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Water-soluble sulfates in pulp and paper

1. Scope

- 1.1 This method is for the volumetric determination of water-extractable sulfate in pulp or paper.
- 1.2 The method is suitable for sulfate content from 0.01% up to 1%. It can be modified to extend this range.
- 1.3 This method does not measure total sulfur. It is not designed for the analysis of papers containing fillers composed of sulfides, sulfites, or sulfates. Sulfites and thiosulfates do affect results of the test.

2. Summary

A pulp or paper specimen is disintegrated in water and boiled. The suspension is filtered, and the filtrate is then concentrated. Metallic ions are removed from the concentrate by passage through an ion exchange column. An aliquot of the deionized concentrate is diluted with alcohol, Arsenazo III indicator is added, an excess of barium perchlorate is added, and the excess is back titrated with sulfuric acid.

3. Significance

The sulfate content of rosin sized paper is important as an indicator of alum carried with the wet sheet.

4. Apparatus

- 4.1 *Laboratory blender*, approximately 1000 mL (1 qt) capacity, similar to high-speed kitchen type blenders.
- 4.2 *Büchner funnel and flask*, 150 mm.
- 4.3 *Filter paper*, 150 mm diameter, coarse texture.
- 4.4 *Balance*, laboratory, 100 g capacity, accurate to 0.001 g.
- 4.5 *Balance*, 2 kg capacity, accurate to 0.1 g.
- 4.6 *Beakers*, 100, 150, 250, and 1000 mL capacity; 1000 mL beaker tared to nearest 0.5 g.
- 4.7 *Hot plate*, preferably with temperature control.
- 4.8 *Ion exchange column*, an 11 x 300 mm column with Teflon stopcock is recommended, but other sizes are satisfactory (see Appendix for preparation and regeneration). A regular 50 mL buret may be used as a substitute.
- 4.9 *Magnetic stirrer*, with a 25 mm Teflon-coated bar.
- 4.10 *Pipet*, 5, 10, and 25 mL capacity.
- 4.11 *Buret*, 10 mL or larger.
- 4.12 *Graduated cylinder*, 250 mL.
- 4.13 *Funnel*, 80 mm diameter.

5. Reagents

Approved by the Standard Specific Interest Group for this Test Method

- 5.1 *Barium perchlorate*, 0.0100 N $\text{Ba}(\text{ClO}_4)_2$; dissolve 2.0 g $\text{Ba}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}$ in distilled water to make 1000 mL. Accurately standardize with 0.01N H_2SO_4 using test procedure.
- 5.2 *Sulfuric acid*, 0.01N H_2SO_4 . Prepare 0.100N H_2SO_4 by diluting 3.0 mL (5.5 g) of pure, concentrated H_2SO_4 (specific gravity 1.84) to 1000 mL with distilled water. Standardize with sodium carbonate using the procedure for hydrochloric acid in TAPPI T 610 "Preparation of Indicators and Analytical Reagents, and Standardization of Volumetric Solutions." Dilute exactly 100 mL of 0.100N H_2SO_4 to 1000 mL with distilled water.
- 5.3 *Arsenazo III indicator*, dissolve 0.2 g Arsenazo III in 100 mL of distilled water.
- 5.4 *Cation exchange resin*, Dowex 50 W-X8 or similar.
- 5.5 *Isopropyl alcohol*, reagent grade.
- 5.6 *Glass wool*.
- 5.7 *Hydrochloric acid*, approximately 1N. Dilute 85 mL of concentrated HCl (sp. gr. 1.20) to 1000 mL with distilled water.
- 5.8 *Magnesium carbonate*.
- 5.9 *Hydrogen peroxide*, 30%.
- 5.10 *Sodium hydroxide*, approximately 0.1N. Dissolve 4 g NaOH in distilled water and dilute to 1000 mL.

6. Sampling

6.1 Obtain a clean sample of pulp (in accordance with a predetermined sampling plan) or paper (in accordance with TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product") from the shipment or batch to be tested and tear into small pieces. Use clean rubber gloves and/or tongs, and be extremely careful not to contaminate the sample while handling.

6.2 If the moisture content is not known, determine it on a duplicate sample by drying to constant weight in accordance with TAPPI T 412 "Moisture in Pulp, Paper and Paperboard" or TAPPI T 550 "Determination of Equilibrium Moisture in Pulp, Paper and Paperboard for Chemical Analysis."

7. Test specimens

- 7.1 Weigh out duplicate specimens of approximately 10 g (oven dry basis) to the nearest 0.01 g.
- 7.2 Correct for moisture content (weight A).

8. Procedure

- 8.1 *Water extraction*
 - 8.1.1 Disintegrate pulp or paper specimen in 800 to 900 mL of sulfate-free distilled water using laboratory blender. Transfer the slurry with minimum distilled water rinse to a tared 1000 mL beaker. The slurry is at approximately 1% consistency.
 - 8.1.2 Gently boil slurry on hotplate for 60 min without water addition.
 - 8.1.3 Weigh beaker and contents to nearest 0.5 g (weight B).
 - 8.1.4 Filter slurry using Büchner funnel and coarse paper into clean, dry flask without rinsing. Transfer most of the filtrate into a dry tared beaker.
 - 8.1.5 Weigh beaker and contents to nearest 0.5 g (weight C).
 - 8.1.6 Concentrate filtrate to approximately 100 mL on hotplate.
 - 8.1.7 Weigh beaker and contents to nearest 0.5 g (weight D).
- 8.2 *Sulfate determination of concentrated extract*
 - 8.2.1 Pass 25 mL of extract from 8.1.6 through the ion exchange column and discard. Pass an additional 60 mL of extract through the column for analysis. This will have the same sulfate content as that entering the column.
 - 8.2.2 Pipet 25.0 mL of column effluent into a 250 mL beaker. A smaller volume should be used if effluent is expected to contain over 150 ppm sulfate.
 - 8.2.3 Add 125 mL isopropyl alcohol. The solution must contain over 70% alcohol for sharp end point.
 - 8.2.4 Add four drops of Arsenazo III indicator solution.
 - 8.2.5 Add 0.01N $\text{Ba}(\text{ClO}_4)_2$ from a buret to the solution while stirring with a magnetic stirrer until the solution turns blue. Add 1.0 additional mL. Record volume added.
 - 8.2.6 Back titrate with 0.01N H_2SO_4 to true rose pink color. The solution will gradually change from blue