

WI \_\_\_\_\_ 060802.05 \_\_\_\_\_

T \_\_\_\_\_ 204 \_\_\_\_\_

DRAFT NO. \_\_\_\_\_ 2 \_\_\_\_\_

DATE \_\_\_\_\_ March 12, 2007 \_\_\_\_\_

**TAPPI**

WORKING GROUP

CHAIRMAN \_\_\_\_\_ Michael Buchanan \_\_\_\_\_

SUBJECT

CATEGORY \_\_\_\_\_ Chemical Properties \_\_\_\_\_

RELATED

METHODS \_\_\_\_\_ See "Additional Information" \_\_\_\_\_

**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 (Proposed revision of T 204 cm-97) (Underscores and strikethroughs indicate changes from Draft 1)**

### **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 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% C<sub>2</sub>H<sub>5</sub>OH by volume, or denatured with 5% methanol, having a residue after evaporation of less than 0.005%.

4.2 *Benzene*, purified C<sub>6</sub>H<sub>6</sub>, having a residue after evaporation of less than 0.001%.

4.3 *Dichloromethane*, U.S.P. 98% CH<sub>2</sub>Cl<sub>2</sub>, 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% CH<sub>3</sub>COCH<sub>3</sub> 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  $2.0 \pm 0.1$  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."

## 7. Procedure

7.1 Place the extraction thimble with specimen in clean and dry Soxhlet extraction apparatus. Connect the Soxhlet extraction apparatus in upright position to a clean and dry round bottom extraction flask.

**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 must be conducted in a chemical fume hood.

7.2 Fill the extraction flask with 150 mL of the required solvent.

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 to 5 hour 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 to 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 hour at  $105 \pm 3^\circ\text{C}$ , cool in a desiccator, and weigh to the nearest 0.1 mg.

**NOTE 3.2:** 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.

## 8. Calculation

Calculate the extractive content as follows:

$$\text{Extractables, \%} = [(W_e - W_b)/W_p] \times 100$$

where

$W_e$  = oven-dry weight of extract, g

$W_p$  = oven-dry weight of wood or pulp, g

$W_b$  = oven-dry weight of blank residue, g

## 9. Report

9.1 Report the solvent soluble matter to the nearest 0.1%, as the average of the two determinations, based on the moisture-free wood or pulp.

9.2 Report the solvent used.

## 10. Keywords

Solvents, Extraction, Wood, Pulp, Ethanol, Benzene, Dichloromethane, Acetone, Volatility, Wood resin, Extractives, Fats, Hydrocarbons, Phytosterols, Sterols, Pitch, Wax

## 11. Precision

11.1 The precision of the method for wood is as follows: repeatability, 11% (ethanol-benzene) and 4% (dichloromethane); reproducibility, 20% (ethanol-benzene) and 21% (dichloromethane). These values were determined in accordance with definitions of these terms in TAPPI T 1206 "Precision Statement for Test Methods."

11.2 The above values were determined in a round robin test involving five laboratories testing two materials, pine and oak, with both solvents.

11.3 For pulp: Although reproducibility of this test with dichloromethane has not been measured, an interlaboratory study of ether solubility conducted with the TAPPI Reference Pulp (2) has produced the following precision data repeatability (within-laboratory) = 0.25%; reproducibility (among laboratories) = 0.44%. These are absolute values not expressed in the same terms as 11.1.

11.4 Performance data for Acetone extractives has been reported in the equivalent Pulp and Paper Technical Association of Canada (PAPTAC) ~~Canadian Pulp and Paper Association (CPPA)~~ Standard G.13 and G.20. For wood, the test precision was determined using data from 12 replicate determinations on each of two wood samples (pine and aspen). The pooled coefficient of variation of the 24 replicates was 3.3%. The repeatability, based on the average of duplicate tests, was 6.5%. For pulp, the pooled coefficient of variation of 22 replicate determinations on two pulp samples (TMP and kraft) was 6.3%. The repeatability, based on the average of duplicate tests, was 13%.

## 12. Additional Information

12.1 Effective date of issue: to be determined.

12.2 Related methods: ASTM D 1107, D 1108, D 1794; Canadian, PAPTAC G.13 G.20; German UZOPCUI IV/43; Scandinavian SCAN C7 and C8; ISO 624-1974.

12.3 This method has been reclassified as Classical by committee action in 1997.

12.4 The ethanol-toluene mixture in previous revisions of this method was removed from this procedure by committee action in 1997 due to problems with this mixture.

12.5 The previously available TAPPI Provisional Method for acetone extractives of wood and pulp (T 280 pm-99) has been withdrawn and is incorporated into this version of T 204.

## References

1. Levitin, N., "The Extractives of Birch, Aspen, Elm and Maple: Review and Discussion," *Pulp Paper Mag. Can.* **71** (16): 81 (August 21, 1970).
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*Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department.* ■

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T \_\_\_\_\_ 204 \_\_\_\_\_

DRAFT NO. \_\_\_\_\_ 1 \_\_\_\_\_

DATE \_\_\_\_\_ October 23, 2006 \_\_\_\_\_

**TAPPI**

WORKING GROUP

CHAIRMAN \_\_\_\_\_ Michael Buchanan \_\_\_\_\_

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