

ASTM-D1293

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FSC 6810

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Standard Test Methods for pH of Water¹

This standard is issued under the fixed designation D 1293; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover the determination of pH by electrometric measurement using the glass electrode as the sensor. Two test methods are given as follows:

	Sections
Test Method A—Precise Laboratory Measurement	8 to 15
Test Method B—Routine or Continuous Measurement	16 to 24

1.2 Test Method A covers the precise measurement of pH in water utilizing at least two of seven standard reference buffer solutions for instrument standardization.

1.3 Test Method B covers the routine measurement of pH in water and is especially useful for continuous monitoring. Two buffers are used to standardize the instrument under controlled parameters, but the conditions are somewhat less restrictive than those in Test Method A.

1.4 Both test methods are based on the pH scale established by NIST (formerly NBS) Standard Reference Materials.²

1.5 Neither test method is considered to be adequate for measurement of pH in water whose conductivity is less than about 5 $\mu\text{S}/\text{cm}$. Refer to Test Methods D 5128 and D 5464.

1.6 Precision and bias data were obtained using buffer solutions only. It is the user's responsibility to assure the validity of these test methods for untested types of water.

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:

- D 1066 Practice for Sampling Steam³
- D 1067 Test Methods for Acidity or Alkalinity of Water³
- D 1129 Terminology Relating to Water³
- D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits³

¹ These test methods are under the jurisdiction of ASTM Committee D-19 on Water and are the direct responsibility of Subcommittee D19.03 on Sampling Water and Water-Formed Deposits, Analysis of Water for Power Generation and Process Use, On-Line Water Analysis, and Surveillance of Water.

Current edition approved Dec. 10, 1999. Published March 2000. Originally published as D 1293 – 53 T. Last previous edition D 1293 – 95.

² "Standard Reference Materials: Standardization of pH Measurements" Wu and Koch, NBS Special Publications No. 260-53, 1988.

³ *Annual Book of ASTM Standards*, Vol 11.01.

D 1193 Specification for Reagent Water³

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water³

D 3370 Practices for Sampling Water from Closed Conduits³

D 5128 Test Method for On-Line pH Measurement of Water of Low Conductivity³

D 5464 Test Methods for pH Measurement of Water of Low Conductivity³

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁴

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *pH, n*—the pH of an aqueous solution is derived from E , the electromotive force (emf) of the cell

glass electrode | solution || reference electrode

(where the double vertical line represents a liquid junction) when the electrodes are immersed in the solution in the diagrammed position, and E_s is the electromotive force obtained when the electrodes are immersed in a reference buffer solution.

With the assigned pH of the reference buffer designated as pH_s , and E and E_s expressed in volts is the following:⁵

$$\text{pH} = \text{pH}_s + \frac{(E - E_s)F}{2.3026 RT}$$

where:

F = Faraday,

R = gas constant, and

T = absolute temperature, t ($^{\circ}\text{C}$) + 273.15.

The reciprocal of $F/2.3026 RT$ is known as the slope of the electrode, and is the expected difference in observed voltage for two measurements one pH unit apart. Values of the slope at various temperatures are given in Table 1.

4. Summary of Test Method

4.1 The pH meter and associated electrodes are standardized

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

⁵ Bates, R. G., *Determination of pH: Theory and Practice*, 2nd Ed., J. Wiley and Sons, New York, 1973, p. 29.

TABLE 1 Slope Factor at Various Temperatures

Temperature, °C	Slope, millivolts
0	54.20
5	55.19
10	56.18
15	57.17
20	58.17
25	59.16
30	60.15
35	61.14
40	62.13
45	63.13
50	64.12
55	65.11
60	66.10
65	67.09
70	68.09
75	69.08
80	70.07
85	71.06
90	72.05
95	73.05

against two reference buffer solutions that closely bracket the anticipated sample pH. The sample measurement is made under strictly controlled conditions and prescribed techniques.

