

T 204 cm-07

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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.

Solvent extractives of wood and pulp

1. Scope

1.1 This method describes a procedure for determining the amount of solvent-soluble, non-volatile material in wood and pulp. Three different solvent systems may be employed, the selection depending on the safety and reproducibility requirements of each particular laboratory. In general, dichloromethane extraction gives lower amounts of extractives than either acetone or ethanol/benzene. Extraction with 1/3 ethanol and 2/3 benzene typically gives the highest level of extractives due to the additional dissolution of low molecular weight carbohydrates and polyphenols. These generalizations do not apply to all wood species, including some tropical hardwoods containing high molecular weight polyterpene resins.

1.2 Because of health, safety and regulatory concerns associated with the use of benzene and dichloromethane, acetone has been added as an alternative solvent for the determination of extractives in wood and pulp.

2. Significance

2.1 Soluble materials or extractives in wood consist of those components that are soluble in neutral organic solvents. The amount of solvent extractable matter is markedly influenced by seasoning or drying of the wood.

2.2 The dichloromethane-extractable content of wood is a measure of such substances as waxes, fats, resins, sterols and non-volatile hydrocarbons.

2.3 The ethanol-benzene extractable content of wood consists of certain other dichloromethane-insoluble components, such as low-molecular-weight carbohydrates, salts, polyphenols and other water-soluble compounds in addition to those substances mentioned in 2.2.

2.4 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.

2.5 No single organic solvent is capable of removing all these substances and different solvents remove different combinations of components. The ethanol-benzene mixture appears to provide the most complete removal of

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residual solvent-extractable substances in pulp.

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 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*, steam bath 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

- 4.1 *Ethanol*, approximately 95% C₂H₅OH by volume, or denatured with 5% methanol, having a residue after evaporation of less than 0.005%.
- 4.2 *Benzene*, purified C₆H₆, having a residue after evaporation of less than 0.001%.
- 4.3 *Dichloromethane*, U.S.P. 98% CH₂Cl₂, having a residue after evaporation of less than 0.002%.
- 4.4 *Ethanol-benzene mixture*. Mix together 1 volume of ethanol and 2 volumes of benzene.
- 4.5 *Acetone*, reagent grade, minimum purity of 99.5% CH₃COCH₃ by volume, having a residue after evaporation of less than 0.001%.

5. Safety precautions

Ethanol, benzene and acetone are highly flammable materials; 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. Benzene is a particularly toxic substance. Care should be taken to avoid inhalation, ingestion, or body contact. Dichloromethane causes narcosis when inhaled and can be irritating to the eyes. Refer to the chemical caution statement of 1.2.

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 wood meal sufficient to pass a 0.40 mm (40 mesh) screen and determine the moisture content of the sample according to TAPPI T 264 "Preparation of Wood for Chemical Analysis." For each solvent used, at least 4 g are required for determination in duplicate.
 - 6.1.2 Place 2.0 ± 0.1 grams of the air-dry sample into a tared extraction thimble and weigh to the nearest 1mg.
- 6.2 *For pulp*:
 - 6.2.1 Obtain a representative sample of air-dry pulp sufficient to provide two 10 gram test specimens and two specimens for moisture determinations.
 - 6.2.2 Cut or tear representative pieces of the pulp and weigh duplicate test specimens of approximately 10 grams to the nearest 0.001 g in tared extraction thimbles. At the same time, weigh separate specimens for an accurate moisture determination by oven drying as described in TAPPI T 210 "Sampling and Testing Wood Pulp Shipments for Moisture."