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Starch in paper

1. Scope

1.1 This method describes the qualitative and the quantitative determination of unmodified starches and starches modified only by conventional oxidation techniques or enzyme conversion, which are used for beater addition or surface sizing.

1.2 This procedure measures the total starch content and does not differentiate between starch inside and on the surfaces of the paper.

1.3 Some starches that are cationic, substituted, grafted, or combined with resins are not within the scope of this procedure, because special techniques are required for them. Test to determine whether the starch is appropriate for this procedure by checking for extractability. If it can be extracted quantitatively and forms the iodine complex, it should be able to be determined by this method.

1.4 This method is applicable only for papers with fully bleached pulp, because lignin and lignin remnants can also react with iodine to form a color complex.

2. Significance

The amount of actual starch content is a measure of the starch sizing content added to the paper. It can also be used as a measure of wet end starch retention in the papermaking process.

3. Qualitative

3.1 Apparatus

3.1.1 *Glassware and other equipment:* beakers, 50 and 150 mL; graduated cylinder, 100 mL; small filter funnel; filter paper.

3.2 Reagent

3.2.1 *Iodine solution*, about 0.001N I₂. Make a 0.01N stock solution of iodine by dissolving 0.13 g I₂ in a

solution of 2.6 g KI in 5 mL of water and diluting to 100 mL. Dilute one part of this with nine parts of water, to a pale yellow color, each time a test for starch is made.

3.3 *Test specimen.* From each test unit of a sample obtained in accordance with TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product," weigh a representative specimen of approximately 0.5 g and cut into pieces 5 to 10 mm square.

3.4 *Procedure.* Boil the paper specimen for several minutes with 10 mL of water. Filter, cool the filtrate, and add one drop of the dilute I₂ solution. A blue coloration indicates starch. A faint violet coloration should be disregarded, as nonstarch constituents of paper sometime give this reaction.

4. Quantitative

4.1 Apparatus

4.1.1 *Disintegrator*, for disintegrating the specimen in water. A domestic high-speed electric mixer is preferable, but any other means, e.g., glass beads and bottle, may be used.

4.1.2 *Fritted glass filter funnels*, coarse, about 50 mL or larger, or Büchner funnels and general purpose ashless filter paper with medium speed and retention, other equivalent acid washed filter paper, or glass microfiber filters. When using fritted glass filters, clean by passing 5N NaOH through the filter and washing once with hot water. If this does not remove all starch residue, heat the frit in a 480°C furnace in an oxidizing atmosphere.

4.1.3 *Filtration flask*, 500 mL or larger.

4.1.4 *Centrifuge*, preferably with a capacity of over 50 mL.

4.1.5 *Spectrophotometer*, for absorption measurements at 580 nm. Either a double beam spectrophotometer or a filter comparator may be used. In either case, use the same instrument and conditions for a calibration curve as for the specimen.

4.1.6 *Other equipment*, 50, 100, and 500 mL volumetric flasks; 25 and 2.5 mL pipets; 250 mL beaker; boiling water or steam bath, stopwatch.

4.1.7 *Hood*, for use in carrying out extractions with HCl. A standard laboratory fume hood is adequate to protect personnel and laboratory environment from the effects of HCl fumes.

4.1.8 **Glassware for this procedure must be scrupulously clean. The laboratory can verify this by running a blank with cotton linters through the entire section 4.4 procedure. If residual starch is found, the laboratory should modify its glassware cleaning procedures. For example, after cleaning glassware can be heated in a muffle furnace for 1-2 hours at 425°C to destroy any remaining traces of starch.**

4.2 Reagents

4.2.1 *Hydrochloric acid*, concentrated HCl, also solutions diluted 1 + 1 (approximately 6N), 1 + 9 (approx. 1.2N) and 1 + 19 (approx. 0.6N).

4.2.2 *Potassium iodide-iodine reagent*, 7.5 g KI and 5 g I₂ per liter. Dissolve 7.5 g KI in 10 mL of water, add 5 g I₂. Dilute to 1 L after iodine is dissolved.

4.2.3 *Cotton linters*.

4.2.4 *Reagent grade deionized water*, meeting ASTM D1193, Type II specifications.

4.3 *Test specimens.* From each test unit of a sample obtained in accordance with TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product," weigh to the nearest 5 mg, two 1-g representative specimens consisting of small (5 to 10 mm) strips. Do not dry-grind the paper, since it may lose some of its starch, especially if it contains a filler. At the same time, unless the moisture content is known to within 1%, weigh another 1.0-g specimen for determining the moisture in accordance with TAPPI T 412 "Moisture in Paper and Paperboard."

4.4 Procedure

4.4.1 Transfer the first specimen to the disintegrator, disintegrate in 60 ± 20 mL of deionized warm water (40-80°C) and quantitatively transfer to a 250-mL beaker, using enough rinsing warm water to make the specimen up to 100 mL. If the sample seems to lack sufficient body to form a substantial pad, add some cotton linters (less than 1 g). Heat on the bath to just below the boiling point for 15 minutes after temperature in the beaker reaches 92°C.

4.4.2 Transfer the contents of the beaker to the funnel on the filtration flask. Drain and wash the residue with 10-12 mL of hot deionized water. Remove suction from the flask. Cool to room temperature in an ice bath. Extract the pad with HCl, closely controlling the extraction times to prevent losses of starch by hydrolysis. For the first HCl extraction, add 25 mL of the (1 + 1) HCl to the filter crucible, allow to stand for 3 minutes (preferably stir with a glass rod to break up the pad) and apply the suction. Remove suction from the flask. Repeat the 3 minute extraction with 25