Designation: D 548 – 97 (Reapproved 2002)

An American National Standard

Standard Test Method for Water-Soluble Acidity or Alkalinity of Paper[1]

This standard is issued under the fixed designation D 548; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the water-soluble acidity or alkalinity of paper.[2]

1.2 It may be applied to writing, printing, and sized industrial paper but is not intended for testing electrical insulating papers nor those containing alkaline fillers or coatings such as casein or calcium carbonate.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, or Related Product[3]

D 644 Test Method for Moisture Content of Paper and Paperboard by Oven Drying[3]

D 1193 Specification for Reagent Water[4]

D 1293 Test Methods for pH of Water[4]

D 1968 Terminology Relating to Paper and Paper Products[3]

E 70 Test Method of pH of Aqueous Solutions with the Glass Electrode[5]

2.2 *TAPPI Standard:*

T 1206 Precision statement for test methods[6]

3. Terminology

3.1 *Definitions*—Definitions shall be in accordance with Terminology D 1968 and the *Dictionary of Paper*.[6]

4. Summary of Test Method

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

4.2 This test method, based on the work of Kohler and Hall,[2] measures the titratable acidity or alkalinity (end point at 7.0 pH) 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.

5. Significance and Use

5.1 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.

6. Apparatus

6.1 *pH meter*, a commercial pH meter including a glass electrode, reference electrode, and electronic circuitry that will measure electromotive force and indicate pH directly with a precision of 0.01 pH unit (see Fig. 1). A pH meter of Type II, III, or IV as described in Test Methods D 1293 is satisfactory, and shall be calibrated and used in accordance with Specification E 70.

6.2 *Balance*, an analytical balance sensitive to 0.1 mg.

6.3 *Hot Plate*, with temperature regulator.

NOTE 1—A steam or oil bath that can be maintained at 100°C may be substituted, if available.

6.4 *Glassware, Acid- and Alkali-Resistant*:

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[1] This test method is under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and is the direct responsibility of Subcommittee D06.92 on Test Methods.

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[2] This test method is based on the method for acidity described by S. Kohler, and G. Hall, in "Acidity in Paper," (*The Paper Industry*, Vol 7, No. 7, Edward B. Fritz, pub., October 1925, pp. 1056-1063) with some modifications developed at the National Bureau of Standards. In this revised method, one extraction is specified instead of the three extractions originally specified, because Kohler subsequently found that one extraction is sufficient for classification of paper. See "Investigation into the Determination of Acidity and Copper Number in Paper," *Meddelande 56 Statens Provingsanstalt*, Stockholm, 1932. In this article, it is stated that the acid number thus obtained is about three fourths of that obtained by three extractions.

Studies of this test method and other acidity methods are reported by B. L. Wehmhoff, in *Technical Association Papers*, Technical Association of the Pulp and Paper Industry, May 1930 and May 1931.

[3] *Annual Book of ASTM Standards*, Vol 15.09.

[4] *Annual Book of ASTM Standards*, Vol 11.01.

[5] *Annual Book of ASTM Standards*, Vol 15.05.

[6] Available from the Technical Association of the Pulp and Paper Industry, Technology Park, P.O. Box 105113, Atlanta, GA 30348.

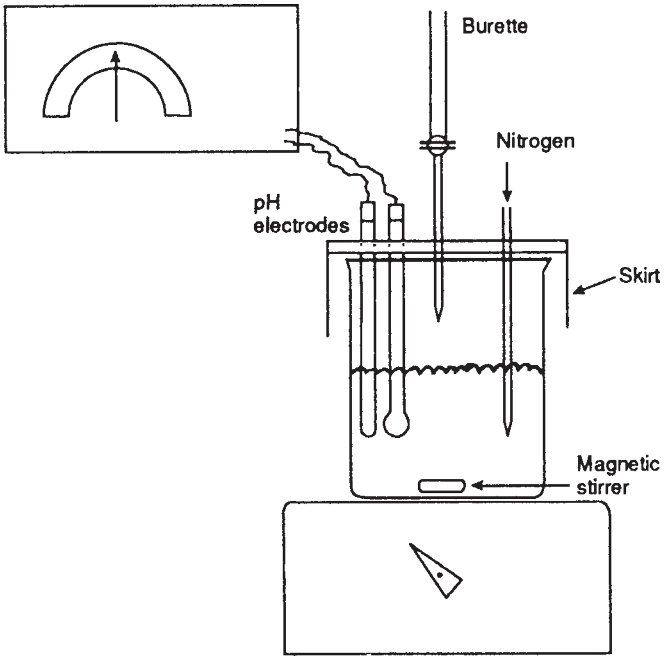

FIG. 1 pH Meter Assembly

6.4.1 *Erlenmeyer Flask*, 500-mL with 24/40 standard taper joint.

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

6.4.3 *Burets*, two 50-mL.

6.4.4 *Beaker*, 400-mL.

6.4.5 *Graduated Cylinders*, 100- and 250-mL.

6.5 *Buchner Funnel*, 60-mL, glass, with medium fritted disk.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specification of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[7] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Hydrochloric or Sulfuric Acid, Standard Solution (0.01N)*—Prepare and standardize a 0.01N solution of hydrochloric acid (HCl) or sulfuric acid (H_2SO_4). Calculate normality to four decimal places.

7.3 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean freshly boiled and cooled reagent water, Type I or II, as described in Specification D 1193.

7.4 *Sodium Hydroxide, Standard Solution (0.01N)*—Prepare and standardize a 0.01N solution of NaOH. Calculate normality to four decimal places.

