

WI _____ 060802.18 _____

T _____ 211 _____

DRAFT NO. _____ 2 _____

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TAPPI

WORKING GROUP

CHAIRMAN _____ N/A; method reaffirmed _____

SUBJECT

CATEGORY _____ Chemical Properties _____

RELATED

METHODS _____ See "Additional Information" _____

CAUTION:

This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. 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 method, the user must 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.

**Ash in wood, pulp, paper and paperboard:
combustion at 525°C
(Five-year review of T 211 om-02)
(No changes since Draft 1)**

1. Scope

This method for determination of ash can be applied to all types and grades of wood pulp paper, and paper products. For the determination of ash by combustion at 900°C, see TAPPI T 413 "Ash in Wood, Pulp, Paper and Paperboard: Combustion at 900°C."

2. Summary

A test specimen is ignited in a muffle furnace at 525°C. A separate test specimen is analyzed for the percentage moisture. The resulting weight of ash and moisture level in the sample are used to calculate the percentage ash present at 525°C on a moisture-free sample basis.

3. Significance

3.1 The ash content of the sample may consist of: (1) various residues from chemicals used in its manufacture, (2) metallic matter from piping and machinery, (3) mineral matter in the pulp from which the paper was made, and (4) filling, coating, pigmenting and/or other added materials. The amount and composition of the ash is a function of the presence or absence of any of these materials or others singly or in combination. No specific *qualitative* meaning is attached to the term “ash” as used in this test method. Where a further qualitative examination of the ash is desired, this method may be used in combination with TAPPI T 421 “Qualitative (Including Optical Microscopic) Analysis of Mineral Filler and Mineral Coating of Paper,” and major components of the ash identified.

3.2 The combustion of cellulose to form volatile combustion products occurs at about 300°C. For papers or pulp containing no added fillers or coatings, ignition at **either** 525°C or 900°C will yield essentially identical results of a few tenths percent ash or less. Examples of such papers include “ashless” filter papers manufactured for chemical analysis, or dissolving grade pulps.

3.3 For samples containing fillers, coatings or pigments *which undergo negligible change in weight upon ignition of either 525 °C or 900 °C*, such as the oxides of silicon or titanium, and where other fillers, coatings or pigments are known to be absent, ignition at either temperature may be taken as a semi-quantitative measure of the percentage of such material present in the sample.

3.4 In most cases, the ash content of paper and paperboard will contain inorganic residues from the pulp, inorganic residues from paper making chemicals, and loading or filling materials deliberately added. In such cases, the significance of the ash level determined will vary depending upon which ashing temperature is used and the identity of the materials added.

3.5 For papers containing only cellulose and calcium carbonate, ignition at 525°C will remove cellulose, and moisture, but will leave as ash the calcium carbonate essentially intact. Ignition at 900°C will convert the calcium carbonate to calcium oxide. In such cases, these methods may be used in conjunction to provide a good estimate of added calcium carbonate levels.

3.6 For papers containing cellulose and clays or materials of indefinite composition and/or variable thermal decomposition, significance of ash level may require significant confirmation regarding the materials added, qualitative analysis of the ash as described in T 421, or both, and even then care in determining data significance will be required.

3.7 The user of this test method must confirm that 525°C is the correct ashing method to use and the significance of results based on an understanding of the composition of the sample ash and the information desired. This method may be used in conjunction with TAPPI T 413 “Ash in Wood, Pulp, Paper and Paperboard: Combustion at 900°C” for a more comprehensive understanding of the non-cellulosic materials present in the sample.

4. Definitions

4.1 *Ash*, the material remaining, calculated on the basis of the dry weight of the original sample, after the sample is ignited at a specified temperature.

4.1.1 *Ash at 525 °C*, the ash content of the sample when the ignition temperature is 525°C.

5. Apparatus¹

5.1 *Crucibles*, platinum, porcelain or silica, 50 to 100 mL, with covers.

5.2 *Electric muffle furnace*, suitable for maintaining a temperature of 525 ± 25°C. Control of temperature and its accuracy are critical for this method.

6. Sampling

6.1 Obtain 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.

6.2 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.”

7. Test Specimens

7.1 Condition the sample in the atmosphere near the balance to the moisture equilibrium.

7.2 The test specimen shall consist of small pieces of the sample so selected as to be representative. The specimen weight shall be at least 1 g moisture-free and may be increased to sufficient size to yield an ash weight of not less than 10 mg and preferably over 20 mg.

7.3 Prepare sufficient test specimens so that the Procedure, Section 9.1 may be performed at least twice for each test unit.

7.4 The test specimen shall be weighed on an analytical balance to the nearest 0.1 mg.

¹Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list in the bound set of TAPPI Test Methods, or may be available from the TAPPI Quality and Standards Department.

8. Determination of moisture

8.1 At the same time as the test specimen is being weighed (Section 7), weigh the sample for determination of moisture content in accordance with T 264 "Preparation of Mechanical Pulps for Testing" (wood), T 210 (pulp) or T 550 "Determination of Equilibrium Moisture in Paper and Paperboard for Chemical Analysis" (paper and paperboard).

9. Procedure

9.1 *Moisture determination.* Continue and complete moisture determination as described in the relevant procedure (see 8.1).

