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CAUTION:

This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. 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 method, the user must 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.

Rosin in paper and paperboard

1. Scope

1.1 This method describes empirical procedures for the quantitative determination of rosin in paper and paperboard.

1.1.1 Total extractable resin includes rosin added as a sizing agent, natural resins from the pulp, fatty acids and esters added as sizing and defoaming agents, and other natural and synthetic resins.

1.1.2 The quantitative procedures aimed at determining rosin in the presence of volatile or non-volatile neutral extractives will include as rosin any fatty acid present in the alcohol extract.

1.2 Qualitative tests, indicating the presence or absence of rosin added as a sizing agent, will be found in Section 12; *Qualitative tests*.

1.3 This method is not suitable for the quantitative determination of fatty acids and fatty salts in paper due to partial esterification during extraction.

1.4 This method is not applicable to the determination of wet strength resins.

1.5 It has been found that melamine formaldehyde resin does not interfere with the rosin determination (1).

2. Significance

2.1 The reaction products of rosin size and aluminum salts are most commonly used internally to control permeability of paper to aqueous liquids (sizing). Rosin content may be expected to bear some relationship to sizing effectiveness; however, chemical and physical conditions during papermaking can have an overriding effect on the relationship.

2.2 Rosin content of paper is generally conceded to affect permanence and physical properties of paper.

3. Definitions

3.1 Rosin (colophony) is a natural resin derived from pine gum, tall oil, and the solvent extract of pine stumps, knots, and top wood. It is largely a mixture of resin acids combined with a small amount of neutral materials.

3.2 Rosin, as used in this method, also refers to various chemically modified (fortified) rosins used in making sizing agents.

4. Summary

4.1 The insoluble resinates present in the paper are hydrolyzed and dissolved by extraction with an acidulated alcohol extraction solvent. The alcohol extract is then extracted with ether, in which nonresinous materials such as inorganics, starch, and glue are insoluble, yielding a total extractable "resin." Neutral materials (hydrocarbon waxes and oils, esters, etc.) are separated from the total extract by saponification and extraction. Further details are given by Launer (2).

4.2 If mineral matter is present which reacts with HCl or is dissolved by it [see TAPPI T 421 "Qualitative (Including Microscopic) Analysis of Mineral Filler and Mineral Coating of Paper"], the sample is pretreated with aqueous HCl.

5. Apparatus

- 5.1 *Extraction apparatus*, Soxhlet, medium, 40 mm i.d. body, or equivalent.
- 5.2 *Gooch crucible*, 40-mL.
- 5.3 *Glass fiber filter papers*, 24-mm diameter.
- 5.4 *Crucible holder*, for 40-mL Gooch crucible.
- 5.5 *Filtering flask with side tube*, 250-mL.
- 5.6 *Beakers*, 250-mL.
- 5.7 *Separatory funnels*, two 250-mL or larger.
- 5.8 *Reflux condenser and flask*, 250-mL flask or larger.
- 5.9 *Steam bath*, for reflux condenser.
- 5.10 *Glass wool or fritted glass filter*
- 5.11 *Drying oven*, capable of maintaining an internal temperature of $105 \pm 3^\circ\text{C}$.
- 5.12 *Balance*, having a capacity of at least 125 g and readable to ± 0.0001 g.
- 5.13 *Chemical fume hood*, having a face velocity of at least 24.4 m/min (80 ft/min) or as required by local safety regulations.

6. Reagents and materials

- 6.1 *Hydrochloric acid*, concentrated HCl; also approximately 1M.

NOTE 1: CAUTION: handle with adequate ventilation, in a hood. Material is very corrosive and the breathing of fumes and contact with the skin should be avoided.

- 6.2 *Extraction solvent*, mixture of 4 mL of concentrated HCL in 1000 mL of 95% ethanol.
- 6.3 *Ether*, diethyl, anhydrous, *peroxide-free*.

NOTE 2: Material is *highly flammable* and must be handled with adequate ventilation, in a hood equipped with a down-draft exhaust system. *Avoid all potential ignition sources. Test the ether for the presence of peroxides by the following:* To a 25-mL glass-stoppered cylinder of colorless glass, add 10 mL of the ether to be tested and 1 mL of a freshly prepared 10% aqueous solution of potassium iodide (1 g KI in 10 mL distilled water). Shake the cylinder and contents occasionally over a 1-h period. Protect the cylinder and contents from light. At the end of the hour, view the contents of the cylinder transversely against a white background; no color should be seen in either liquid. If color is seen, the ether contains peroxides and must not be used.

