Designation: D4972 - 13

Standard Test Method for pH of Soils¹

This standard is issued under the fixed designation D4972; 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 measurement of the pH of soils for uses other than for corrosion testing. Such measurements are used in the agricultural, environmental, and natural resources fields. This measurement determines the degree of acidity or alkalinity in soil materials suspended in water and a $0.01\ M$ calcium chloride solution. Measurements in both liquids are necessary to fully define the soil's pH. This variable is useful in determining the solubility of soil minerals and the mobility of ions in the soil and assessing the viability of the soil-plant environment. A more detailed discussion of the usefulness of this parameter is not warranted here; however, it can be found in many discussions of the subject. A few such discussions are given as Refs $(1-6)^2$ at the end of the text.
- 1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.3 All measured and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.
- 1.4 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:³

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D1193 Specification for Reagent Water

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D6026 Practice for Using Significant Digits in Geotechnical Data

G51 Test Method for Measuring pH of Soil for Use in Corrosion Testing

3. Terminology

- 3.1 Definitions:
- 3.1.1 For common definitions of terms used in this standard, refer to Terminology D653.

4. Summary of Test Method

4.1 Measurement of the pH of soils in both suspensions of water and a calcium chloride solution are made with either a potentiometer using a pH sensitive electrode system (Method A), or pH sensitive paper (Method B). The potentiometer is calibrated with buffer solutions of known pH. The pH sensitive paper is a less accurate measurement and should only be used for a rough estimate of the soil pH. The electrode must be used for this measurement unless the pH sensitive paper is specified.

Note 1—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection and the like. Users of this standard are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

5. Significance and Use

- 5.1 The pH of the soil is a useful variable in determining the solubility of soil minerals and the mobility of ions in the soil and assessing the viability of the soil-plant environment.
- 5.2 pH measurements are made in both water and a calcium chloride solution because the calcium displaces some of the exchangeable aluminum. The low ionic strength counters the dilution effect on the exchange equilibrium by setting the salt concentration of the solution closer to that expected in the soil solution. The pH values obtained in the solution of calcium chloride are slightly lower than those measured in water due to the release of more aluminum ions which then hydrolyses.

¹ This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.22 on Soil as a Medium for Plant Growth.

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² The boldface numbers in parentheses refer to a list of references at the end of this standard.

³ 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.

Therefore, both measurements are required to fully define the character of the soil's pH.

- 5.3 For the purpose of this test method the test soil must be sieved through a 2-mm (No. 10) sieve. Measurements on soils or soil fractions having particle sizes larger than 2 mm by this test method may be invalid. If soil or soil fractions with particles larger than 2 mm are used, it must be stated in the report since the results may be significantly different.
- 5.4 All water used for this test method must be ASTM Type III or better. Type III water is defined by Specification D1193. It is prepared by distillation, ion exchange, reverse osmosis, or a combination thereof.

6. Interferences

- 6.1 This test method as measured by a pH probe has possible interferences due to a suspension effect or sedimentation potential. Users interested in a detailed discussion of the mechanism of this effect can find it in Refs (5) and (6).
- 6.2 This effect is the main reason Test Method G51 can not be used for general measurement of pH outside of that for corrosion analysis. Test Method G51 measures pH (an aqueous parameter) without adding any aqueous phase to the soil. This results in excessive soil particle-pH probe contact that overestimates the activity of the hydrogen ions in solution and is therefore unacceptable for general soil analysis.
- 6.3 The suspension effect can be mitigated by careful attention to 10.1.

7. Apparatus

- 7.1 *Method A, pH Meter*—Potentiometer equipped with an electrode system. Follow the manufacturer's instructions for the pH meter used. A silver/silver chloride electrode system or similar is also acceptable.
- 7.2 *Method B, pH Paper*—pH paper sensitive to a pH range from 1 to 12, with resolution to the nearest 0.2 pH unit.

8. Reagents

- 8.1 Purity of Reagents—Reagent grade chemicals should be used in all tests. Unless otherwise indicated, it is intended that all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.
- 8.2 Purity of Water—All water used for this test method must be ASTM Type III or better. Type III water is defined by Specification D1193. It is prepared by distillation, ion exchange, reverse osmosis, or a combination thereof.

8.3 Acid Potassium Phthalate Buffer Solution (0.05 M)—Dissolve 10.21 g (dried 1 hour at 105 \pm 5°C) of potassium phthalate in water and dilute to 1 L. The pH of this solution should be 4.0 at 20°C. Protect the solution against evaporation and against contamination with molds. Replace the solution when mold is noticed. The effect of temperature is as follows:

This illustrates that the pH of the solution does not change over the range in temperature from 5 to 37°C.

