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An American National Standard
Modification of Technical Association
of Pulp and Paper Industry
Standard Method T 6 m-59

Standard Test Method for Alcohol-Benzene Soluble Matter in Cellulose¹

This standard is issued under the fixed designation D 1794; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of alcohol-benzene soluble matter in cellulose and is applicable to dissolving-type cellulose pulps prepared from cotton or wood.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

D 1348 Test Methods for Moisture in Cellulose²

2.2 Other Documents:

Occupational Safety and Health Standards and Interpretations, Part 1910³

3. Summary of Test Method

3.1 A sample is extracted with a 1 + 2 alcohol-benzene solution in a Soxhlet apparatus as a measure of the waxes, fats, resins, and oils present.

4. Significance and Use

4.1 This test method provides an estimate of waxes, lipids, resins, and other organic soluble residues that were not removed from the fiber during the pulping process.

5. Apparatus

5.1 *Extraction Apparatus*, Soxhlet, consisting of a 300-mL flask, an extractor with a 50 by 250-mm body, and a Hopkins type condenser.

5.2 *Extraction Thimble*, either (1) standard thickness paper, 43 by 123 mm, (2) Alundum, 45 by 127 mm, medium-porosity, or (3) glass with coarse-porosity fritted-glass disk, 45 by 130 mm.

5.3 *Extraction Heater and Support*, heater with three-heat switches.

5.4 *Oven*, gravity-convection, maintained at $105 \pm 3^\circ\text{C}$.

5.5 *Dish*, evaporating, milk, aluminum, 75 mm in diameter by 20 mm deep.

5.6 *Desiccator* with efficient desiccant such as anhydrous calcium sulfate, activated alumina, or magnesium perchlorate. Calcium chloride is not satisfactory.

6. Reagent

6.1 *Alcohol-Benzene Solvent*—Mix 1 volume of Formula 30 ethyl alcohol with 2 volumes of benzene.

NOTE 1: Caution—Benzene is regulated as a "cancer hazard" as outlined in OSHA Regulations.

7. Procedure

7.1 Weigh 8 to 12 g of loose pulp, to the nearest 0.01 g, into an extractor thimble that has previously been extracted with the alcohol-benzene solvent. For sheet pulp, cut a sample into strips about 1/4 in. (6.4 mm) wide and 3 in. (76 mm) long, and weigh about 20 g into a thimble. Weigh a separate portion for a moisture determination in accordance with Methods D 1348.

7.2 Place the extraction thimble with specimen in the extractor and connect the flask. Pour 250 mL of the alcohol-benzene solvent into the body of the extractor. Connect the assembled extractor to the condenser and place the flask on the extraction heater. Turn on the cooling water to the condenser and adjust the heater to cause the solvent to reflux at such a rate as to cause siphoning 6 to 8 times/h. Continue the extraction for 5 h, beginning the timing when the solvent first begins to drop from the condenser into the extractor.

7.3 When the extraction is complete, turn off the heater. Disconnect the flask at a time when most of the solvent has collected in the extractor.

7.4 Heat the evaporating dish in the oven at 105°C for 30 min, cool in a desiccator, and weigh to the nearest 0.0001 g. Filter the solution from the flask through a sheet of qualitative paper into the weighed drying dish. Rinse the flask and filter with three 5-mL portions of the alcohol-benzene solvent.

7.5 Place the dish on a steam bath and evaporate just to dryness. Then place the dish in the oven at 105°C for 1 h, cool in a desiccator, and weigh.

8. Calculation

8.1 Calculate the percent of alcohol-benzene soluble matter, S , on the dry basis as follows:

$$S = [(A - B)/W \times [(100 - M)/100]] \times 100$$

where:

A = weight of dish and residue, g,

B = weight of dish, g,

W = weight of specimen, g, and

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.36 on Cellulosics.

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² Annual Book of ASTM Standards, Vol 06.02.

³ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.