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pH and electrical conductivity of hot water extracts of pulp, paper, and paperboard

1. Scope

This procedure provides for the extraction of pulp, paper, and paperboard samples using boiling reagent water followed by determination of the pH and conductivity of the extract.

2. Significance

2.1 pH measurement of the extract indicates presence of either acidic or basic ions which could contribute to potential degradation of the cellulose in the end product.

2.2 Conductivity measurement indicates the level of residual ionic impurities in pulp and thus not only shows the effectiveness of washing in the pulp manufacturing process but also identifies the suitability of pulp for use in electrical and antitarnish product applications. Extract conductivity of highly purified (high alpha) pulps increases on aging from the time of manufacture.

3. Apparatus

3.1 *pH meter*, any commercial pH meter that includes a glass electrode and a reference electrode or a “combination electrode.” The meter must be capable of indicating pH to 0.1 unit or less.

3.2 For referee testing, the pH meter and electrodes shall conform to ASTM Standard D 1293, Standard Test Method for water, Section 10, Apparatus, with the pH meter being of Type II, Type III, or Type IV of Table 2 of ASTM D 1293.

3.3 *Erlenmeyer flasks*, 500 mL, with standard taper joint.

3.4 *Water-cooled condensers*, with standard taper joints.

3.5 *Fritted glass funnel*, with suction flask.

3.6 *Miscellaneous glassware*: graduated cylinder, 1000 mL; 50 mL beaker for pH; tall-form beakers, 100 or 200 mL, for conductivity measurements.

NOTE 1: Proper cleaning of the glassware is essential. Soap or detergents should be avoided, and acid cleaning with ample rinsing is recommended.

Approved by the Standard Specific Interest Group for this Test Method

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- 3.7 *Electric hot plate.*
- 3.8 *Conductivity bridge*, with a dip-type cell having platinum electrodes securely mounted 1 cm apart. The face area of each electrode must be at least 2 cm². The electrodes should be platinized if measurements are made at low frequency (60 Hz). A commercial conductivity meter may also be used. See also Additional Information, Section 11.3.
- 3.9 *Constant temperature bath*, maintained at 25 ± 0.5°C.
- 3.10 *Thermometer*, accurate to 0.25°C.

4. Reagents

- 4.1 *Reagent water.*
- 4.1.1 ASTM D 1193, Specification for Reagent Water, describes procedures for preparing and testing water of the quality required by this test method. For referee testing, the term “water” as used in this test method should be understood to mean Type II or Type I reagent water as defined in ASTM D 1193.
- 4.2 *Standard buffer solutions.*

NOTE 2: For the purposes of this test method, all chemicals must be of reagent grade and conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used provided that it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

4.2.1 *Potassium hydrogen phthalate buffer*, pH 4.01 at 25°C. Dissolve 10.21 g of HKC₈H₄O₄ in water and dilute to 1000 mL.

NOTE 3: As the buffer capacity of this solution is rather low, protect the solution from contamination with acids and alkalis; however, the pH is not affected by atmospheric carbon dioxide. The solution should be replaced after six weeks, or earlier if mold appears.

4.2.2 *Phosphate buffer*, pH 6.86 at 25°C. Dissolve 2.40 g of monobasic potassium phosphate, KH₂PO₄, and 3.55 g anhydrous dibasic sodium phosphate, Na₂HPO₄, in carbon dioxide-free water and dilute to 1000 mL.

NOTE 4: The Na₂HPO₄ will absorb moisture at relative humidities exceeding about 40% at 25°C and should be dried for 2 h at 110-130°C before use. This buffer solution is quite stable when protected from undue exposure to the atmosphere.

4.2.3 *Potassium hydrogen tartrate buffer*, pH 3.56 at 25°C. Add 1 g of KHC₄H₄O₆ to 100 mL water at about 30°C, shake for several minutes, cool to 25°C, decant, and filter to secure an approximately saturated solution.

NOTE 5: The pH is insensitive to changes of concentration, so elaborate precautions to ensure saturation are unnecessary. The temperature of saturation may vary from 22 - 28°C. Excess solid should be removed as it may cause erratic potentials at the liquid junctions. The tartrate solution is subject to mold, accompanied by an increase in pH. Consequently, it cannot be used for more than a few days unless a preservative, such as a crystal of thymol, is added.

4.2.4 *Borax buffer*, pH 9.18 at 25°C. Dissolve 3.81 g of Na₂B₄O₇ • 10H₂O in carbon dioxide-free water and dilute to 1000 mL. The Na₂B₄O₇ need not be dried before use, but should be kept tightly sealed to prevent moisture or CO₂ uptake before use.

NOTE 6: The concentration of this solution is so low that changes in the water content of the borax will not affect the pH of the solution; however, exposure to atmospheric carbon dioxide may change its value. It is advisable to replace the solution about a month after preparation.

- 4.2.5 *Commercial buffers at 4, 7, and 10 pH.* These are permitted provided they are of the proper pH value.
- 4.3 *Potassium chloride solution.* Prepare a 0.01N solution using reagent grade KCl which has been dried for 2 h at 105°C. Cool in a desiccator and dissolve 0.7455 g in reagent water and make up to 1000 mL in a volumetric flask at 20°C.