Designation: D778 – 97 (Reapproved 2007)

An American National Standard

Standard Test Methods for Hydrogen Ion Concentration (pH) of Paper Extracts (Hot-Extraction and Cold-Extraction Procedures)[1]

This standard is issued under the fixed designation D778; 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 These test methods cover two procedures for determination of the hydrogen ion concentration, expressed in terms of pH, of an aqueous extract of paper.

1.2 These test methods may be applied to writing, printing, and sized industrial papers.

1.3 These test methods are not intended to be used for determination of pH of insulating papers, for that see Test Methods D202.

1.4 The test methods appear as follows:

	Section
Test Method A—pH of Paper (Unfiltered Extract) After Extraction for 1 h in Cold (25 ± 5°C) Water	11.1
Test Method B—pH of Paper (Unfiltered Extract) After Extraction for 1 h in Boiling Water	11.2

1.5 Specifications may be based on Test Method A or Test Method B, or both.

1.6 Where a specification or specific instructions or agreement to use only one of these test methods is absent, determine pH using both procedures, and interpret results based on Section 12.

1.7 *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:*[2]

D202 Test Methods for Sampling and Testing Untreated Paper Used for Electrical Insulation

D585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, and Related Product

D1193 Specification for Reagent Water

D1293 Test Methods for pH of Water

D1968 Terminology Relating to Paper and Paper Products

E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

3. Terminology

3.1 Definitions shall be in accordance with Terminology D1968 and the *Dictionary of Paper.*[3]

4. Summary of Test Methods

4.1 Test Method A consists of the extraction of a 1-g test specimen in 70 mL of cold distilled water (25 ± 5°C) for 1 h, and determination of pH without filtration using a commercial pH meter.

4.2 Test Method B consists of the extraction of a 1-g test specimen in 70 mL of boiling distilled water for 1 h, followed by cooling of the extract and determination of the pH without filtration using a commercial pH meter.

5. Significance and Use

5.1 The acidity or alkalinity of a paper sample extract is important because of the effect of paper acidity or paper alkalinity on the permanence of the paper. Although acidity or alkalinity may be determined by titration the pH is often more indicative of the stability of paper than is the total acidity or alkalinity.

6. Apparatus

6.1 *pH Meter*—Use a pH meter of Type II or Type III as specified in Test Methods D1293. In general terms this is a pH meter accurate to 0.01 pH units with temperature compensation suitable for making pH measurements over the 0 to 14-pH range.

6.2 Reflux condensers, preferably water cooled, West or Allihn Type, with 300-mm jackets, standard taper inner joints

[1] These test methods are under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and are the direct responsibility of Subcommittee D06.92 on Standard Documents Relating to Paper and Paper Products.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Technical Association of the Pulp and Paper Industry (TAPPI), 15 Technology Parkway South, Norcross, GA 30092, http://www.tappi.org.

and drip tips; or air condensers, 10-mm diameter and 1000 mm long with ST inner joints and drip tips, ST joints to fit the flasks (required on hot-extraction procedure, only).

7. Reagents

7.1 *Distilled Water*—The distilled water used in these test methods must comply with Type II or Type III reagent water as specified in Specification D1193.

7.2 *Standard Buffer Solutions*:

7.2.1 If buffer solutions are prepared in the laboratory, their preparation and corporation must comply with applicable sections of Test Method E70 or Test Methods D1293. Some knowledge of the expected pH may be required to indicate which buffers (pH values) will be required for use.

7.2.2 Commercially available pH buffers may be used if desired. Common pH values are 4.0, 7.0, and 10.0. Purchase the ones appropriate for samples being tested.

8. Sampling

8.1 *Acceptance Sampling*—Acceptance sampling shall be in accordance with Practice D585.

8.2 *Sampling for Other Purposes*—The sampling and the number of the test specimens depends on the purpose of the testing. Practice E122 is recommended.

9. Test Specimen

9.1 A composite evaluation is considered appropriate for these test methods unless otherwise specified.

9.2 Take an equal number of sheets (one or more) from each test unit, but not less than a total of five sheets, and combine the sheets from all test units. Holding the sheets at one end, cut and cross-cut the other end into 5 to 10-mm square.

