WI 220808.06

T 244

DRAFT NO. 03 SARG

DATE October 26, 2023

WORKING GROUP CHAIR N/A

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-insoluble ash in wood, pulp, paper, and paperboard

(Proposed Reconfirmation of Classical Method T 244 cm-11)

1. Scope and summary

1.1 This method describes a procedure for determining the acid-insoluble ash content of cellulose samples.

1.2 The sample is dry-ashed and the residue treated with hydrochloric acid. The insoluble residue is filtered, washed, ignited, and weighed.

1.3 This method measures all acid insoluble material. It is a faster and less accurate method than other referenced methods. For a more accurate but time-consuming wet-ash procedure for determining the silicates and silica content of pulp, see TAPPI T 245 "Silicates and Silica in Pulp (Wet Ash Method)."

2. Significance

Silica or silicates, the major components of the acid-insoluble ash, are normally present in wood and pulp in very small quantities. If excessive amounts are present and carried into the finished paper, particularly of "fine" or specialty grades, they may cause undesirable abrasion of metal repeatedly applied to the paper, such as punches, dies, knives, or type. Paper and paperboard may be filled or coated with clay (alumino-silicates) making the acid-insoluble ash content considerably higher.

3. Apparatus[1]

3.1 *Platinum crucible*, or dish, 50 mL.

3.2 *Muffle furnace,* maintained at 525 ± 25°C.

3.3 *Filter paper*, ash-free, double acid-washed, higher retention, of a type recommended for use with the finest precipitates.

4. Reagents

Hydrochloric acid, 6*M*. Carefully add one volume concentrated HCl to one volume distilled water. Use caution in making and handling of this reagent.

5. Sampling

5.1 Sampling wood: Obtain, grind, and screen a sample of wood in accordance with TAPPI T 257 "Sampling and Preparing Wood for Analysis" and TAPPI T 264 "Preparation of Wood for Chemical Analysis" or a sample of pulp in accordance with TAPPI T 210 "Weighing, Sampling, and Testing Pulp for Moisture." Do not take cut edges or other parts of pulp where mineral contamination may have occurred.

5.2 Sampling pulp: Obtain a sample of pulp in accordance with TAPPI T 210 "Weighing, Sampling, and Testing Pulp for Moisture." Do not take cut edges or other parts of pulp where mineral contamination may have occurred.

5.3 Sampling paper or paperboard: Obtain a sample of paper or paperboard in accordance with TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product."

6. Test specimen

[1] Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list, available as part of the CD or printed set of Standards, or on the TAPPI website general Standards page..

The size of the specimen, approximately 25 g, depends on the ash content and should be adjusted so that the weight of the acid-insoluble ash will be at least 10 mg. Tear pulp, paper, and paperboard samples into small pieces. For wood, use approximately 25 g of the ground and screened material prepared in 5.1. Weigh duplicate specimens to the nearest 0.01g.

7. Procedure

7.1 As directed in TAPPI T 211 "Ash in Wood and Pulp, Paper, and Paperboard: Combustion at 525°C," determine moisture level in the sample and ash the pulp in portions in a platinum dish, previously ignited in the muffle furnace to a constant weight (to the nearest 0.1 mg). Ignite the sample at 525°C in the muffle furnace until the residue is free from black carbon particles.

NOTE 1: This method may also be applied to the ash resulting when T 413 (ashing at 900°C) is utilized, in which case this must be noted in the report.

Cool the dish to room temperature, add 5 mL 6*M* HCl, and evaporate carefully to dryness on a steam bath. Add a second 5-mL portion of 6*M* HCl and again evaporate to dryness. Add a third 5-mL portion of 6*M* HCl to the residue, heat on the steam bath, and dilute with 20 mL of distilled water.

7.2 Filter the solution through the ash-free filter paper, being careful to remove all the residue from the dish by means of a rubber policeman, and wash the residue several times with hot distilled water until the filtrate is free from chloride ions (check with silver nitrate solution).

NOTE 2: The filtrate resulting when this method is used on pulp or paper and paperboard may be utilized for the determination of trace metals such as Cu, Fe, Mn, and Ca where their determination on an ashed sample is required. In such cases, collect the filtrate in a volumetric of suitable size (50 ml in most cases) and follow the analysis method of the trace metal procedure being used. Such procedure should not be followed when using this method for wood, as the grinding and screening procedures (see 5.1) may contaminate the sample with trace amounts of metals.

7.3 Place the filter paper containing the insoluble residue in the platinum dish. Heat very carefully until the water has evaporated, and then ignite the residue in the muffle furnace at 525ºC until free from carbon. Allow the dish to cool in a desiccator and weigh to the nearest 0.1 mg.

8. Calculation

Calculate the acid-insoluble ash content as:

$$\text{Acid-insoluble ash, ppm} = \frac{a \times 1000}{m}$$

where

a = weight of insoluble residue, mg
m = weight of the oven-dry sample, g

9. Report

Report the average result for the acid-insoluble ash as parts per million (milligrams per kilogram), to the nearest 5 parts per million.

10. Precision

10.1 Repeatability (within a laboratory) = 70 ppm

10.2 Reproducibility (between laboratories) = not known

10.3 These terms are in accordance with the definitions in TAPPI T 1200 “Interlaboratory evaluation of test methods to determine TAPPI repeatability and reproducibility.”

11. Keywords

Wood, Pulp, Paper, Paperboard, Ash content, Acid insolubles, Silicates, Silica, Impurities, Ash

12. Additional information

12.1 Effective date of issue: To be assigned.

12.2 Related methods: ISO Standard 776; BS 4496.

12.3 This method was made classical by committee action in 1999. This method was revised and reconfirmed in 2011 to make editorial corrections for better clarity.

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