NOTE 3: To eliminate the explosion and fire hazards associated with anhydrous ether, dichloromethane may be used as a substitute. If this is done, however, it must be so stated in the report, the values given in Section 11. Precision may not apply, and results may be slightly different.

If neutral materials are to be separated:

6.4 *Potassium hydroxide*, approximately 0.5M alcoholic solution KOH: add 34 g reagent KOH pellets to 1000 mL of 95% ethanol, 90% methanol, or 99% 2-propanol, and dissolve by shaking. Let stand 24 h and decant clear solution.

NOTE 4: Handle with care, avoiding contact with the skin and the breathing of mists during preparation.

6.5 *Sodium chloride*, NaCl crystals.

6.6 *Sulfuric acid*, approximately 2.5M H₂SO₄.

6.7 *Methyl orange indicator*.

7. Sampling and test specimens

Prepare from each test unit obtained in accordance with TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product" two or more test specimens of at least 10 g each, cut into strips about 6 x 40 mm (about 0.25 by 1.5 in.). Do not grind the paper, because additional nonresinous materials may then be extracted.

NOTE 5: For paperboard, such as containerboard, pressboard, binder board, and pasted board, it is advisable to split material into thinner portions to facilitate extraction of the resin whenever possible, prior to weighing.

8. Procedures

8.1 *Procedure A* to determine (total extractable resin).

8.1.1 Allow the specimen strips to reach moisture equilibrium with the atmosphere surrounding the balance and weigh 5 to 7 g of strips to the nearest 0.01 g. Unless known to within 1%, determine the moisture content of a separate specimen similarly conditioned, in accordance with TAPPI T 412 "Moisture in Paper and Paperboard."

8.1.2 If the paper contains mineral matter which reacts with or is dissolved by HCl (see TAPPI T 421), immerse the test specimen to be extracted in 1M HCl for 5 min, drain, wash free from acid, and dry at room temperature.

8.1.3 Place a thin layer of glass wool over the siphon tube entrance of the Soxhlet apparatus to prevent the paper from blocking the tube. Crease the strips to be extracted with small zigzag folds and place them lengthwise in the siphon cup of the extractor, taking care to avoid packing them tightly together.

NOTE 6: When using a Soxhlet extractor, it is permissible to weigh the paper in a coarse fritted glass thimble and place the thimble in the extractor cup.

8.1.4 Add sufficient extraction solvent to the siphon cup to completely cover the specimen and allow to stand for 5-10 min without draining. In addition, add to the extractor flask 1.5 times the volume required to fill the cup. Extract at such a reflux rate that about 250-350 mL of solvent is distilled per hour. Extract for a period of 2 h for uncoated or nonsurface-sized papers and 2.5 h for coated or surface-sized papers or for paperboards. Do not continue the extraction much longer than specified, since additional high molecular weight materials other than rosin might be extracted and reported as rosin.

8.1.5 When the extraction period is completed, transfer the solvent from the flask to a suitable beaker. Rinse the flask several times with alcohol into the beaker and evaporate on the steam bath until the odors of alcohol and HCl are no longer noticeable. Place the beaker in an oven at 105 ± 3°C for 15 min, cool to room temperature, and add 20 mL of anhydrous ether. The rosin should dissolve in from 5 to 30 s unless covered by foreign materials, in which case it should be scraped with a stirring rod.

8.1.6 Allow to stand 15 to 20 min to further the coagulation and settling of insoluble matter. Filter the solution and about 20 mL of ether rinsings of the beaker through the Gooch crucible fitted with the glass fiber filter paper into the filtering flask. Refilter the solution through the same paper if necessary to remove cloudiness. After filtering, rinse the filter 3 times with less than 20 mL of ether and quantitatively transfer the ether solution from the flask to a carefully dried and tared beaker.

8.1.7 Evaporate the ether to apparent dryness on the steambath, then dry in an oven at 105 ± 3°C for 15 min, cool, and weigh to the nearest 0.001 g. Repeat the drying and weighing until the weight is constant to ± 0.001 g. If weight loss is obtained on the third weighing, use Procedure C under 8.3 below.

CAUTION: Do not place ether-wet material in an oven with exposed electrical contact. Explosion and/or fire may result.