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WORKING GROUP  
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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.

**Hot water extractable acidity or alkalinity of paper**  
***(Five-year review of Official Method T 428 om-13)***  
***(revisions from Draft 1 incorporated)***  
**(No changes from Draft 2)**

**1. Scope**

1.1 This method, based on the work of Kohler and Hall (*J*), measures the titratable acidity or alkalinity (end point at pH 7.0) of an aqueous extract of paper (filtered and extracted by boiling water for 1 h). It specifies one extraction and so does not measure the total acidity or alkalinity of paper, for which exhaustive extraction is required.

1.2 It may be applied to writing, printing, and sized industrial paper but is not intended for testing electrical insulating papers.

**NOTE 1:** This method measures the pH of the bulk extract as prepared in the method, and as such, measures the combined acidity and alkalinity of the base paper and any added fillers or coating materials.

## 2. Summary

This method consists of a boiling water extraction of the specimen followed by an alkalinity-acidity titration of the extract solution.

## 3. Significance

The pH determination measures the extent to which the paper alters the hydrogen-hydroxyl ion equilibrium of pure water, and the acidity-alkalinity determination measures the quantity of extracted ionic material that contributes to that equilibrium change. These tests are useful for routine acceptance testing, research work, or the evaluation and classification of different papers.

## 4. Definitions

4.1 *Paper acidity*, the extent to which water-soluble materials in the paper alter the hydrogen-hydroxyl ion equilibrium of pure water causing an excess of hydrogen ions as measured by titrating with a standardized solution of alkali under specified conditions.

4.2 *Paper alkalinity*, the extent to which water-soluble materials in the paper alter the hydrogen-hydroxyl ion equilibrium of pure water causing an excess of hydroxyl ions as measured by titrating with a standardized solution of acid under specified conditions.

## 5. Apparatus

5.1 *pH meter*, a pH Meter of Type II, III, or IV as described in Table 2, Laboratory pH Meter, ASTM Specification D 1293, Standard Test Method for pH of Water is satisfactory. The pH meter shall be calibrated and used in accordance with ASTM Specification E 70, Standard Test Method for Determining pH of Aqueous Solutions with the Glass Electrode.

5.2 *Erlenmeyer flask*, 500-mL with a 24/40 standard taper joint.

5.3 *Condenser*, West or Allihn type, with 300-mm jacket; or an air condenser about 12 mm OD and 750 mm long; each with a 24/40 standard taper drip-tip joint.

5.4 *Büchner funnel*, 60-mL, glass, with medium fritted disk.

5.5 *Burets*, two 50-mL.

5.6 *Beaker*, 400-mL.

5.7 *Graduated cylinders*, 100- and 250-mL.

5.8 *Hot plate*, preferably with temperature adjustment.

5.9 *Magnetic stirrer*.

## 6. Reagents

6.1 *Sodium hydroxide*, 0.01N NaOH, accurately standardized (see TAPPI T 610 “Preparation of Indicators and Standard Solutions”).

6.2 *Hydrochloric or sulfuric acid*, 0.01N HCl or H<sub>2</sub>SO<sub>4</sub>, accurately standardized (see TAPPI T 610).

6.3 *Distilled or deionized water*, the water used in this test method shall conform to Reagent Water, Type II or Type I as described in ASTM Specification D 1193, Standard Specification for Reagent Water.

6.4 *Carbon dioxide-free gas, nitrogen, or purified air*. Air, if used, should be oil-free. Pass the air through a gas washing bottle containing at least 200 mL of 3N H<sub>2</sub>SO<sub>4</sub> and then through a tower or U-tube containing ascarite or soda lime, 120-150 mm long, before connecting to the gas dispensing tube.

## 7. Sampling

Obtain a sample of the paper in accordance with TAPPI T 400 “Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product.”

## 8. Test specimen

From each test unit of the sample, cut two representative portions into pieces approximately 5-10 mm square. Keep the portions separate. Allow them to come to equilibrium with the atmosphere near the balance; then weigh out a 5-g test specimen from each portion, each weighed to the nearest milligram. If the moisture content is not known, immediately weigh another specimen and determine its moisture content as described in TAPPI T 412 “Moisture in Paper and Paperboard.”

## 9. Procedure

9.1 Transfer the two weighed specimens to the 500-mL Erlenmeyer flasks, add 250 mL of boiling water to each, and attach condensers.

9.2 To a third 500-mL Erlenmeyer flask add 250 mL of boiling water. This constitutes the Blank. Carry this Blank through all the remaining steps of the Procedure.

**NOTE 2:** The air condenser may be used only if the temperature of the hot plate can be controlled.

9.3 Place the flask on a hot plate and boil gently, taking care not to exceed the capacity of the condenser. Heat at this temperature for  $60 \pm 5$  min with occasional shaking. At the end of this period, pour the contents of the flask into the Büchner funnel. Wash with at least 25 mL of hot distilled water. Apply suction to complete the filtration and cool the extract rapidly in a rubber-stoppered Erlenmeyer flask.

9.4 As soon as the extract reaches room temperature, transfer it to a 400-mL beaker, and, using a pH meter,

titrate with acid or alkali to a pH of 7.0, while passing nitrogen or CO<sub>2</sub>-free air through the specimen as shown in Fig. 1.

