Designation: D 3971 – 89 (Reapproved 1999)

Standard Test Method for Dichloromethane-Soluble Matter in Cellulose[1]

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1. Scope

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

1.2 *This standard does not purport to address all of the safety concerns, if any, 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 Documents

2.1 *ASTM Standards:*

D 1348 Test Methods for Moisture in Cellulose[2]

3. Summary of Test Method

3.1 A sample is extracted with dichloromethane in a Soxhlet apparatus as a measure of the waxes, fats, resins, and oils present.

4. Significance and Use

4.1 Dichloromethane-soluble materials are typically referred to as extractives. These extractives are comprised of organic materials that originated in the wood or cotton. The measure is an indication of the efficiency of removal of these substances during pulping and bleaching. The extractive level is of concern to dissolving pulp users since the presence of large amounts of extractives could inhibit the processing of cellulose into the desired derivative.

5. Apparatus

5.1 *Extraction Apparatus*:

5.1.1 Soxhlet.

5.1.2 *Flask, 250-mL.*

5.1.3 *Extractor Tube*, with standard taper 45/50 top joint.

5.1.4 Allihn Standard Taper, 45/50 or

5.1.5 *Friedrichs Standard Taper*, 45/50 type condenser.

5.2 *Extraction Thimble*, either (*1*) standard thickness paper, 43 by 123 mm, (*2*) aluminum oxide, 34 by 100 mm, medium porosity, or (*3*) glass with coarse-porosity fritted-glass disk, 45 by 130 mm.

5.3 *Heating Device*—Steam bath or heating mantle.

5.4 *Oven*, maintained at 105 ± 3°C.

5.5 *Dish*, evaporating, disposable, light aluminum, 63 mm in diameter by 17.5 mm deep.

5.6 *Desiccator* with efficient desiccant.

6. Reagent

6.1 *Dichloromethane*, ACS, 99 % CH_2Cl_2, having a residue after evaporation of less than 0.002 %.

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 dichloromethane. For sheet pulp, cut a sample into strips about 10 mm wide and 70 mm long, and weigh about 20 g into a thimble. Weigh a separate portion for a moisture determination in accordance with Test Methods D 1348.

7.2 Place the extraction thimble with sample in the extractor and connect the flask. Pour 250 mL of the dichloromethane into the body of the extractor. Connect the assembled extractor to the condenser and place the flask in the heating device. Turn on the cooling water to the condenser and adjust the heating rate to cause siphoning 6 to 8 times per hour. Continue the extraction for 5 h.

7.3 Heat the evaporating dish in the oven at 105°C for 30 min, cool in a desiccator, and weigh to the nearest 0.1 mg.

7.4 When the extraction is complete, disconnect the flask at a time when most of the solvent has collected in the extractor. Partially evaporate the solvent in the extraction flask to a volume of 15 to 20 mL. Transfer the extract to the tared weighing dish by washing with three 5-mL portions of fresh solvent.

7.5 Place the dish on a steam hot plate and evaporate just to dryness. Then place the dish in the oven at 105°C for 1 h, cool in a desiccator, and weigh to the nearest 0.1 mg.

[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.36 on Cellulose and Cellulose Derivatives.

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[2] *Annual Book of ASTM Standards*, Vol 06.03.