



Standard Test Method for pH of Aqueous Solutions With the Glass Electrode¹

This standard is issued under the fixed designation E 70; 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.

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

1. Scope

1.1 This test method specifies the apparatus and procedures for the electrometric measurement of pH values of aqueous solutions with the glass electrode. It does not deal with the manner in which the solutions are prepared. pH measurements of good precision can be made in aqueous solutions containing high concentrations of electrolytes or water-soluble organic compounds, or both. It should be understood, however, that pH measurements in such solutions are only a semiquantitative indication of hydrogen ion concentration or activity. The measured pH will yield an accurate result for these quantities only when the composition of the medium matches approximately that of the standard reference solutions. In general, this test method will not give an accurate measure of hydrogen ion activity unless the pH lies between 2 and 12 and the concentration of neither electrolytes nor nonelectrolytes exceeds 0.1 M.

1.2 *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 1193 Specification for Reagent Water²

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals³

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

3. Terminology

3.1 Definition:

3.1.1 *pH*—defined formally as the negative logarithm to the base 10 of the conventional hydrogen ion activity. See Appendix X1.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 For the purpose of this test method, the term “meter” shall apply to the instrument used for the measurement of potential (either in millivolts or in terms of pH units), the term “electrodes” to the glass electrode and the reference electrode, and the term “assembly” to the combination of the meter and the electrodes. The performance of the meter shall be differentiated from that of the electrodes.

4. Significance and Use

4.1 pH is, within the limits described in 1.1, an accurate measurement of the hydrogen ion concentration and thus is widely used for the characterization of aqueous solutions.

4.2 pH measurement is one of the main process control variables in the chemical industry and has a prominent place in pollution control.

5. Apparatus

5.1 *pH meters*—Many excellent pH meters are available from commercial sources. To some extent, the choice of meter will depend on the desired precision of measurement. The meter may operate on a null-detection principle or may utilize digital readout or a direct deflection meter with a large scale. Power may be supplied by batteries or a-c operation may be provided. The maximum grid current drawn from the glass electrode during measurement shall not exceed 2×10^{-12} A. Automatic or manual adjustment shall allow for changes in $F/(RT \ln 10)$ when the temperature of the assembly is altered. For referee work, or in case of dispute, meters capable of discriminating changes of pH to 0.01 unit (0.6 mV) or less shall be used.

5.2 Reference Electrodes and Glass Electrodes:

¹ This test method is under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and is the direct responsibility of Subcommittee E15.01 on General Standards.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 15.05.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

5.2.1 The saturated calomel electrode and the 3.5 M calomel electrode are suitable as reference electrodes in pH assemblies (Note 1). If the saturated electrode is used, a few crystals of solid potassium chloride shall be present in the chamber surrounding the electrode element at each temperature. The design of the electrode shall permit a fresh liquid junction between the solution of potassium chloride and the buffer or test solution to be formed for each test and shall allow traces of solution to be readily removed by washing.

NOTE 1—Other reference electrodes of constant potential may be used, provided no difficulty is experienced in standardizing the assembly as described in Section 8.

5.2.2 The silver-silver chloride electrode also is used widely as a reference electrode.

5.2.3 Commercial glass electrodes are designed for certain specific ranges of pH and temperature; consequently, the pH and temperature of the test solutions shall be considered in selecting the glass electrode for use. The pH response shall conform with the requirements set forth in Section 7. The leads shall be shielded from the effects of body capacitance.

5.2.4 If the assembly is in intermittent use, the ends of the electrodes shall be immersed in distilled water between measurements. The high-alkalinity type of glass electrode shall be stored in the borax buffer solution. For prolonged storage, glass electrodes may be allowed to become dry, and reference electrodes shall be capped to prevent undue evaporation.

NOTE 2—New glass electrodes and those that have been stored dry shall be conditioned as recommended by the manufacturer. Requirements for the physical dimensions and shape of the electrodes and the composition of the internal reference solution are not considered part of this test method.