7.5 *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_2SO_4 and through a tower or U-tube containing ascarite or soda lime, 120 to 150 mm long, before connecting to the gas dispensing tube.

8. Sampling

8.1 Sample the material to be tested in accordance with Practice D 585.

9. Test Specimen

9.1 From each test unit of the sample, cut three representative test specimens of approximately 20 g each.

9.2 Cut each specimen into 5 to 10-mm squares with a pair of scissors.

10. Procedure

10.1 Allow the prepared test specimen to reach moisture equilibrium with the atmosphere of the balance case.

10.2 Weigh two 5-g portions to the nearest 1 mg for extraction.

10.3 At the same time, and using the third prepared sample, determine the moisture content of the material in accordance with Test Method D 644.

10.4 Transfer the weighed portions of sample (see 10.2) to two 500-mL Erlenmeyer flasks and add 250 mL of boiling water to each flask. In some cases, the fibers absorb water slowly and tend to float on the surface of the water. This may be avoided by first adding small portions of the water and shaking well until the fibers are thoroughly saturated.

10.5 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.

10.6 After the water is added, affix a condenser to each flask. The air condenser may be used if an oil or water bath regulated to 100°C is used. When using a hot plate, the West or Allihn type condenser (see 6.5.2) must be used.

10.7 Place the flask with condenser affixed on the heat source. Allow a few minutes for the sample to come to the bath temperature, or, in the case of the hot plate, to begin to gently boil. Maintain temperature for 60 ± 5 min swirling occasionally.

10.8 At the end of this period, pour the contents of the flask into a Buchner funnel without other filtering medium. Wash the fibers remaining in the flask into the Buchner funnel with 10 mL of hot distilled water.

10.9 Apply gentle suction.

10.10 Wash the fibers in the Buchner funnel with 25 to 50 mL of hot distilled water. Apply suction to complete the filtration and cool the extract rapidly in a stoppered Erlenmeyer flask.

10.11 As soon as the extract reaches room temperature, quantitatively transfer it to a 400-mL beaker.

10.12 *Titration*—Titrate the sample with standard acid or base as follows:

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[7] "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY and the "United States Pharmacopeia."

10.12.1 Pass a gentle stream of nitrogen or carbon dioxide-free air through the extract (or blank) as shown in Fig. 1. Continue to sweep the sample with nitrogen or carbon dioxide-free air during the entire titration.

10.12.2 Follow the manufacturer's instructions for operating the pH meter, or consult Test Methods D 1293 or Test Method E 70.

10.12.3 Using a pH meter, determine the pH of the extract (or blank). If the initial pH is below 7.0, the extract (or blank) is acidic, and the titration is done with the standardized sodium hydroxide. If the initial pH is above 7.0, the extract (or blank) is alkaline, and the titration is done with standardized hydrochloric or sulfuric acid.

10.12.4 Using a pH meter, titrate with acid or alkali to a pH of 7.0, while passing nitrogen or CO_2-free air through the specimen as shown in Fig. 1. Follow the manufacturers instructions for operating the pH meter, or consult Test Methods D 1293 or Test Method E 70.

11. Calculations

11.1 Calculate milliequivalents of acid or base consumed per gram, as follows:

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

$$\text{milliequivalents of reagent consumed} = V \times N \tag{1}$$

where:
V = reagent required to reach pH 7.0, mL, and
N = normality of the reagent used.

11.1.2 Correct the milliequivalents of reagent (acid or base) consumed by the sample for the blank titration as follows:

11.1.2.1 If the sample extract and the blank are *both* acidic or *both* alkaline, *subtract* the milliequivalents of the blank from the milliequivalents of the sample.

11.1.2.2 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.

11.1.3 Calculate the milliequivalents of acidity or alkalinity per gram for each sample by dividing the corrected milliequivalents by the sample weight (see 10.2) corrected for moisture (see 10.3).

11.2 Calculate as percent SO_3 or percent NaOH present.

11.2.1 Where the calculation for 11.1 shows the sample to be acidic (that is the original sample pH was below 7.0 (see 10.12.3) calculate SO_3 present as:

$$SO_3, \% = \frac{\text{milliequivalents of base consumed} \times 0.040}{W} \times 100 \tag{2}$$

where:
W = sample weight (10.2) corrected for moisture (10.3).

NOTE 2—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.

NOTE 3—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.

11.2.2 Where the calculation in 11.1 shows the sample to be basic (that is the original sample pH was above 7.0 (see 10.12.3)) calculate percent NaOH as:

$$NaOH, \% = \frac{\text{milliequivalents of acid consumed} \times 0.040}{W} \times 100 \tag{3}$$

where:
W = sample weight (10.2) corrected for moisture (10.3).

11.3 The milliequivalents of *acid consumed* (11.2) is numerically equivalent to the milliequivalents of base *present* in the sample. Likewise, the milliequivalents of *base consumed* (11.2) is numerically equivalent to the milliequivalent of *acid present* in the sample.

12. Report

12.1 The milliequivalents of acid or base present in the sample, or the percent alkalinity as calculated in 11.2 may be reported, as agreed by parties involved in the testing.

13. Precision

13.1 *Repeatability (Within a Laboratory)*—Duplicate test determinations on low-acidity paper on each of three different days in one laboratory agreed to 0.01 % acidity. It is estimated from this limited information that repeatability of test results for low-acidity paper would be about 0.02 % acidity.

13.2 The reproducibility (between laboratories) is not known.

13.3 The above terms are used in accordance with the definitions in TAPPI T 1206.

14. Keywords

14.1 paper products; water-soluble acidity; water-soluble alkalinity

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