5. Significance and Use

5.1 The pH of water is a critical parameter affecting the solubility of trace minerals, the ability of the water to form scale or to cause metallic corrosion, and the suitability of the water to sustain living organisms. It is a defined scale, based on a system of buffer solutions² with assigned values. In pure water at 25°C, pH 7.0 is the neutral point, but this varies with temperature and the ionic strength of the sample.⁶ Pure water in equilibrium with air has a pH of about 5.5, and most natural uncontaminated waters range between pH 6 and pH 9.

6. Purity of Reagents

6.1 Reagent grade chemicals shall be used in all tests, except as specifically noted for preparation of reference buffer solutions. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type I.

⁶ The relative acidity or alkalinity measured by pH should not be confused with total alkalinity or total acidity (for example, Test Methods D 1067). Thus, 0.1 M HCl and 0.1 M acetic acid have the same total acidity, but the HCl solution will be more acidic (approximately pH 1 versus pH 3.).

⁷ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

7. Sampling

7.1 Collect samples in accordance with Practice D 1066, Specification D 1192, or Practices D 3370, whichever is applicable.

TEST METHOD A—PRECISE LABORATORY MEASUREMENT OF pH

8. Scope

8.1 This test method covers the precise measurement of pH in water under strictly controlled laboratory conditions.

9. Interferences

9.1 The glass electrode reliably measures pH in nearly all aqueous solutions and in general is not subject to solution interference from color, turbidity, colloidal matter, oxidants, or reductants.

9.2 The reference electrode may be subject to interferences and should be chosen to conform to all requirements of Sections 10 and 12. Refer also to Appendix X1.3.

9.3 The true pH of an aqueous solution or extract is affected by the temperature. The electromotive force between the glass and the reference electrode is a function of temperature as well as pH. The temperature effect can be compensated automatically in many instruments or can be manually compensated in most other instruments. The temperature compensation corrects for the effect of changes in electrode slope with temperature but does not correct for temperature effects on the chemical system being monitored. It does not adjust the measured pH to a common temperature; therefore, the temperature should be reported for each pH measurement. Temperature effects are discussed further in Appendix X1.2.

9.4 The pH response of the glass electrode/reference electrode pair is imperfect at both ends of the pH scale. The indicated pH value of highly alkaline solutions may be too low, by as much as 1 pH, depending on electrode composition and sample conditions. See X1.5.1. The indicated pH value of strong aqueous solutions of salts and strong acids having a pH less than 1, will often be higher than the true pH value. Interferences can be minimized by the selection of the proper glass and reference electrodes for measurements in highly alkaline or acidic solutions.

9.5 A few substances sometimes dispersed in water appear to poison the glass electrode. A discussion of this subject is given in Appendix X1.4.

10. Apparatus

10.1 *Laboratory pH Meter*—Almost all commercially available meters are of the digital type and will have either manual or automatic calibration, and either manual or automatic temperature (slope) correction. All four types are permissible. However, readability to 0.01 pH is essential (Section 14), and the ability to read in millivolts is useful in troubleshooting.

10.2 *Glass Electrode*—The pH response of the glass electrode shall conform to the requirements set forth in 12.1 through 12.5. The glass electrode lead wire shall be shielded. New glass electrodes and those that have been stored dry shall be conditioned and maintained as recommended by the manufacturer.

