

3.1.1 Wood for chemical analysis should be ground to a fine particle size to permit complete reaction of the wood with the reagents used in the analysis. T 257 specifies that the wood should be ground to pass a 0.4-mm (40-mesh) screen.

3.1.2 Chemical methods for analysis of wood typically call for utilization of the entire amount of material without further fractionation. The fine material might contain a disproportionate quantity of some wood constituents, and its removal could alter the chemical composition as analyzed.

3.1.3 In some chemical methods, the fine material interferes with the analysis. It clogs fine filters or passes through coarse filters, producing erroneous results. Comminuted wood samples need to have fines removed by fractionation.

3.2 Extraction

3.2.1 Wood extractives are materials soluble in neutral solvents and are not generally considered as part of the wood substance. These materials should be removed before any chemical analysis of wood substance, except where the extraction process and subsequent washing could interfere with certain chemical analysis.

3.2.2 The acetone removes mainly wood resin material, including fatty and resin acids, sterols, waxes and fats. Hot water is used to extract tannins, gums, sugars, starches, and coloring matter.

4. Apparatus

4.1 Extraction thimbles, alundum or fritted glass, coarse porosity; or pre-extracted paper.

NOTE 2: Fritted glass thimbles must be cleaned after each use by soaking overnight in a chromic acid cleaning solution.

4.2 Extraction apparatus, with ground glass joints, consisting of:

4.2.1 Extraction flask, 250 mL.
4.2.2 Soxhlet extraction tube, inside diameter 30-40 mm, capacity to top of siphon about 100 mL.
4.2.3 Graham or Allihn type condenser.
4.3 Glassware, weighing bottle, glass, preferably “shallow” type or weighing dish, aluminum; filtering flask, 1000 mL; Büchner funnel.
4.4 *Heating device*, to contain hot water; or steambath or electric heating mantle.

4.5 Chemical fume hood, having a face velocity of at least 24.4 m/min (80 ft/min) or as required by local safety regulations.

5. **Reagents and materials**

5.1 Acetone, reagent grade, minimum purity of 99.5% \( \text{CH}_3\text{COCH}_3 \) by volume, having a residue after evaporation of less than 0.001%. Water, Type II reagent water as described in ASTM D1193 or better. The relevant properties of Type II reagent water as follows:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total matter, max</td>
<td>0.1 mg/L</td>
</tr>
<tr>
<td>Electrical conductivity, max</td>
<td>1.0 µS/cm at 25°C</td>
</tr>
<tr>
<td>Minimum color retention time of potassium permanganate</td>
<td>60 min</td>
</tr>
<tr>
<td>Maximum soluble silica</td>
<td>not detectable</td>
</tr>
</tbody>
</table>

6. **Safety precautions**

6.1 Acetone is highly flammable; therefore approved electric or steam heating has been specified. This entire procedure must be performed in a chemical fume hood. Caution should be taken to avoid an open flame. Care should be taken to avoid inhalation, ingestion, or body contact. It is recommended to wear plastic gloves suitable for acetone. Be sure to refer to the chemical caution statement on page 1.

7. **Sampling and test specimens**

7.1 In accordance with T 257, obtain a composite sample of wood meal.

7.2 Take a specimen for extraction, sufficient for the chemical analyses to be performed including moisture determination.
8. Procedures

8.1 Preparation of extractive-free wood

8.1.1 Clean and dry the extraction flask. Place the material to be extracted in the extraction thimble and place the thimble in position in the Soxhlet apparatus. Place a small cone of fine mesh screen wire in the top of the thimble to prevent any loss of the specimen.

8.1.2 Extract with 200 mL of solvent for 6 to 8 h, keeping the liquid boiling briskly so that siphoning from the extractor is no less than four times per hour.

NOTE 3: The volume of solvent must be sufficient to ensure that there is at least 50 mL of solvent remaining in the extraction flask when the Soxhlet extraction tube is ready for siphoning.

8.1.3 After extraction with acetone, transfer the wood to a Büchner funnel and wash with suction using several portions of hot water to remove the acetone.

8.1.4 Transfer the sample to a 1000-mL Erlenmeyer flask and add 500 mL of boiling distilled water. Heat the flask for 1 h in the hot water bath. Cover the flask with a watch glass to prevent evaporation. Keep the water at boiling temperature and surround the flask in the bath with boiling water. After extraction, filter on a Büchner funnel through a Whatman No. 5 filter paper or equivalent and wash with 500 mL of boiling distilled water. Allow the wood to air-dry thoroughly.

8.1.5 Mix and store in an air tight container in a cool location. Determine the moisture content in wood (8.2) and withdraw specimens for analysis as required.

8.2 Moisture content

8.2.1 Weigh a specimen from 8.1.5 of approximately 2 g to the nearest 0.001 g (A) in a tared weighing bottle. Dry for 2 h in an oven at 105 ± 3°C, cool in a desiccator, replace stopper, and open the stopper momentarily to equalize the air pressure and weigh. Return bottle to the oven for 1 h; repeat the cooling and weighing as above for successive hourly periods until constant weigh (B) is reached, that is, until successive weighings do not change by more than 0.002 g.

8.2.2 Calculate the percentage of moisture in wood to the nearest 0.1%:

\[
\text{Moisture content, } \% = \left( \frac{(A-B)}{A} \right) \times 100 \text{ where A and B are the sample weights after subtracting the weight of the sample bottles.}
\]

9. Keywords

Sample preparation, Wood, Analysis, Chemical analysis, Moisture content, Ethanol, Benzene
10.  **Precision**

10.1  Precision, in accordance with the definitions in TAPPI T 1200 “Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility” is not applicable to the preparation of extractive-free wood.

10.2  Repeatability of moisture content by oven-drying was found in one laboratory to be 0.2% in terms of moisture content. Reproducibility and comparability = not known.

11.  **Additional information**

11.1  Effective date of issue: To be assigned.


11.3  This method was reclassified as Classical by committee action in 1997, and revised by Standard-Specific Interest Group (SSIG) vote in 2006. Revisions in this version were due to safety concerns about the use of benzene; in this revision, the chemicals were switched from benzene and ethanol to acetone.

**Reference**


*Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department.*