WI 220802.08

T 434

BALLOT NO. 03 SARG

DRAFT NO. 02

DATE 6/1/2023

WORKING GROUP CHAIR To Be Determined

SUBJECT CATEGORY Chemical Properties

RELATED METHODS See "Additional Information"

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 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.

Acid-soluble Iron in Paper

(Proposed Reconfirmation of Classical Method T 434 cm-10)
(Draft 2, no changes from Draft 1)

1. Scope

Acid-soluble iron is considered that portion of the iron present which is potentially chemically reactive, as distinguished from the insoluble or "fixed" iron which might occur as a silicate or other complex compound in clay filler. This procedure for acid-soluble iron is preferable to that of extracting the ash from the paper with acid, because it requires less time and avoids change in the nature of iron compound which may result from ashing. The method is applicable to paper or paperboard containing approximately 10-200 ppm acid-soluble iron.

2. Apparatus

2.1 *Pipets,* 10, 25, and 50 mL.

2.2 *Volumetric flasks*, 50 and 1000 mL.

2.3 *Graduated cylinders*, 5, 10, and 1000 mL.

2.4 *Beakers*, 100, 150, and 600 mL.

2.5 *Funnel,* Büchner, 7.5 cm diameter.

2.6 *Filter funnel*, glass, 65 mm.

2.7 *Nessler tubes*, 50 mL.

2.8 *Spectrophotometer*, or photometer with 508 nm filter.

2.9 *Filter paper*, 11 cm diameter paper is characterized as an ashless, acid washed paper with medium porosity, medium flow rate, and a particle retention size of 8 μm for use in gravimetric analysis, and the 7-cm diameter paper is characterized as an ashless, acid washed paper with fine porosity, slow flow rate, and a particle retention size of 2.5 μm for use in critical gravimetric analysis.

2.10 *Other apparatus*: pH meter (or congo red paper); stirring rods.

3. Reagents

3.1 *Nitric acid*, concentrated HNO_3.

3.2 *Hydrochloric acid*, concentrated HCl and dilute (1:3).

3.3 *Ammonium hydroxide*, concentrated NH_4OH and dilute (1:4).

3.4 *o-Phenanthroline solution.* Dissolve 0.25 g of *o*-phenanthroline in 100 mL of water.

3.5 *Hydroxylamine hydrochloride solution.* Dissolve 10 g of hydroxylamine hydrochloride in water and dilute to 100 mL.

3.6 *Standard iron solution*. Dissolve 14.0 g of pure ferrous ammonium sulfate, $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$, in 500 mL of water containing 20 mL of concentrated H_2SO_4. Dilute to exactly 1000 mL and mix well. Pipet 25 mL of this solution into a 1000-mL volumetric flask, dilute to the mark, and mix. One milliliter of this solution now contains 50 μg of iron, which for 1 g of paper is equivalent to 50 parts of iron per million.

4. Test specimens

Sample the paper in accordance with TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, or Related Product." From each test unit take a representative portion of at least 20 g for duplicate test specimens. Cut or tear each specimen into small squares (about 15 mm on a side).

NOTE 1: The grinding of very hard, resistant paper may be necessary, but avoid grinding in an iron mill because of contamination.

5. Procedure

5.1 If not known, determine moisture content of the paper in accordance with TAPPI T 412 “Moisture in Paper and Paperboard” or TAPPI T 208 “Moisture in Wood, Pulp, and Paperboard by Toluene Distillation,” (withdrawn 1998) under conditions prevailing at the time the specimens are weighed.

5.2 Weigh a specimen of about 5 g of paper to the nearest 0.005 g and transfer it to a 100-mL beaker. Add about 50 mL of hot concentrated HCl (60-80°C) and macerate the paper for 5 min with a glass rod. Filter through an acid-washed 7-cm-diameter paper on the Büchner funnel. Wash the residue with three 50-mL portions of hot water, applying suction after each addition. Add a few drops of concentrated HNO_3 to the combined filtrates, make ammoniacal with concentrated NH_4OH, and boil until the odor of NH_3 is nearly gone.

NOTE 2: If all NH_3 is evaporated, some precipitate may redissolve.

5.3 Filter through an 11-cm-diameter filter paper in the glass filter funnel and wash the residue on the paper thoroughly with hot water. Place the funnel containing the filter paper and contents-$Fe(OH)_3$-the neck of a 50-mL volumetric flask. Add 5 mL of dilute HCl to the funnel. After the acid has passed through the funnel, wash the paper with about 10 mL of water. Add a second 5-mL portion of dilute HCI to the funnel and again wash with water. Dilute the filtrate to the mark and mix.

5.4 Place an aliquot of the solution (10 or 25 mL depending on the estimated iron content of the paper) in a 100-mL beaker. Add 3 mL of the hydroxylamine hydrochloride solution and mix. Add 3 mL of *o*-phenanthroline reagent and adjust the pH to 3-4 with dilute ammonium hydroxide (1:4). A pH meter is preferable for this adjustment, but congo red paper may be used instead. (The pH must be controlled within the specified limits to prevent interference of copper.) Transfer the colored solution to a 50-mL volumetric flask and dilute to the mark with water. Mix and let stand for 30 min. Measure the absorbance of the solution at 508 nm against a reagent blank that is prepared by the same procedure as the specimen. Either a spectrophotometer or a filter photometer may be used to determine the absorbance.

NOTE 3: Alternatively, for possibly greater accuracy at comparatively low iron concentrations, color comparison may be made in Nessler tubes, by diluting an aliquot of the specimen solution in a 50-mL Nessler tube, and comparing with standards made in the same way with the standard iron solution. Make a blank determination with the reagents and apply a correction if necessary.

NOTE 4: When using a spectrophotometer or filter photometer, obtain a calibration curve by plotting the absorbance readings versus the amount of the iron on linear graph paper. (For instruments using cells with a l-cm light path, use amounts ranging from 0 to 250 μg of the standard iron solution.) Pipet aliquots of these standard iron solutions into 100-mL beakers and also prepare a blank omitting the iron solution, then proceed as above, starting with “Add 3 mL of hydroxylamine hydrochloride solution...”

6. Calculation

$$\text{Iron, ppm} = \frac{\text{Iron, } \mu g \times 50}{\text{g specimen} \times \text{mL of aliquot}}$$

7. Report

Report the average result from each test unit, in parts by weight, by acid-soluble iron per million parts of moisture-free paper.

8. Precision

Duplicate determinations may be expected to agree within 5%, when performed in the same laboratory by the same operator.

9. Keywords

Paper, Iron, Acid solubles, Paperboard

10. Additional information

10.1 Effective date of issue: To Be Assigned.

10.2 This method, formerly T 434 os-68, has been reclassified as a Classical Method. Such procedures are no longer in common use or have been superceded by advanced technology; they are technically sound, have a history of use, and contain a body of literature references that make their preservation valuable.

10.3 For a list of references for this method, *see Tappi* **50** (9): 206A (1967).

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■