6. Reagents and Materials

6.1 The pH(S) of six recommended standard solutions at several temperatures is listed in Table 1. The buffer solutions shall be prepared from highly purified materials sold specifically as pH standards (Note 3). Potassium hydrogen phthalate and the two phosphate salts shall be dried at 110°C for 1 h

before use, but borax and sodium bicarbonate shall not be heated above room temperature. Potassium dihydrogen citrate shall be dried for 1 h at 80°C, and sodium carbonate shall be ignited for 1 h at 270°C before use. The standard solutions shall be prepared as described in 6.4-6.9. They shall be preserved in bottles of chemically resistant glass or polyethylene and shall be replaced at an age of 6 weeks, or earlier if a visible change should occur in the solution.

NOTE 3—Six of the buffer salts can be obtained in the form of standard reference materials from the National Bureau of Standards. These materials are numbered as follows:

Buffer Salt	SRM No.
Potassium hydrogen phthalate	185
Potassium dihydrogen phosphate	186I
Disodium hydrogen phosphate	186II
Borax	187
Sodium bicarbonate	191
Sodium carbonate	192

The pH(S) values may vary slightly from one lot to another; consequently, the values given on the SRM certificate should be used in preference to those given in Table 2, if slight differences exist.

6.2 Commercial standard buffers are available. For the most exact measurements, the value of the commercial buffer should be verified using one of the recommended standard buffers in Table 1.

6.3 *Distilled Water*—The conductivity of the distilled water shall not exceed $2 \times 10^{-6} \text{ s} \cdot \text{cm}^{-1}$. For the preparation of the citrate, phthalate, and phosphate solutions, the water need not be freed of dissolved carbon dioxide. The water used for the borax standard and the carbonate standard shall be boiled for 15 min or purged with air free of carbon dioxide and shall be protected with a soda-lime tube or equivalent (Note 4) while cooling and in storage. The pH of the carbon dioxide-free water shall be between 6.6 and 7.5 at 25°C. The temperature of the water used to prepare the standards shall be within 2°C of 25°C. The amounts of the buffer salts given in 5.3 through 5.8 are weights in air near sea level determined with brass weights.

NOTE 4—The water used for preparing the standard buffer solutions shall be Types I or II reagent water in accordance with Specification

TABLE 1 pH(S) of Standard Solutions^{A,B}

Temperature, °C	A	B	C	D	E	F
0	3.863	4.003	6.984	7.534	9.464	10.317
10	3.820	3.998	6.923	7.472	9.332	10.179
20	3.788	4.002	6.881	7.429	9.225	10.062
25	3.776	4.008	6.865	7.413	9.180	10.012
30	3.766	4.015	6.853	7.400	9.139	9.966
35	3.759	4.024	6.844	7.389	9.102	9.925
40	3.753	4.035	6.838	7.380	9.068	9.889
50	3.749	4.060	6.833	7.367	9.011	9.828
60	...	4.091	6.836	...	8.962	...
70	...	4.126	6.845	...	8.921	...
80	...	4.164	6.859	...	8.885	...
90	...	4.205	6.877	...	8.850	...

^A The compositions of the standard solutions are:

A—KH₂ citrate, $m = 0.05 \text{ mol kg}^{-1}$

B—KH phthalate, $m = 0.05 \text{ mol kg}^{-1}$

C—KH₂PO₄, $m = 0.025 \text{ mol kg}^{-1}$; Na₂HPO₄, $m = 0.025 \text{ mol kg}^{-1}$

D—KH₂PO₄, $m = 0.008695 \text{ mol kg}^{-1}$; Na₂HPO₄, $m = 0.03043 \text{ mol kg}^{-1}$

E—Na₂B₄O₇, $m = 0.01 \text{ mol kg}^{-1}$

F—NaHCO₃, $m = 0.025 \text{ mol kg}^{-1}$; Na₂CO₃, $m = 0.025 \text{ mol kg}^{-1}$

where m denotes molality.

^B For a discussion of the manner in which these pH(S) values were assigned, see Chapter 4 of the book by Bates, R. G., *Determination of pH, Theory and Practice*, John Wiley and Sons, Second edition, New York, 1973.