9.3 If the paper is more than 0.012 in. (0.30 mm) in thickness, or greater in apparent density than 0.90 g/cm^3, the specimen must be split by delamination into thickness of not over 0.008 in. (0.20 mm) before it is cut into the small squares.

9.4 From each quantity of cut paper, weigh a test specimen of 1 ± 0.01 g, using an equal amount of material from each layer in cases where the sample has been delaminated as required in 9.3. Do not include any material in the specimen that has been touched by the fingers.

9.5 Perform determinations at least in duplicate. In special cases where it is necessary to evaluate different portions of the lot separately, take a portion from each test unit of the sample and perform duplicate determinations on each portion independently, using the procedure specified for the composite.

10. Calibration

10.1 Calibration of the pH meter must be done following applicable sections of Test Methods D1293 or Test Method E70. The instruction manual of the specific pH meter must be consulted for any operations specific to that pH meter.

11. Procedure

11.1 *Test Method A—pH of Paper (Unfiltered Extract) After Extraction for 1 h in Cold (25 ± 5°C) Water:*

11.1.1 Transfer the test specimen to a 100-mL beaker. Add about 5 mL of cold (25 ± 5°C) distilled water and macerate with a flattened glass stirring rod until the specimen is wet.

11.1.2 Add more water to bring the volume to 70 mL, stir well, cover with a watch glass, and allow to stand for 1 h at 25 ± 5°C. The specimen may stand for 3 or 4 h **(1)**[4] if no contamination occurs.

11.1.3 When making referee or research tests, pass pure nitrogen or CO_2-free air through the solution until the pH is measured.

NOTE 1—Air may be cleaned by passing it through a gas washing bottle containing at least 200 mL of 3 *N* H_2SO_4 and then through a tower or U-tube containing ascarite or soda lime, 120 to 150 mm long.

11.1.4 Measure the pH on the prepared extract (see 11.1.1 through 11.1.3) as specified in 11.3.

11.2 *Test Method B pH of Paper (Unfiltered Extract) After Extraction for 1 h in Boiling Water*:

11.2.1 Transfer the test specimen to a 125-mL Erlenmeyer flask. Add about 70 mL of distilled water, stir well, and attach the condenser.

NOTE 2—Water-cooled condensers are desirable. The air condensers may be used if the temperature of the hot plate can be controlled as indicated in 11.2.2.

11.2.2 Place the flask on the hot plate. Reduce the hot plate temperature as necessary using the hot plate controls and boil gently for 1 h, taking care not to exceed the capacity of the condenser. The temperature should be maintained between 98 and 100°C (208 and 212°F).

11.2.3 At the end of the extraction period cool the flask in running water (to about 35 to 40°C), with the condenser tube in place and its upper end protected by a loosely fitted small beaker. Replace the condenser with a glass stopper and cool at room temperature. Transfer the mixture to a 100-mL beaker.

11.2.4 Transfer the extracts for referee or research tests to the 100-mL beakers while hot. Pass nitrogen or CO_2-free air through these extracts while cooling and until the pH is measured.

11.2.5 Measure the pH on the prepared extract (see 11.2.1 through 11.2.4) as specified in 11.3.

11.3 *pH Measurement*:

11.3.1 Keep a nitrogen or CO_2-free air cap over the solution during pH measurement. Stir and measure to 0.1 pH unit the pH of the unfiltered mixture in accordance with the instructions for the pH meter used.

11.3.2 Through observation, make certain that no plies of paper adhere to the electrodes during pH measurement.

11.3.3 Before recording the pH, leave the electrodes in the solution until there is no measurable drift in 30 s.

11.3.4 Wash the electrodes with distilled water after each measurement and recalibrate frequently.

11.3.5 Keep the electrodes, buffer, test solution, and wash water at the same temperature and set the compensator for this temperature.

12. Interpretation of Results

12.1 The pH of an extract of a paper sample indicates the extent to which components of the paper alter the hydrogen-hydroxyl equilibrium of pure water. In the case where the paper

[4] The boldface numbers in parentheses refer to a list of references at the end of the text.