9.2 *Ash determination*

9.2.1 Carefully clean the empty crucible and ignite in a muffle furnace at $525 \pm 25^\circ\text{C}$ for 30-60 minutes. After ignition, cool slightly and then place in a desiccator, containing indicating-grade anhydrous alumina. When cooled to room temperature, weigh the ignited crucible on the analytical balance to the nearest 0.1 mg.

9.2.2 Transfer the test specimen to the crucible - and, with the lid ajar, gently carbonize the specimen in the crucible on the hearth of the furnace or directly over a low flame of a bunsen burner. Alternately, place the crucible, with lid removed, in a furnace at about 100°C . Raise the temperature to 525°C slowly so that the sample becomes carbonized without flaming. Sample must be charred, not burned so that the temperature of the sample does not exceed 525°C . If the crucible is too small to hold the entire specimen, gently char the portion added and add more as the sample chars, but in either case protect the contents of the crucible with a lid or cover so that a portion of the ash will not be blown from the crucible and/or the sample will not burn. When the residue has ceased to char, place the crucible with specimen into the furnace at $525 \pm 25^\circ\text{C}$ and remove the lid after the crucible seems to have reached the temperature of the furnace.

9.2.3 When the specimen is completely combusted as indicated by the absence of black particles, remove the crucible from the furnace, replace the cover, and allow to cool somewhat; then place in a desiccator containing indicating grade anhydrous alumina and cool to room temperature. Weigh the crucible with ash to the nearest 0.1 mg. Repeat the ignition and weighing until the weight of the ash is constant to ± 0.2 mg.

10. Calculation

Calculate the ash content as follows:

$$\text{Ash, \%} = \frac{A \times 100}{B}$$

where

A = weight of ash, g

B = weight of test specimen, g moisture-free

11. Report

Report the ash content in the sample as an average of two determinations to the nearest 0.01% when the ash content is below 10%, or to the nearest 0.1% if the ash content is 10% and higher.

12. Precision

12.1 The following estimates of repeatability and reproducibility are based on an interlaboratory trial conducted in 2002 involving 8 laboratories. This data is based on 6 materials including, two grades of wood chips, two grades of wood pulp, one grade of uncoated bond, and one grade of coated bond. The precision estimates are based on two determinations per test result and three test results per lab, per material. A more detailed chart of example results is included below:

Wood Chips

Repeatability (within a lab) = 0.18 (% ash)

Reproducibility (between laboratories) = 0.32 (% ash)

Wood Pulp

Repeatability (within a lab) = 0.06 (% ash)

Reproducibility (between laboratories) = 0.09 (% ash)

Uncoated Writing Papers

Repeatability (within a lab) = 0.44 (% ash)

Reproducibility (between laboratories) = 0.53 (% ash)

Coated Writing Papers

Repeatability (within a lab) = 1.49 (% ash)

Reproducibility (between laboratories) = 2.99 (% ash)

Repeatability and reproducibility are estimates of the maximum difference (at 95%) which should be expected when comparing test results for materials similar to those described above under similar test conditions. These estimates may not be valid for different materials or testing conditions.

Data Table of Ash Content Results

Material	Grand Mean	Std Dev Btwn Labs	Repeatability		Reproducibility		Labs Included
			r	%r	R	% R	
Hardwood Chips	0.465	0.125	0.22	48%	0.39	84%	7
Softwood Chips	0.366	0.079	0.15	40%	0.25	68%	7
Softwood Pulp #2	0.165	0.036	0.06	34%	0.11	66%	8
Softwood Pulp	0.168	0.019	0.07	44%	0.08	48%	8
Uncoated Bond	9.69	0.14	0.44	4.5%	0.53	5.4%	8
Coated Bond	31.83	0.98	1.49	4.7%	2.99	9.4%	8

13. Keywords

Ash, Wood, Pulp, Paper, Paperboard, Combustion products, Combustion

14. Additional Information

14.1 Effective date of issue: to be determined.

14.2 This revision reduces the ashing temperature to 525°C from 575°C to make the method more broadly applicable to wood, pulp, paper and related products. The temperature of 575°C causes partial degradation of carbonates when present. This problem is eliminated by the lower combustion temperature of 525°C.

14.3 A procedure for combustion at 900°C is found in TAPPI T 413. Results by the two methods may differ, based on the loading materials present (see Section 3).

14.4 Methods for combustion at temperatures other than of T 211 (525°C) or T 413 (900°C) are published. These may give different results based on loading materials present.

14.5 No attempt is made to harmonize these procedures with ISO standards as combustion temperatures for paper and pulp ashing have been the topic of unresolved debate for several years within ISO.

14.6 The ashing temperatures in T 211 and T 413 are identical with those in ASTM D 586 “Standard Test Method for Ash in Paper. Method A: Ash Content Upon Ignition at 525°C and Method B: Ash Content Upon Ignition at 900°C.”

14.7 Related Methods include ASTM D 1102; PAPTAC G.10, BS 4896, AS/NZS 1301.P3, SCAN C6, ISO 1762; BS 3631, PAPTAC G.11, ISO 2144, all of which may incorporate different ashing temperatures whose impacts may be understood by a careful reading of Section 3 (Significance).

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