

T 280 pm-99

PROVISIONAL METHOD - 1999

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CAUTION:

This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this test method, the user should determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Acetone extractives of wood and pulp

1. Scope

1.1 This method describes a procedure for determining the amount of acetone-soluble, non-volatile material in wood and pulp.

1.2 Because of health, safety and regulatory concerns associated with the use of benzene and dichloromethane, acetone has been selected to replace the routine use of these solvents for the determination of extractives in wood and pulp.

2. Significance

2.1 For the purpose of this method, solvent-soluble materials or extractives in wood consist of those components that are soluble in acetone.

2.2 The acetone extractable content of wood is a measure of such substances as fatty acids, resin acids, sterols, waxes and non-volatile hydrocarbons. The amount is markedly influenced by seasoning or drying of the wood.

2.3 Since the pulping process usually removes most water-soluble and volatile compounds that are also soluble in organic solvents, the solvent extractable material in pulp may be considered to consist primarily of resin and fatty acids and their esters, waxes, and unsaponifiable substances.

2.4 Because acetone is both more polar and water-miscible than dichloromethane or benzene-ethanol, the quantity of acetone extractable material, especially in wood, may be higher than that found with the other solvents. This procedure will not give the same results as benzene-ethanol or dichloromethane extractions.

3. Apparatus

3.1 *Extraction thimbles*, alundum, porosity RA 98; fritted glass, coarse porosity; or pre-extracted paper.

3.2 *Extraction apparatus*, with ground glass joints. A compact form of Soxhlet apparatus is preferable, consisting of:

3.2.1 Soxhlet extraction flask of 250-mL capacity.

3.2.2 Soxhlet extraction tube, inside diameter 30-40 mm, capacity to top of siphon about 100 mL, height of siphon tube about 55 mm. (This type is specified because siphoning is more rapid than for extractors with higher siphon tubes.)

3.2.3 Graham or Allihn-type condenser.

3.3 *Weighing dish*, aluminum preferred; or weighing bottles.

3.4 *Heating device*, steambath or heating mantle.

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

4. Reagents and materials

Approved by the Chemical Properties and Pulp Properties Committee of the Process & Product Quality Division

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4.1 *Acetone*, Reagent Grade, minimum purity of 99.5% CH_3COCH_3 by volume, having a residue after evaporation of less than 0.001%.

5. Safety precautions

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 open flame. Care should be taken to avoid inhalation, ingestion, or body contact. Be sure to refer to the chemical caution statement on page 1.

6. Sampling and test specimens

6.1 For wood:

6.1.1 In accordance with TAPPI T 257 "Sampling and Preparing Wood for Analysis," obtain a sample of air-dried sawdust to pass a 0.40 mm (40 mesh) screen and determine its moisture content according to TAPPI T 264 "Preparation of Wood for Chemical Analysis." At least 4 g are required for determination in duplicate.

6.1.2 From the air-dry sample, weigh to the nearest 5 mg, in a tared extraction thimble, a specimen equivalent to 2 ± 0.1 g of moisture-free wood.

6.2 For pulp:

6.2.1 Obtain a representative sample of pulp, which should be air-dry, sufficient to provide two 10 g test specimens, and to provide for moisture determination. Cut or tear this sample into pieces approximately 10 mm square.

6.2.2 Weigh duplicate test specimens of approximately 10 g to the nearest 0.01 g in tared extraction thimbles. At the same time, weigh a separate specimen for an accurate moisture determination by oven drying as described in TAPPI T 210 "Sampling and Testing Wood Pulp Shipments for Moisture."

7. Procedure

7.1 Clean, then dry the Soxhlet extraction flask. Place the extraction thimble and specimen in position in the Soxhlet apparatus.

NOTE 1: For wood, place a small cone of fine-mesh screen wire or a fritted-glass disk on the top of the thimble to prevent any loss of the specimen.

Steps 7.2 through 7.4 should be conducted in a chemical fume hood.

7.2 Fill the extraction flask with 150 mL of acetone.

7.3 Connect the flask to the extraction apparatus, and start water flow to the condenser section. Adjust the heaters to provide a boiling rate which will cycle the specimens for not less than 24 extractions over a 4-5-h period. If the extraction is left unattended, a provision should be made to shut off the heat if the water and/or the electricity shuts off unexpectedly.

7.4 Remove the flask from the apparatus and partially evaporate the solvent in the extraction flask to a volume of 20-25 mL. Transfer the extract to the tared weighing dish by washing with small amounts of fresh solvent. Handle the weighing dish with forceps or tongs. Evaporate the solvent to near dryness while in the chemical fume hood.

7.5 Dry the dish and contents in an oven for 1 h at $105 \pm 3^\circ\text{C}$, cool in a desiccator, and weigh to the nearest 0.1 mg.

NOTE 2: Drying to constant weight is not feasible, due to volatility or chemical changes produced by prolonged drying.

7.6 Run a blank determination with the solvent used in the test. Evaporate 150 mL of the solvent to dryness, and weigh the residue to the nearest 0.1 mg. Correct the weight of the dried extract by the weight of residue found.