10.3 Reference Electrode—This may be used as separate “half cell,” or it may be purchased integral with the glass pH electrode body, as a combination electrode. The internal reference element may be calomel (mercury/mercurous chloride), silver/silver chloride, or an iodide-iodine redox couple. For best performance, the reference element should be the same type in both the reference electrode and inside the pH electrode. For all three types, the junction between the reference filling solution and the sample may be either a flowing or nonflowing junction. The flowing liquid junction-type unit ensures that a fresh liquid junction is formed for each measurement and shall be used for Test Method A determinations. If a saturated calomel electrode is used, some potassium chloride crystals shall be contained in the saturated potassium chloride solution. If the reference electrode is of the flowing junction type, the design of the electrode shall permit a fresh liquid junction to be formed between the reference electrode solution and the buffer standard or tested water for each measurement and shall allow traces of solution to be washed from the outer surfaces of the electrodes. To ensure the desired slow outward flow of reference electrode solution, the solution pressure inside the liquid junction should be kept somewhat in excess of that outside the junction. In nonpressurized applications, this requirement can be met by maintaining the inside solution level higher than the outside water level. If the reference electrode is of the nonflowing junction type, these outward flow and pressurization considerations do not apply. The reference electrode and junction shall perform satisfactorily as required in the standardizing procedure described in 12.1 through 12.5. A discussion of reference electrodes is given in Appendix X1.3.

10.4 Temperature Compensator—The thermocompensator is a temperature-sensitive resistance element immersed in the water sample with the electrodes. The thermocompensator automatically corrects for the change in slope of the glass electrode (with change of temperature) but does not correct for

actual changes in sample pH with temperature. The automatic thermocompensator is not required if the water temperature is essentially constant and the analyst chooses to use the manual temperature compensation feature of the pH meter.

11. Reagents

11.1 Reference Buffer Solutions—The pH values of the reference buffer solutions measured at several temperatures are listed in Table 2. Table 3 identifies each buffer salt by its National Institute of Standards and Technology (NIST) number and provides a recommended drying procedure prior to use. The current renewal of each NIST standard reference material should be used. Keep the five reference buffer solutions with pH less than 9.5 in bottles of chemically resistant glass. Keep the calcium hydroxide solutions in a plastic bottle that is nonporous to air (that is, polypropylene or high density polyethylene). Keep all the reference buffer solutions well-stoppered and replace if a visible change is observed.

11.1.1 Borax Reference Buffer Solution ($pH_s = 9.18$ at 25°C)—Dissolve 3.80 g of sodium tetraborate decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) in water and dilute to 1 L.

11.1.2 Calcium Hydroxide Reference Buffer Solution ($pH_s = 12.45$ at 25°C)—Prepare pure calcium hydroxide ($\text{Ca}(\text{OH})_2$) from well-washed calcium carbonate (CaCO_3) of low-alkali grade by slowly heating the carbonate in a platinum dish at 1000°C and calcining for at least 45 min at that temperature. After cooling in a desiccator, add the calcined product slowly to water with stirring, heat the resultant suspension to boiling, cool, and filter through a funnel having a fritted-glass disk of medium porosity. Collect the solid from the filter, dry it in an oven at 110°C , and crush it to a uniform and fine granular state. Prepare a saturated calcium hydroxide solution by vigorously shaking a considerable excess (about 3 g/L) of the fine granular product in water at 25°C in a stoppered plastic bottle (that is, polypropylene or high density polyethylene) that is essentially nonporous to gases. Allow the gross excess of solid to settle

TABLE 2 pH_s of Reference Buffer Solutions^A

Temperature, °C	Tetroxalate Solution	Tartrate Solution	Phthalate Solution	Phosphate Solution	Borax Solution	Sodium Bicarbonate Sodium Carbonate	Calcium Hydroxide Solution
0	1.67	...	4.00	6.98	9.46	10.32	13.42
5	1.67	...	4.00	6.95	9.39	10.25	13.21
10	1.67	...	4.00	6.92	9.33	10.18	13.00
15	1.67	...	4.00	6.90	9.28	10.12	12.81
20	1.68	...	4.00	6.88	9.23	10.06	12.63
25	1.68	3.56	4.00	6.86	9.18	10.01	12.45
30	1.68	3.55	4.01	6.85	9.14	9.97	12.29
35	1.69	3.55	4.02	6.84	9.11	9.93	12.13
40	1.69	3.55	4.03	6.84	9.07	9.89	11.98
45	1.70	3.55	4.04	6.83	9.04	9.86	11.84
50	1.71	3.55	4.06	6.83	9.02	9.83	11.71
55	1.72	3.55	4.07	6.83	8.99	...	11.57
60	1.72	3.56	4.09	6.84	8.96	...	11.45
70	1.74	3.58	4.12	6.85	8.92
80	1.77	3.61	4.16	6.86	8.89
90	1.79	3.65	4.19	6.88	8.85
95	1.81	3.67	4.21	6.89	8.83

^AFor a discussion of the manner in which these pH values were assigned, see Bates, R. G., “Revised Standard Values for pH Measurements from 0 to 95°C ,” *Journal of Research*, NBS, Vol 66A, 1962, p. 179. The reference values were obtained without a liquid junction, which has an uncertainty of ± 0.005 . Liquid junction electrode values may have an uncertainty of ± 0.012 , with uncertainty ± 0.03 for the tetroxalate and the $\text{Ca}(\text{OH})_2$. More recent values have been published in *pH Measurement* by Helmuth Galster, VCH Publishers, Inc., New York, 1991.

TABLE 3 National Institute of Standards and Technology (NIST) Materials for Reference Buffer Solutions

NIST Standard Reference Material Designation	Buffer Salt ^A	Drying Procedure
187	Borax (sodium tetraborate decahydrate)	Drying not necessary (this salt should not be oven-dried)
186	disodium hydrogen phosphate	2 h in oven at 130°
186	potassium dihydrogen phosphate	2 h in oven at 130°C
185	potassium hydrogen phthalate	2 h in oven at 110°C
188	potassium hydrogen tartrate	drying not necessary
189	potassium tetroxalate dihydrate	should not be dried
191	sodium bicarbonate	should not be dried
192	sodium carbonate	2 h in oven at 275°C
2193	calcium carbonate	see NIST material certificate

^AThe buffer salts listed can be purchased from the Standard Reference Materials Program, National Institute of Standards and Technology, Gaithersburg, MD 20899.

and filter the solution with suction through a fritted-glass funnel of medium porosity. The filtrate is the reference buffer solution. Contamination of the solution with atmospheric carbon dioxide renders it turbid and indicates need for replacement.

11.1.3 *Phosphate Reference Buffer Solution* ($\text{pH}_s = 6.86$ at 25°C)—Dissolve 3.39 g of potassium dihydrogen phosphate (KH_2PO_4) and 3.53 g of anhydrous disodium hydrogen phosphate (Na_2HPO_4) in water and dilute to 1 L.

11.1.4 *Phthalate Reference Buffer Solution* ($\text{pH}_s = 4.00$ at 25°C)—Dissolve 10.12 g of potassium hydrogen phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$) in water and dilute to 1 L.

11.1.5 *Tartrate Reference Buffer Solution* ($\text{pH}_s = 3.56$ at 25°C)—Shake vigorously an excess (about 75 g/L) of potassium hydrogen tartrate ($\text{KHC}_4\text{H}_4\text{O}_6$) with 100 to 300 mL of water at 25°C in a glass-stoppered bottle. Filter, if necessary, to remove suspended salt. Add a crystal of thymol (about 0.1 g) as a preservative.

11.1.6 *Tetroxalate Reference Buffer Solution* ($\text{pH}_s = 1.68$ at 25°C)—Dissolve 12.61 g of potassium tetroxalate dihydrate ($\text{KHC}_2\text{O}_4 \cdot \text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 L.

11.1.7 *Sodium Bicarbonate—Sodium Carbonate Reference Buffer Solution* ($\text{pH}_s = 10.01$ at 25°C)—Dissolve 2.092 g of sodium bicarbonate (NaHCO_3) and 2.640 g of sodium carbonate (Na_2CO_3) in water and dilute to 1 L.

11.2 *Other Buffer Solutions*—A buffer solution other than that specified may be used as a working standard in the method providing that in each case such a solution is first checked against the corresponding reference buffer solution, using the procedures of the method, and is found to differ by not more than ± 0.02 pH unit.

11.3 *Commercial Buffer Solutions*—Commercially available prepared buffer solutions are not acceptable for the standardization in Test Method A.

12. Standardization of Assembly

12.1 Turn on the instrument, allow it to warm up thoroughly, and bring it to electrical balance in accordance with the manufacturer's instructions. Wash the glass and reference electrodes and the sample container with three changes of water or by means of flowing stream from a wash bottle. Form a fresh liquid junction if a sleeve-type reference junction is used. Note the temperature of the water to be tested. If temperature compensation is to be manual, adjust the temperature setting of the meter to correspond to the temperature of the water to be tested and allow time for all buffers, solutions, and electrodes to equilibrate thermally.

12.2 Select at least two reference buffer solutions, the pH_s values of which closely bracket the anticipated pH (refer to Table 2). Warm or cool the reference solutions as necessary to match within 2°C the temperature of the solution to be tested. Fill the sample container with the first reference buffer solution and immerse the electrodes. Stir the solution as described in 13.3.

12.3 Set the pH_s value of the reference buffer solution at the temperature of the buffer, as read from Table 2 or interpolated from the data therein, according to the manufacturer's instructions.

12.4 Empty the sample container and repeat, using successive portions of the reference buffer solution, until two successive readings are obtained without adjustment of the system. These readings should differ from the pH_s value of the buffer solution by not more than ± 0.02 pH unit.

NOTE 1—If the temperature of the electrode differs appreciably from that of the solution to be tested, use several portions of solution and immerse the electrodes deeply to assure that both the electrodes and the solution are at the desired temperature. To reduce the effects of thermal lag, keep the temperature of electrodes, reference buffer solutions, and the wash as close to that of the water sample as possible.

12.5 Wash the electrodes and the sample container three times with water. Place the second reference buffer solution in the sample container, and measure the pH. Set the temperature corrected value of the second reference buffer solution according to the meter manufacturer's instructions. Use additional portions of the second reference buffer solution, as before, until two successive readings differ by not more than ± 0.02 pH unit. The assembly shall be judged to be operating satisfactorily if the reading obtained for the second reference buffer solution agrees with its assigned pH_s value within 0.05 (or less) pH units.

12.6 If only an occasional pH determination is made, standardize the assembly each time it is used. In a long series of measurements, supplemental interim checks at regular intervals are recommended. Inasmuch as commercially available pH assemblies exhibit different degrees of measurement stability, conduct these checks at intervals of 30 min, unless it is ascertained that less frequent checking is satisfactory to ensure the performance described in 12.2 to 12.5.

13. Procedure

13.1 Standardize the assembly with two reference buffer solutions as described in 12.2 to 12.5 and then wash the electrodes with three changes of water or by means of a flowing stream from a wash bottle.

13.2 Place the water sample in a clean glass beaker provided with a stirring bar and either a thermometer (for meters with manual temperature compensation) or an ATC probe (for meters with automatic temperature compensation).

13.3 Stir during the period of pH measurement at a rate that will prevent splashing and that will avoid loss or gain of acidic or basic gases by interchange with the atmosphere. When necessary, stir briskly enough to intermix the phases of a nonhomogeneous water sample. Stop the stirrer during periods of measurement if fluctuations in readings are observed. (See Appendix X1.3.4 and X1.4.3).

13.4 Insert the electrodes and determine a preliminary pH value (since this value may drift somewhat, it should be considered an estimated value). Measure successive portions of the water sample until readings on two successive portions differ by no more than 0.03 pH unit, and show drifts of less than 0.02 pH unit in 1 min. Two or three portions will usually be sufficient if the water is well buffered.

13.5 Record the pH and temperature of the sample.

13.6 Measure the pH of slightly buffered waters (that are in equilibrium with air) essentially as described in 13.1 to 13.5, but measure the pH of successive portions until the readings for two successive portions differ by no more than 0.1 pH unit. Six or more portions may be necessary.

NOTE 2—Take special precautions if the sample is not in equilibrium with the carbon dioxide of the atmosphere protecting the sample from exposure to the air during measurement. *Measurement of unbuffered or slightly buffered samples is more reliably made in flow-type cells as described in Note 4.* Test Methods D 5464 describe additional precautions that should be taken if the electrical conductivity of the sample is less than about 5 $\mu\text{S}/\text{cm}$.

14. Report

14.1 Report the temperature of the measurement of the nearest 1°C.

14.2 Report the pH of the test solution to the nearest 0.01 pH unit when the pH measurement lies between 1.0 and 12.0.

14.3 Report the pH of the test solution to the nearest 0.1 pH unit when the pH measurement is less than 1.0 or greater than 12.0.

15. Precision and Bias ⁸

15.1 The information summarized in this section was derived from an interlaboratory study performed in 1973 on four buffer solutions having pH values of approximately 3.7, 6.5, 8.2, and 8.4. Eleven laboratories (fourteen operators, with one laboratory providing four operators) analyzed each solution in duplicate and replicated the analysis on another day for a total of 224 determinations. A variety of commercial meters was used in this study. It is assumed that all measurements were made at room temperature.

15.2 Statistical treatment of the data conforms to the recommendations of Practice D 2777. Further information, based on a different statistical interpretation, can be found in Test Method E 70.

15.3 *Precision*—The overall and single-operator precision

of this test method varies with pH as shown in Fig. 1.

15.4 *Bias*—The pH values of the buffer solutions, as determined using a gaseous hydrogen electrode, are compared with values obtained using this test method in Table 4.

15.5 Precision and bias data were obtained using buffer solutions only. It is the user's responsibility to assure the validity of the standards for untested types of water.

TEST METHOD B—ROUTINE OR CONTINUOUS MEASUREMENT OF pH

16. Scope

16.1 This test method is used for the routine measurement of pH in the laboratory and the measurement of pH under various process conditions.

17. Summary of Test Method

17.1 A direct standardization technique is employed in this test method for routine batch samples. Two buffers are used to standardize the instrument under controlled parameters, but the conditions are somewhat less restrictive than those in Test Method A. An indirect standardization procedure is used on flowing systems in which grab samples are removed periodically in order to compare a monitored pH value (of the system) with the reading of a laboratory pH meter.

18. Interferences

18.1 For information on interferences, see Section 9 and Appendix X1.4.

19. Apparatus

19.1 *Laboratory pH Meter*—See 10.1.

19.2 *Glass Electrode*—See 10.2.

19.3 *Reference Electrode*—See 10.3.

19.4 *Temperature Compensator*—See 10.4.

19.5 *Process pH Measurement Instrumentation*—

Instruments that are used for process pH measurements are generally much more rugged than those which are used for very accurate measurements in the laboratory.

19.5.1 *Electrode Chamber*—For process pH measurements; the electrodes and thermocompensator are mounted in an electrode chamber or cell.

19.5.1.1 *Flow-Through Chamber* completely encloses the electrodes and the sample is piped to and from the chamber in a flow-through configuration. Commercially available chambers generally can tolerate temperatures as high as 100°C over a pH range from 0 to 14, and pressures up to 1034 kPa (approximately 150 psi).

19.5.1.2 *Immersion Type Chamber*, suitable for measurement in open streams or tanks, shields but does not completely enclose the electrodes. Immersion-style chambers are available for use at depths to 30 m (100 ft).

19.5.2 *Signal Transmission*—The glass electrode is usually a high-impedance device from which only an extremely small current can be drawn. Shielded cable must be used to connect the electrode to the pH analyzer. The signal can frequently be transmitted up to 300 m (approximately 1000 ft) with no loss in accuracy if the manufacturer's recommendations are followed carefully. However, long runs are vulnerable to electrical

⁸ Supporting data for these test methods have been filed at ASTM Headquarters. Request Research Report RR: D19-1111.