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T _____ 245 _____

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WORKING GROUP

CHAIRMAN _____ Bogdan Marian Tofanica _____

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 Material 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.

**Silicates and silica in pulp (wet ash method)
(Reconfirmation of T 245 cm-07 as a Classical Method)
(no changes from previous draft)**

1. Scope and summary

1.1 This method describes a procedure for determining the silicates and silica content of pulp.

1.2 The pulp is wet-ashed using nitric and sulfuric acids. The insoluble residue is filtered, washed, ignited, and weighed as silicon dioxide.

1.3 For a faster but less accurate procedure for measuring all acid insoluble material see TAPPI T 244 "Acid-Insoluble Ash in Pulp."

1.4 The ashing temperature used in this method has been changed from 575°C (T 245 om-88) to 525°C. This is consistent with recent changes in TAPPI T 211 "Ash in Wood and Pulp," and is also consistent with current ASTM and ISO ash methods. The purpose behind this is to prevent the degradation of calcium carbonate that can occur at the higher temperature. Please note that the precision statement contained in section 10 was developed using an ashing step at 575°C, and has not been repeated at the lower ashing method of 525°C.

2. Significance

Silica or silicates are normally present in pulp in very small quantities. If excessive amounts are present and carried into the finished paper of some “fine” or specialty grades, they may cause undesirable abrasion of metal repeatedly applied to the paper, such as punches, dies, knives, or type.

3. Apparatus

- 3.1 *Kjeldahl flask*, 500 mL.
- 3.2 *Heating mantle*, sized to accommodate a 500-mL flask and with a variable heat controller.
- 3.3 *Platinum dish*, 50 mL.
- 3.4 *Muffle furnace*, maintained at $525 \pm 25^\circ \text{C}$.
- 3.5 *Filter paper*, ashless, acid-washed, medium porosity filter paper for use with very fine precipitates.

Particle retention should be $>2.5\mu\text{m}$.

- 3.6 *Other apparatus*, graduated cylinders, 10-, 25-, and 100-mL.

4. Reagents

- 4.1 *Nitric acid*, concentrated HNO_3 .

NOTE 1: Fuming HNO_3 has been used in the related method for paper; however, concentrated HNO_3 is suggested as a substitute because of the even more hazardous nature of the fuming acid.

- 4.2 *Sulfuric acid*, concentrated H_2SO_4 .

5. Sample

- 5.1 Select a sample of pulp representative of the shipment or batch to be tested and tear it into pieces.
- 5.2 If the moisture content is not known, determine the moisture content of another sample in accordance with TAPPI T 210 “Sampling and Testing Wood Pulp Shipments for Moisture.” Use percent moisture to determine the oven dry weight of the sample, expressed in grams.

6. Test specimen

The size of the specimen, approximately 25 g, depends on its silica content and should be adjusted so that the weight of the silica will be at least 0.01 g. Weigh duplicate specimens to the nearest 0.01g.

7. Procedure

CAUTION: 1) Because of the corrosive fumes, carry out the digestion in a well-ventilated hood.
2) Because of the danger of adding water to concentrated acid, perform steps 7.2 and 7.3 while wearing chemical goggles, face shield, long heavy rubber gloves, and use a splash shield in front of the flask.

7.1 Place the weighed specimen in a clean 500-mL Kjeldahl flask. Add 100 mL of concentrated HNO₃, warm until the pulp becomes a thin paste, then boil gently until a low-viscosity solution results. Evaporate the solution to a volume of 40-50 mL, cool, and add 20 mL of concentrated H₂SO₄.

7.2 Heat until white fumes of SO₃ are evolved. Clarify the solution by adding, with extreme caution and without cooling the flask, small volumes (10-20 mL) of concentrated HNO₃, allowing the acid to run slowly down the inside wall of the flask. Reheat the flask after each addition until white SO₃ fumes are again evolved. Repeat this operation as often as necessary to produce a clear, straw-colored solution, which does not darken on further heating. Complete the wet oxidation by heating until SO₃ fumes reappear. This will remove the last traces of HNO₃ from the solution.

7.3 Cool the solution to room temperature and carefully dilute to approximately 250 mL with distilled water using a squirt bottle for the distilled water. Boil gently for a few minutes. Filter through the recommended filter paper, being careful to quantitatively transfer the solids. Wash the residue with a few small portions of boiling distilled water (normal use calls for 100-125 mL total).

NOTE 2: If trace metals (Cu, Fe, Mn, or Ca) are to be determined in the same pulp specimen, collect the filtrate in a volumetric flask for further treatment.

7.4 Place the filter paper containing the residue in a platinum dish, previously ignited to a constant weight (to the nearest 0.1 mg). Heat the dish very carefully until the water has evaporated and then ignite the paper and residue in the muffle furnace at $525 \pm 25^{\circ}\text{C}$ until free from black carbon particles. Allow the dish to cool in a desiccator and reweigh it.

8. Calculation

Calculate the silica content as:

$$X = 1000 a/m$$

where

X = silicates and silica in parts per million (mg/kg) as SiO_2 .

a = weight of insoluble residue, mg.

m = weight of the oven-dry pulp, g.

9. Report

Report the average result for the silicates and silica in pulp in ppm of SiO_2 , to the nearest 10 ppm (mg/kg).

10. Precision

NOTE 3: This precision statement was generated using the higher ashing temperature of 575°C and has not been redone at the current ashing temperature of 525°C.

10.1 Repeatability (within a laboratory) = 20 ppm.

10.1.1 The repeatability value is based on an intralaboratory study conducted in accordance with TAPPI T 1200 "Interlaboratory Evaluation of Test Methods Used with Paper and Board Products," using pulp samples containing these levels of silica (ppm): 10, 100, 200, 300, etc., up to 2000.

10.2 Reproducibility (between laboratories) = not known; in accordance with the definitions of these terms in TAPPI T 1206 "Precision Statement for Test Methods."

11. Keywords

Silica, Silicates, Pulp, Impurities, Wet ash method

12. Additional information

12.1 Effective date of issue: to be assigned.

12.2 This method has been revised to include an ashing step at 525°C. Please see Scope and Precision statements that discuss this change.

12.3 For pulp samples containing minute amounts of silica (less than 10 ppm), ASTM Standard Method D 2438 “Silica in Cellulose” should be used.

12.4 Related methods: SCAN-C9 (Silicates and Silica in Pulp); ANSI P3.17; ASTM D 2438.

12.5 This method was reclassified as a Classical method in 1998. This 2007 version contains no revisions other than making generic the description of the filter paper.

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