- 8.4 Calcium Chloride Stock Solution (1.0 M)—Dissolve 147 g of CaCl₂ * 2H₂O in water in a 1-L volumetric flask, cool, dilute to volume with water, and mix.
- 8.5 Calcium Chloride Solution (0.01 M)—Dilute 20.0 mL of stock 1.0 M CaCl₂ solution to 2 L with water. The pH of this solution should be between 5 and 7.
- 8.6 Phosphate Buffer Solution (0.025 M)—Dissolve 3.40 g of KH₂PO₄ and 3.55 g of KH₂HPO₄ in water and dilute to 1 L. Dry salts 2 h at 130°C before use. The pH of this solution should be 6.9 at 20°C. The effect of temperature is as follows:

°C	pH
0	7.0
10	6.9
20	6.9
30	6.8
40	6.8

8.7 Carbonate Buffer Solution (0.025 M)—Dissolve 2.10 g of NaHCO3 and 2.65 g of Na₂CO₃ in water and dilute to 1 L. Dry salts 2 hours at 130°C before use. The pH of this solution should be 10.1 at 20°C. The effect of temperature is as follows:

°C	рН
0	10.3
10	10.2
20	10.1
30	10.0
40	9.9

9. Calibration of pH Meter

9.1 Calibrate the pH meter using the buffer solutions in Section 8 or other NIST traceable purchased buffers. Select buffers so that the expected pH value of the tested soil is bracketed. Adjustment of the pH meter should follow the manufacturer's direction.

10. Procedure

- 10.1 When making measurements with the pH electrode, place the electrode into the partially settled suspension to mitigate the suspension effect.
- 10.2 For both methods, begin with an air dried soil that has been sieved through a 2-mm (No. 10) sieve to remove the coarser soil fraction. Air drying the soil is necessary to accomplish sieving and to control the amount of water present at the time of measurement.

Note 2—Some laboratories have reported that 10~ml of water and 10~g of soil is insufficient material to fully submerge the pH electrode on their equipment. In these cases, a larger sample such as 40~ml of water and 40~g of soil is permitted

10.3 pH in Distilled Water—For both methods, weigh out approximately 10 g of air dried soil. Place the soil into a glass

⁴ 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.

container and add approximately 10 mL of water. Mix thoroughly and let stand for 1 h.

- 10.4 Method A-Read pH on pH meter.
- 10.5 Method B-Read pH on pH paper.
- 10.6 pH in 0.01 M Calcium Chloride Solution—For both methods weigh out approximately 10 g of air dried soil. Place the soil into a glass container and add approximately 10 mL of the 0.01 M CaCl₂ solution. Mix thoroughly and let stand for 1 h.
 - 10.7 Method A—Read pH on pH meter.
 - 10.8 Method B-Read pH on pH paper.
- 10.9 The mixture should be at approximately room temperature (15 to 25° C) at the time of pH measurement.

11. Report:Test Data Sheet(s)/Form(s)

- 11.1 Record as a minimum the following general information:
- 11.1.1 Sample/specimen identifying information, such as Project No., Boring No., Sample No., Depth, etc.
- 11.1.2 Any special selection and preparation process, such as removal of gravel or other materials.
 - 11.1.3 Technician name, method used and date.
 - 11.2 Record as a minimum the following test information:
 - 11.2.1 Report the pH of the soil to the first decimal place
- 11.2.2 Specify which of the pH measurements is in water and which is in the calcium chloride solution.
- 11.2.3 Specify whether the determinations were made with Method A or Method B.
- 11.2.4 Record if size fractions other than sieved through the 2 mm (No. 10) sieve are used, it must be stated in the report since the results may be significantly different.

12. Precision and Bias

12.1 Precision:

- 12.1.1 Within-Laboratory Precision—The within laboratory standard deviations for Method A are 0.031 (pH units) for the water mixture and 0.139⁵ (pH units) for the calcium chloride mixture. Therefore, results of two properly conducted tests in the same or different laboratories should not differ by more than 0.065⁵ (pH units) for the water mixture and 0.389 pH units for the calcium chloride mixture. The within-laboratory standard deviations for Method B are 0.189 (pH units) for the water mixture and 0.212⁵ (pH units) for the calcium chloride mixture. Therefore, results of two properly conducted tests in the same or different laboratories should not differ by more than 0.53⁵ (pH units) for the water mixture and 0.60 pH units for the calcium chloride mixture.
- 12.1.1.1 The precision of Method A presented was determined by the National Technical Center of the United States Department of Agriculture. In their evaluation they used 174 replicates for the water mixture and 32 replicates in testing the calcium chloride mixture.
- 12.1.1.2 The precision of Method B presented was determined by the United States Army Environmental Hygiene Agency. In their evaluation they used 25 replicates in testing each mixture.
- 12.1.2 Between-Laboratory Precision—The between-laboratory standard deviation has not been determined for either method. Subcommittee D18.22 is actively seeking data to evaluate the between laboratory precision of this test method.
- 12.2 *Bias*—There is no accepted reference value for this test method; therefore, bias cannot be determined.

13. Keywords

13.1 acidity; alkalinity; pH; reaction; soil

REFERENCES

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- (5) Hunter, R. J., Zeta Potential in Colloid Science, Academic, New York, NY. 1981.
- (6) Perrin, D. D., and Dempsey, B., Buffers for pH and Metal Ion Control, Chapman and Hall, London, United Kindom, 1974.

⁵ These data satisfy the 1S and D2S requirements outlined in Practice C670.

SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D4972 – 01 (2007)) that may impact the use of this standard. (Approved Nov. 1, 2013.)

(1) Revised Sections 1, 8, 9, 11

(2) Added Note 1 and Note 2.

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