Ash in wood, pulp, paper and paperboard: combustion at 900°C
(Five-year review of T 413 om-17)

1. Scope

This method for determination of ash can be applied to all types of wood, pulp, paper and paperboard.

2. Summary

2.1 A test specimen is ignited in a muffle furnace at 900°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 900°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,” or by whatever means that is appropriate, 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 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 900°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 211 “Ash in Wood and Pulp, Paper and Board Combustion at 525°C” for a more comprehensive understanding of the non-cellulose 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 the specified temperatures.

4.1.1 *Ash at 900°C*, the ash content of the sample when the ignition temperature is 900°C. (A programmable electric muffle furnace may be used, also.)

5. **Apparatus**

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

5.2 Electric muffle furnace, suitable for maintaining a temperature of 900 ± 25°C.

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.
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 TAPPI T 262 “Preparation of Mechanical Pulps for Testing” (wood), TAPPI T 210 Weighing, Sampling and Testing Pulp for Moisture (pulp), or TAPPI 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 900 ± 25°C for 30-60 minutes. After ignition, cool slightly and then place the crucible 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. Ignite the specimen in the crucible on the hearth of the furnace until well carbonized or directly over a low flame of a Bunsen burner. If the crucible is too small to hold the entire specimen, either gently burn the portion added and add more as the flame subsides (cool before doing to prevent burns) or use a larger crucible (150 or 250 mL), but in either case protect the content of the crucible with a lid or cover so that a portion of the ash will not be blown from the crucible. When the residue has ceased to burn with a flame, place the crucible with specimen into the furnace at 900 ± 25°C and remove the lid after the crucible seems to have reached the temperature of the furnace.

NOTE 1: If a programmable furnace is used, then an appropriate program must be used to ensure the proper combustion is done, such as, a slow ramp up to 300-325°C, hold to do initial combustion, then ramp up to ultimate temperature and hold.

NOTE 2: To avoid the use of an open flame with a Bunsen burner, the following can be done: place the crucible in a cool muffle furnace, bring the temperature to 105°C for 5 – 10 minutes. Then bring the temperature up to 325°C carefully to burn off the sample. Hold for 30 – 60 minutes. Make sure to protect the sample as described above in 9.2.2. Then raise the temperature up to 525°C for an hour and then up to 900°C and continue as described above.

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.
10. Calculation

Calculate the ash content as follows:

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

where

\[
A = \text{ weight of ash, g}
\]

\[
B = \text{ weight of test specimen, g moisture-free}
\]

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 data from interlaboratory study conducted in October 1996. The materials on which this data is based were 2 samples of pulp (hardwood and softwood) 2 samples of uncoated paper (with and without CaCO₃) and 2 samples of coated paper (with and without CaCO₃). Participants were asked to follow ISO Draft International Standard ISO 2144 dated 1995-12-03 which is technically equivalent to TAPPI T 413 at 900°C. Testing is based on 2 determinations per test result and 3 test results per lab, per material. Results for each material are shown below.

**Pulp**

Repeatability (within a lab) = 0.02%

Reproducibility (between laboratories) = 0.30%

**Paper (Coated and Uncoated)**

Repeatability (within a lab) = 0.30%

Reproducibility (between laboratories) = 1.00%

**NOTE 3:** The values above are in % ash by weight (the reporting unit of the test method).

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.
Ash in wood, pulp, paper and paperboard: Combustion at 900°C

<table>
<thead>
<tr>
<th>Sample</th>
<th>Grand Mean</th>
<th>Btwn Lab SSTD</th>
<th>Within Lab SSTD</th>
<th>r</th>
<th>R</th>
<th>Labs</th>
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<tr>
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<thead>
<tr>
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<th>R</th>
<th>Labs</th>
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<tbody>
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<td>0.0831</td>
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<td>0.1518</td>
<td>0.420</td>
<td>1.104</td>
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<td>0.1451</td>
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<td>1.049</td>
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### Keywords


### Additional information

14.1 Effective date of issue: To be assigned

14.2 A procedure for combustion at 525°C is found in TAPPI T 211 “Ash in Wood, Pulp, Paper and Paperboard: Combustion at 525°C.” Results by the two methods may differ, based on the loading materials present.

14.3 Methods for combustion at temperatures other than that of TAPPI T 211 or T 413 (900°C) are published. These may give different results, based on loading material present.

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


14.6 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).

14.7 This method was revised in 2017 to delete repeated ignition to constant weight because some constituents of the ash may lose mass over time with repeated ignition.

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