## 10. Calculations

10.1 Calculation of milliequivalents of acid or base consumed per gram.

10.1.1 For each extract (or blank), calculate the milliequivalents of reagent (standard acid or standard base) consumed as follows:

$$\text{milliequivalents of reagent consumed} = V \times N$$

where

$V$  = mL reagent required to reach pH 7.0

$N$  = normality of the reagent used

10.1.2 Correct the milliequivalents of reagent (acid or base) consumed by the sample for the milliequivalents of reagent consumed by the blank.

10.1.2.1 If the sample extract is alkaline and the blank is acidic, or the sample extract is acidic and the blank is alkaline, ADD the milliequivalents of the blank to the milliequivalents of the sample extract. Calculate the milliequivalents of acidity or alkalinity per gram for each sample by dividing the corrected milliequivalents by the sample weight corrected for moisture (Section 8). If the sample extract and the blank are either both acidic or both basic, SUBTRACT the milliequivalents of the blank from the milliequivalents of the sample extract.

10.2 Calculate as % SO<sub>3</sub> or % NaOH present.

10.2.1 Where the calculation in 10.1.1 shows the sample to be acidic (that is the original sample pH was below 7.0) calculate SO<sub>3</sub> present as:

$$\text{SO}_3 \text{ (percent)} = \frac{\text{Milliequivalents of base consumed} \times 0.040}{W} \times 100$$

where  $W$  = sample weight (in grams) corrected for moisture (Section 8)

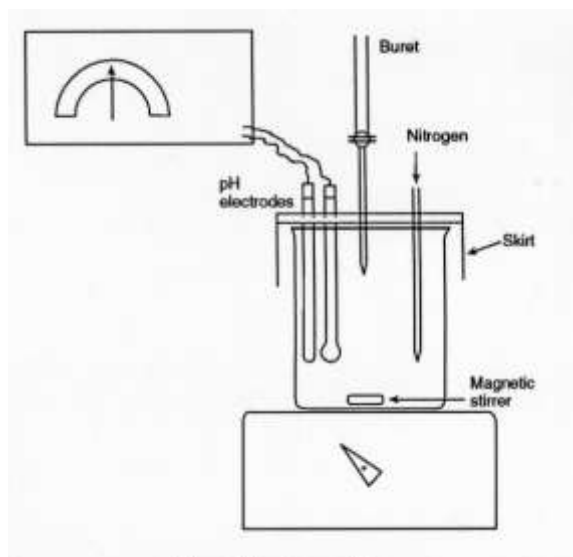
**NOTE 3:** If the sample is *originally acidic* (original pH less than 7) the milliequivalents calculated are those of the *base* used in titration. The milliequivalents of base used (corrected for the blank) are numerically equal to the milliequivalents of acid in the original sample.

If the sample is *originally basic* (original pH greater than 7) the milliequivalents calculated are the *acid* used in titration. The milliequivalents of acid used (corrected for the blank) are numerically equal to the milliequivalents of base in the original sample.

10.2.2 Where the calculation in 10.1.1 shows the sample to be basic (that is the original sample pH was above 7.0) calculate % NaOH as:

$$\text{NaOH (percent)} = \frac{\text{Milliequivalents of acid consumed} \times 0.040}{W} \times 100$$

where  $W$  = sample weight (in grams) corrected for moisture (Section 8)



**Fig. 1.** pH meter assembly.

## 11. Report

11.1 The milliequivalents of acid or base present in the sample, or the % alkalinity as calculated in Section 10 may be reported, as agreed by parties involved in the testing. Report the average value for the two specimens tested.

## 12. Precision

12.1 The following precision statement is prepared in accordance with TAPPI T 1200 “Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility” based on data collected from an interlaboratory trial involving 7 laboratories (see Table 1).

12.2 Repeatability (within a laboratory) is 29.1% tested on an acid sample and 23.7% tested on an alkaline sample.

12.3 Reproducibility (between laboratories) is 46.8% tested on an acid sample and 65.6% tested on an alkaline sample.

**Table 1.** Repeatability and reproducibility.

<i>Sample</i>	<i>Grand Mean (meq/g)</i>	<i>Range (meq/g)</i>	<i>Repeatability</i>		<i>Reproducibility</i>	
			<i>r</i>	<i>r%</i>	<i>R</i>	<i>R%</i>
Bleached paperboard	0.00367 H <sup>+</sup>	0.00313 - 0.00470	0.00107	29.1	0.00172	46.8
Uncoated offset	0.0168 OH <sup>-</sup>	0.0113 - 0.0218	0.0040	23.7	0.0110	65.6

## 13. Keywords

Paper, Acidity, Alkalinity, Extractives

## 14. Additional information

14.1 Effective date of issue: To be assigned.

14.2 A hot extract of paper is usually more acid than a cold extract (2). This is caused by hydrolysis of the aluminum compounds retained in the paper. It has also been found (3) that alkaline products are released by the hydrolysis of melamine-formaldehyde resins during hot-water extraction.

14.4 This method was corrected or revised in 1936, 1939, 1945, 1967, 1977, 1985, 1991, and 1998.

14.5 Related methods: ASTM D548; SCAN P14; TAPPI T 435; PAPTAC G 25.

**Literature cited**

1. Kohler, S., and Hall, G., *Pulp Paper Mag. Canada* **24**(15):423 (1925).
2. Grant, J., *Proc. Tech. Sect.* **36**, *British P & BMA*, 473 (1955).
3. Wilson, W.K., Harvey, J.L., and Padgett, A., *Tappi* **34**(9):410 (1951).

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