TABLE 2 Bias of pH Measurements

Nominal pH	Hydrogen Electrode	Glass Electrode	Difference
3.7	3.715	3.73	+ 0.015
6.5	6.519	6.53	+ 0.011
8.2	8.174	8.18	+ 0.006
8.4	8.478	8.45	- 0.028

D 1193. Precautions shall be taken to prevent contamination of the distilled water with traces of the material used for protection against carbon dioxide.

6.4 *Citrate, Standard Solution A (molality = 0.05; pH(S) = 3.776 at 25°C)*—Dissolve 11.41 g of potassium dihydrogen citrate in distilled water and dilute to 1 L.

6.5 *Phthalate, Standard Solution B (molality = 0.05; pH(S) = 4.008 at 25°C)*—Dissolve 10.12 g of potassium hydrogen phthalate in distilled water and dilute to 1 L.

6.6 *Phosphate, Standard Equimolal Solution C (molality of each phosphate salt = 0.025; pH(S) = 6.865 at 25°C)*—Dissolve 3.388 g of potassium dihydrogen phosphate and 3.533 g of disodium hydrogen phosphate in distilled water and dilute to 1 L.

6.7 *Phosphate, Standard Solution D (1 + 3) (molality of $KH_2PO_4 = 0.008695$, molality of $Na_2HPO_4 = 0.03043$; pH(S) = 7.413 at 25°C)*—Dissolve 1.179 g of potassium dihydrogen phosphate and 4.302 g of disodium hydrogen phosphate in distilled water and dilute to 1 L.

6.8 *Borax, Standard Solution E (molality = 0.01; pH(S) = 9.180 at 25°C)*—Dissolve 3.80 g of sodium tetraborate decahydrate (borax) in distilled water and dilute to 1 L.

6.9 *Carbonate, Standard Solution F (molality of each carbonate salt = 0.025; pH(S) = 10.012 at 25°C)*—Dissolve 2.092 g of sodium bicarbonate and 2.640 g of sodium carbonate in distilled water and dilute to 1 L.

7. Performance Tests of Meter and Electrodes

NOTE 5—Except for measurements of the highest precision, it will usually be unnecessary to perform the tests described in this section. In the usual pH measurement, the stability of the meter, the accuracy of the scale reading, and the pH response of the glass electrode over the range of the measurements are verified by checking the assembly with a series of standard buffer solutions.

7.1 *Assembly*—The assembly shall be judged to be performing satisfactorily if it furnishes, within acceptable limits of accuracy, the correct pH values for the standard buffer solutions listed in Table 2. When the electrodes are immersed in a buffer solution, the measured potential difference shall be substantially constant, and the cause of any instability shall be determined.

7.2 *Meter*—The meter shall be brought to electrical balance in accordance with the manufacturer's instructions. The performance shall then be tested by applying a known variable potential through a resistance of approximately 200 M Ω to the terminals of the meter, the high-resistance lead being connected to the terminal corresponding to the glass electrode. The source of potential may be a precision-type potentiometer with a range of 1100 mV or more and a limit of error not greater than 0.1 mV. The 200-M Ω resistor shall be properly shielded to avoid capacity pickup. Commencing with a value of zero, the

applied potential shall be increased in increments of 100 mV, and the readings of the dial of the meter at balance shall be noted. The process shall be extended to cover the entire range of the meter. In no case shall the difference between the applied voltage and that indicated by the meter differ by more than 1 mV per increment of applied voltage.

NOTE 6—If the cumulative error at the end of the scale exceeds ± 3 mV, a calibration curve for the meter shall be constructed and corrections applied to each measurement of electromotive force or pH. Differences of electromotive force (volts) are converted to corresponding differences of pH by multiplying by $F/(RT \ln 10)$ (Table X1.1). Inasmuch as the meter is made to read correctly at the pH of the standard, the calibration correction to be applied to a pH measurement is the difference between the scale corrections at the pH of the standard and that of the unknown, with due regard for sign.

