

T 237 cm-08

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OFFICIAL STANDARD – 1977
OFFICIAL TEST METHOD – 1983
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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.

Carboxyl content of pulp

1. Scope

This method describes a procedure for the determination of the content of carboxyl groups in bleached and delignified pulps. Lignin and lignin degradation products, especially sulfonic acid groups in sulfite pulps (1) interfere with the determination of carboxyl content, and the method is not applicable to unbleached and semi-bleached pulps.

2. Summary

Pulp is extracted (de-ashed) with dilute hydrochloric acid, washed, reacted with sodium bicarbonate-sodium chloride solution, and filtered. The filtrate is titrated with 0.01N hydrochloric acid to methyl red end point (2).

3. Significance

Carboxyl groups represent the ion-exchange capacity of a pulp, i.e., the ability to absorb metallic cations during the processing. In paper pulps the carboxyl groups contribute to the bonding of fibers and formation of paper and to the retention of rosin size. On the other hand, the absorbed cations are largely responsible for discoloration of pulp and paper on drying. Stability and electric properties of condenser paper depend mainly on the amount of metal ions bound by the carboxyl groups. In dissolving pulps carboxyl groups increase viscosity and decrease pulp solubility.

4. Definition

The carboxyl groups, -COOH, are acidic groups attached to cellulose chains and are formed mainly by oxidation of cellulose during pulping and bleaching processes. A certain level of carboxyl groups is also associated with hemicellulose.

Approved by the Standard Specific Interest Group for this Test Method

5. Apparatus

- 5.1 *Disintegrator*, such as a Waring or an equivalent blender or mixer, suitable for wet disintegration of pulp.
- 5.2 *Filtering funnels*, 150-mL and 300-mL, with fritted glass discs of coarse porosity (nominal pore size 40 to 50 μm).
- 5.3 *Filtering flasks*, 250-mL and 500-mL.
- 5.4 *Other equipment*: Erlenmeyer flask (tared), 250-mL, with glass stopper; pipets, 25- and 50-mL; buret, 50-mL; graduated cylinder, 250-mL; beaker, 600-mL; glass stirring rods.

6. Reagents

- 6.1 *Hydrochloric acid*, 0.1N HCl, not standardized. Add 8.45 mL concentrated HCl to approximately 900 mL distilled water. Make to a total volume of 1000mL.
- 6.2 *Hydrochloric acid*, 0.010N HCl, standardized to the nearest 0.0002N. (See TAPPI T 610 "Preparation of Indicators and Analytical Reagents, and Standardization of Volumetric Solutions" for preparation and standardization of HCl.)
- 6.3 *Sodium hydroxide solution*, 0.01N NaOH, not standardized. Dissolve 0.40 g of solid NaOH in 1000 mL of water.
- 6.4 *Sodium bicarbonate-sodium chloride solution*. Dissolve 0.84 g of NaHCO_3 and 5.85 g of NaCl in water and dilute to 1000 mL.
- 6.5 *Methyl red indicator*, 0.1% solution, in ethanol.
- 6.6 *Distilled water*, saturated with carbon dioxide (CO_2) gas.

7. Sample

Obtain a sample of bleached pulp, equivalent to about 10 g oven-dry, in accordance with TAPPI T 210 "Weighing, Sampling and Testing Pulp for Moisture." If the sample is a pulp sheet, tear or cut it into pieces about 10 mm across.

8. Test specimens

- 8.1 Allow the sample to come to moisture equilibrium in the atmosphere near the balance and weigh out two test specimens of 2.5 ± 0.1 g to the nearest 1 mg.
- 8.2 At the same time weigh out a specimen for moisture determination in accordance with TAPPI T 210 "Sampling and Testing Wood Pulp Shipments for Moisture."

9. Procedure

- 9.1 Disintegrate the test specimen in distilled water and filter on a 300-mL filtering funnel. Place the pulp pad in a 600-mL beaker, add 250 mL of 0.1N HCl and stir with a glass rod to disperse. Allow the dispersion to stand at room temperature for 120 min.
- 9.2 Filter the pulp on a 150-mL filtering funnel and wash with distilled water saturated with CO_2 . Continue washing until the last 10 to 20 mL of the filtrate, after boiling for 1 min, does not require more than 1 or 2 drops of 0.01N NaOH to give a yellow color with methyl red indicator.
- 9.3 Transfer the pulp quantitatively to a tared 250-mL Erlenmeyer flask and weigh to the nearest 0.1 g. Add 50.0 mL of sodium bicarbonate-sodium chloride solution, stopper the flask, and shake to obtain a homogeneous slurry. Allow to stand at room temperature for 60 min.
- 9.4 Filter the slurry on a dry 150-mL funnel into a clean and dry flask. Pipet 25.0 mL of the filtrate into an Erlenmeyer flask and titrate with 0.010N HCl using methyl red indicator. When the first change in color occurs, boil the solution for 1 min to expel the carbon dioxide and continue the titration to the pink end point. Record volume of 0.010N HCl consumed as "A."
- 9.5 Pipet 25.0 mL of the sodium bicarbonate-sodium chloride solution into an Erlenmeyer flask and titrate with 0.010N HCl as in 9.4. Record volume of 0.010N HCl consumed as "B."

10. Calculation

Calculate the carboxyl content in milliequivalents (meq) per 100 g of oven-dry pulp: