

T 235 cm-09

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Alkali solubility of pulp at 25°C

1. Scope

- 1.1 This method for determination of alkali solubility can be applied to bleached and delignified pulps only.
- 1.2 Lignin interferes with the extraction and determination of carbohydrates, and the method cannot be applied to unbleached pulps.

2. Summary

Pulp is extracted with a sodium hydroxide solution of 10%, 18%, or 21.5% at 25°C for 1 h. The dissolved carbohydrates are determined by oxidation with potassium dichromate.

3. Significance

3.1 Low molecular weight carbohydrates (hemicellulose and degraded cellulose) can be extracted from pulps with sodium hydroxide solutions. Solubility of a pulp in alkali thus provides information on the degradation of cellulose and on a loss or retention of hemicellulose during pulping and bleaching processes (1,2).

3.2 In general, a 10% sodium hydroxide solution possesses maximum dissolving power and dissolves both degraded cellulose and hemicellulose.

3.3 Resistance of pulps to 21.5% sodium hydroxide solution has been related to the rayon yield of dissolving pulps (3).

4. Apparatus

4.1 *Pulp dispersion apparatus* (as in Fig. 1 or a similar apparatus), consisting of a variable speed motor and a stainless steel stirrer with a shell. The speed of the motor and the angle of the blades should be adjusted so that no air is drawn into the pulp suspension during stirring.

Approved by the Standard-Specific Interest Group for the Test Method



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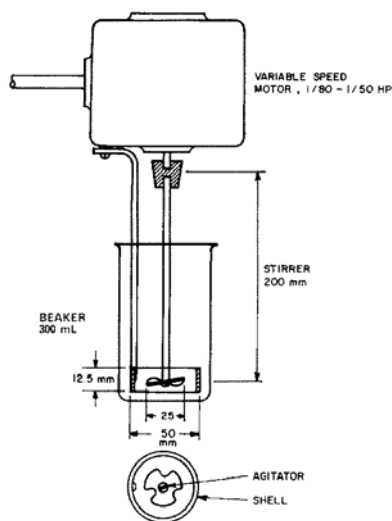


Fig. 1. Pulp dispersion apparatus

- 4.2 *Constant temperature bath*, to maintain a temperature of $25 \pm 0.2^\circ\text{C}$.
- 4.3 *Filtering flask*, 250 mL, and a filtering funnel or crucible, 30 to 50 mL, with a fritted glass disk of coarse (40 to 60 μm) porosity.
- 4.4 *Other equipment*: beakers, tall form, 300 mL; pipets, 5, 10, 25, and 75 mL; conical flasks, 250 mL; graduated cylinders, 25 and 50 mL; glass stirring rods.

5. Reagents

- 5.1 *Sodium hydroxide stock solution*, about 50%. Dissolve reagent-grade NaOH in distilled water and let stand until the suspended carbonates have settled; then decant the clear solution. (Solutions of 50% NaOH are also available commercially.)
- 5.2 *Sodium hydroxide reagent solutions*. Dilute the sodium hydroxide stock solution with CO_2 -free (recently boiled) distilled water and adjust the concentration as specified:
- 5.2.1 *10% (by weight) NaOH solution*, $2.77 \pm 0.005N$; 110.9 g of NaOH/L; specific gravity, 1.109 at $20^\circ/4^\circ\text{C}$.
- 5.2.2 *18% (by weight) NaOH solution*, $5.39 \pm 0.005N$; 215.5 g of NaOH/L; specific gravity, 1.197 at $20^\circ/4^\circ\text{C}$.
- 5.2.3 *21.5% (by weight) NaOH solution*, $6.64 \pm 0.005N$; 265.7 g of NaOH/L; specific gravity, 1.236 at $20^\circ/4^\circ\text{C}$.
- 5.3 *Potassium dichromate solution*, 0.5N. Dissolve 24.52 g of $\text{K}_2\text{Cr}_2\text{O}_7$, in water and dilute to 1 L.
- 5.4 *Sulfuric acid*, concentrated H_2SO_4 , 96 to 98%; specific gravity, 1.84.
- 5.5 *Ferrous ammonium sulfate solution*, 0.1N. Dissolve 40.5 g of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ in water, add 10 mL of concentrated H_2SO_4 , and dilute to 1 L. The solution is not stable, and the exact normality should be determined daily by titration with 0.1N potassium dichromate standard solution.
- 5.6 *Phenanthroline-ferrous sulfate (ferroin) indicator solution*. Dissolve 1.5 g of 1,10-phenanthroline monohydrate, $\text{C}_{12}\text{H}_8\text{N}_2 \cdot \text{H}_2\text{O}$, and 0.7 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in 100 mL of water. (This indicator solution is also available commercially.)

6. Sampling

- 6.1 Obtain a sample of bleached pulp, equivalent to about 10 g oven-dry, in accordance with a predetermined sampling procedure.
- 6.1.1 If the sample is a pulp sheet, split it in layers and tear by hand into small pieces about 10 mm across. Do not cut or shred the pulp by mechanical devices.