7.3 *Glass Electrodes*—The difference of potential between the glass electrode and the standard hydrogen gas electrode shall be measured when both electrodes are immersed in the same portion of various buffer solutions over the pH range in which the glass electrode is to be used. For these comparisons the cell shall be placed in a water bath thermostatically controlled to $\pm 0.1^\circ\text{C}$ near 25°C . The solutions used for this test shall be those listed in Section 6. The standards of pH 9.18 and below (at 25°C) shall be used to test electrodes of the general-purpose type. The borax and carbonate standards shall be used to test the high-alkalinity type of electrode. These buffer solutions shall be supplemented by a 0.1 M carbonate-free solution of sodium hydroxide, the pH of which is approximately 12.8 at 25°C . The difference of potential between the general-purpose glass electrode and the hydrogen electrode shall be independent, within ± 1 mV, of pH changes in the range from 3.8 to 9.18 pH. The difference of potential between the hydrogen electrode and a glass electrode of the high-alkalinity type shall be the same, within + 3 mV, at pH 12.8 as at pH 9.18.

8. Calibration and Standardization

8.1 Turn on the instrument, allow to warm up thoroughly, and bring to electrical balance in accordance with the manufacturer's instructions. Wash the glass and reference electrodes and the sample cup three times with distilled water. Allow the water to drain from the electrodes, but the sample cup may be dried gently with clean absorbent tissue. Note the temperature of the test (unknown) solution and adjust the temperature dial of the meter to the proper setting.

8.2 Select two standard solutions (Note 7) to bracket the anticipated pH, if possible, and warm or cool these standards as necessary to match within 2°C the temperature of the unknown. Fill the sample cup with the first standard and immerse the electrodes. Set the dial of the meter to the pH(S) value of the standard at the appropriate temperature as read from Table 2 or interpolated in the data therein (see Note 3). Engage the operating button and rotate the standardizing knob or asymmetry potential knob until the meter is brought to balance. In direct-reading meters engage the operating-button, or turn the range switch to the proper position, and rotate the asymmetry potential knob until the reading of the dial corresponds to the known pH of the standardizing buffer solution. Fill the sample cup repeatedly with additional portions of the standard solution

until the instrument remains in balance with ± 0.02 pH unit for two successive portions without a change in the position of the asymmetry potential knob. If the temperature of the electrodes differs appreciably from that of the solutions, use several portions of solution and immerse the electrodes deeply to assure that both electrodes and standard are at the desired temperature. In order to reduce the effects of thermal and electrical hysteresis, keep the temperature of electrodes, standard solutions, and wash water as close to that of the unknowns as possible.

8.2.1 Wash the electrodes and sample cup three times with distilled water. Place the second standard in the sample cup, adjust the instrument to the new balance point, and read the pH from the dial. Do not change the setting of the asymmetry potential knob. Use additional portions of the second standard until successive readings of the pH agree within 0.02 unit. Judge the assembly to be operating satisfactorily if the reading obtained for the second standard agrees with the assigned pH(S) of that standard within 0.02 pH unit. When the meter is equipped with a slope control, use this control to correct small errors in the response of the glass electrode by adjusting the reading for the second standard to the known pH value. Discard used portions of the standard buffer solutions.

NOTE 7—Always calibrate the assembly with two buffer solutions to check the response of the electrode at different pH values and to detect a faulty glass electrode or incorrect temperature compensation. The presence of a faulty electrode is indicated by failure to obtain a reasonably correct value for the pH of the second standard solution when the meter has been standardized with the first. A cracked electrode will often yield pH values that are essentially the same for both standards. If an electrode gives an incorrect value or has a sluggish response, it may be dirty. Follow the manufacturer's instructions for cleaning.

8.3 If the anticipated pH of the test solution is less than 3.8, use the phthalate solution for the initial standardization and the citrate solution as the second standard. If the anticipated pH of the test solution is greater than 10.0, use an electrode designed for use at high alkalinities and observe the manufacturer's instructions. Use the borax solution for initial standardization of the assembly. The second standard shall be the carbonate solution. Judge the assembly to be operating satisfactorily if the reading obtained for the carbonate solution agrees with the assigned pH of this standard (Note 8) within 0.03 unit. When the meter is equipped with a slope control use this control to adjust the reading for the second standard (citrate solution or carbonate solution) to the known pH value.

NOTE 8—The change of pH(S) with change of temperature is large for the borax and carbonate standards. Hence, note the temperature of these standards to the nearest 1°C and use to obtain pH(S) by interpolation in the data of Table 1.

8.4 If only an occasional pH determination is made, standardize the assembly each time it is used. In a long series of measurements, supplement initial and final standardizations by a check at intervals of 1 h, or longer if little or no change is found between successive standardizations.

9. Procedure

9.1 pH of Test Solutions:

9.1.1 After the meter has been standardized with two standard solutions (Section 8), wash and dry the electrodes and

the sample cup as described in 8.1. Fill the cup with a portion of the test solution, and obtain a preliminary value for pH. In the case of well-buffered test solutions, one to three portions will usually be sufficient to yield pH values reproducible to ± 0.02 unit and that show drifts of less than ± 0.01 unit in 1 or 2 min.

9.1.2 Measure the pH of water samples and slightly buffered solutions that are in equilibrium with the air as described in 9.1, except measure the pH of successive portions of water or test solutions, with vigorous agitation, until the observed results for two successive portions agree within 0.1 unit. Six or more portions may be necessary. The flow cell may also be used (see 9.2). If the water sample or the slightly buffered test solution is not in equilibrium with the carbon dioxide of the atmosphere, measure with external electrodes in a wide-mouth flask that has been flushed with carbon dioxide-free air, and protect the contents of the flask from exposure to air during the measurement.

9.2 pH of Flowing Streams:

9.2.1 Flow cells and electrode units for immersion in flow channels are an important feature of industrial pH control. In conjunction with electronic recorders and recorder-controllers, they provide the continuous measurements necessary for fully automatic regulation of pH. The flow cell is particularly advantageous for the determination of the pH of water or of sparingly buffered solutions. Simple dip measurements without agitation are subject to appreciable errors due to inadequate washing of the electrodes, solubility of the glass, and absorption of carbon dioxide during the measurement. A rapid flow of solution past the electrode maintains a clean glass interface, retards the tendency for fine solids to collect at the surface, minimizes errors resulting from solubility of the glass, and protects the sample from atmospheric contaminants.

9.2.2 *Flow Cell*—The flow unit may be of metal, glass, rubber, or plastic. If metal pipe connections are employed, they shall all be of the same metal. The volume of the unit shall be small, to permit a high rate of flow. If the cell is not provided with a resistance thermometer for automatic temperature compensation (or if it is used in conjunction with a meter not equipped to utilize this feature), arrangements for monitoring the temperature of the solutions shall be provided. The unit and the leads shall be free from the effects of body capacitance.

9.2.3 *Standardization and pH Determination*—If the assembly is in continuous use, standardize it daily in accordance with the instructions given in Section 8. Use two standards in order to check the proper functioning of the electrodes. For a precision greater than ± 0.1 pH unit below pH 9, the temperature of the standard should be within 2°C of that of the flowing solution. For the measurement of pH, carefully observe the instructions furnished by the manufacturer of the meter or recorder.

9.2.4 *pH of Water and Slightly Buffered Solutions*—Maintain a flow rate sufficient to change the solution in the cell five times per minute. Do not read the pH of water or of a slightly buffered solution until the flow of water or test solution has been continued for at least 15 min following immersion of the electrodes in the standard buffer solution, or until a drift of less than 0.1 pH unit in 2 min is observed. If the pH of the

flowing solution is changing, the glass electrode measurement may lag considerably behind the true pH.

10. Report

10.1 Report the pH to 0.01 unit and the temperature of the test solution to the nearest 1°C.

11. Precision and Bias

11.1 The following criteria should be used for judging the acceptability of results obtained using separate glass and calomel electrodes (Notes 9 and 10):

11.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.006 pH unit at 106 dF. The 95 % limit for the difference between two such runs is 0.02 pH unit.

11.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), Formerly Called Repeatability*—The standard deviation of results, each the average of duplicates, obtained by the same analyst on different days, has been estimated to be 0.022 pH unit at 53 dF. The 95 % limit for the difference between two such averages is 0.06 pH unit.

11.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results, each the average of duplicates, obtained by analysts in different laboratories, has been estimated to be 0.040 pH unit at 12 dF. The 95 % limit for the difference between two such averages is 0.11 pH unit.

NOTE 9—The above precision estimates are based on an interlaboratory study performed in 1973 on four buffer solutions having pH values of approximately 3.7, 6.5, 8.2, and 8.4. Fourteen laboratories analyzed each solution in duplicate and replicated the analysis on another day for a total of 224 determinations. A variety of commercial meters equipped with glass and calomel electrodes were used in this study.⁵ Practice E 180 was

⁵ Supporting data are available from ASTM International Headquarters. Request RR: E15 - 1019.

used in developing these precision estimates.

11.2 *Bias*—The pH values of the buffer solutions, as determined using a hydrogen electrode at 25°C, are compared with the average values obtained using this test method in Table 2.

11.3 The following limited interlaboratory study by ten laboratories in one company suggests that the precision obtainable with new combination electrodes is comparable to that in the 1973 study using separate electrodes.

11.3.1 In 1994 a standard buffer solution of pH 4.63 was sent each laboratory which measured the pH once per day for three days. Each laboratory made the measurements using both a new and an old electrode. The results were analyzed using the techniques in Practice E 691. Because of the design, no estimates for repeatability are possible. The estimates for Laboratory Precision and Repeatability are given in Table 3.

NOTE 10—These estimates of precision apply to optimum conditions, namely for pH measurements of well-buffered aqueous solutions. The precision attainable in measurements of the pH of water and other poorly buffered solutions will, in general, be of a considerably lower order.

12. Keywords

12.1 aqueous solution; buffer; combination electrode; glass electrode; pH; pH meter; reference electrode

TABLE 3 Precision Using Combination Electrodes

	New Electrodes	Old Electrode
Laboratory precision		
Standard deviation	0.020	0.033
Degrees of freedom	20	18
95 % range	0.06	0.04
Reproducibility		
Standard deviation	0.037	0.033
Degrees of freedom	9	8
95 % range	0.10	0.09

APPENDIX

(Nonmandatory Information)

X1. MISCELLANEOUS NOTES

X1.1 The pH of an aqueous solution is derived from E_r , the electromotive force (emf) of the cell:

reference electrode || solution || glass electrode

where:

the double vertical line represents a liquid junction when the electrodes are immersed in the solution, and E_s , the electromotive force obtained when the electrodes are immersed in a standard solution, whose assigned pH is designated pH(S), by the following equation (Note X1.1):

$$\text{pH} = \text{pH}(S) + \frac{(E - E_1)F}{(RT \ln 10)} \quad (\text{X1.1})$$

where:

F = faraday, 96 487 C \times mol⁻¹,

R = gas constant, 8.314 33 J \times K⁻¹ \times mol⁻¹, and

T = absolute temperature, (t °C + 273.15).

NOTE X1.1—Values of $F/(RT \ln 10)$ are given in Table X1.1.

X1.2 For additional information on the concepts of pH and its measurement see the book by R. G. Bates.⁶

⁶ Bates, R.G., Determination of pH, Theory and Practice, Second Edition, John Wiley and Sons, New York, NY, 1973.

TABLE X1.1 Values of $F/(RT \ln 10)$

Temperature, °C	$F/(RT \ln 10), V^{-1}$
0	18.451
5	18.120
10	17.800
15	17.491
20	17.192
25	16.904
30	16.625
35	16.356
40	16.095
45	15.841
50	15.596
55	15.359
60	15.128

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