This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.



Designation: D4972 – 19

Standard Test Methods 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 These test methods cover the measurement of the pH of soils that will pass the 2.00 mm (No. 10) sieve. Such measurements are used in the agricultural, environmental, geotechnical, 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 given in Refs $(1-6)^2$.

1.2 Two methods for measuring the pH of soils are provided. The method to be used shall be specified by the requesting authority. When no method is specified, Method A shall be used. The pH is determined in test water and a calcium chloride solution for both methods.

1.2.1 *Method* A—The pH is measured using a potentiometer having a pH sensitive electrode system. This method can be used for any application and must be used when the application warrants a higher level of resolution.

1.2.2 *Method* B—The pH is measured using pH sensitive paper. This method can be used for any application, however, because paper typically has a lower resolution, it provides an approximate estimate of the pH of the soil and should not be used when the application requires a higher level of resolution (Note 1).

Note 1—For example, paper with a sensitivity to the nearest 1 pH unit placed into a buffer solution of 4 should indicate a pH of 4, however, it would not indicate if the pH is 4.449 or 3.449.

1.3 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 All measured and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.4.1 The procedures used to specify how data are collected/ recorded and calculated in the standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of these test methods to consider significant digits used in analysis methods for engineering data.

1.5 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.6 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

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
- D2487 Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)
- D2488 Practice for Description and Identification of Soils (Visual-Manual Procedures)
- D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as

¹ These test methods are under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.22 on Media for Plant Growth.

Current edition approved May 1, 2019. Published May 2019. Originally approved in 1989. Last previous edition approved in 2018 as D4972 – 18. DOI: 10.1520/D4972-19.

 $^{^{2}\,\}mathrm{The}$ 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.

Used in Engineering Design and Construction

- D4220/D4220M Practices for Preserving and Transporting Soil Samples
- D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing
- D6026 Practice for Using Significant Digits in Geotechnical Data
- D6913 Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis
- G51 Test Method for Measuring pH of Soil for Use in Corrosion Testing

3. Terminology

3.1 Definitions:

3.1.1 For definitions of common technical terms used in this standard, refer to Terminology D653.

4. Summary of Test Method

4.1 This test method is used to determine the pH of a soil. Two similar test specimens are obtained from a representative sample that has been air-dried and screened over a 2.00 mm (No. 10) sieve. From the material passing the 2.00 mm (No. 10) sieve, one specimen is mixed with test water and one is mixed with a calcium chloride solution. They are then thoroughly stirred and both are allowed to stand for one hour prior to testing using Method A or B.

5. Significance and Use

5.1 The pH of the soil is a useful variable in determining the solubility of soil minerals, the mobility of ions in the soil, and assessing the viability of the soil-plant environment.

5.2 pH measurements are made in both test 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. Therefore, both measurements are needed to fully define the character of the soil's pH.

5.3 For the purpose of these test methods, the test specimens are sieved through a 2.00 mm (No. 10) sieve. Measurements on soils or soil fractions having particle sizes larger than 2.0 mm by these test methods may be invalid. If soil or soil fractions with particles larger than 2.0 mm are used, it must be stated in the report since the results may be significantly different.

Note 2—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/etc. 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.

6. Interferences

6.1 When using Method A, a pH probe has possible interferences due to a suspension effect or sedimentation potential. Refs (5) and (6) provide a more detailed discussion of the effect of this mechanism.

6.2 This effect is the main reason Test Method G51 cannot 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 lack of aqueous phase 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 inserting the probe into the aqueous phase of the slurry. The aqueous phase consists of the partially settled suspension.

7. Apparatus

7.1 Use one of the following devices to measure the pH.

7.1.1 *pH Meter*—Potentiometer equipped with an electrode system with a readability to the nearest 0.1 pH unit and an accuracy of ± 0.1 pH unit or better. Follow the manufacturer's instructions for the pH meter used. A silver/silver chloride electrode system or similar is also acceptable.

7.1.2 *pH Paper*—pH-sensitive paper having a typical range from 1 to 12 with sensitivity to the nearest 0.5 pH unit or better. The range of the paper may vary based on the typical pH of the materials being tested. It is acceptable to use paper ranging between other values as long as the range is large enough to bracket the expected pH result.

7.2 *Balance*—Balances shall conform to the requirements of Specification D4753.

7.2.1 To determine the mass of the specimen, the balance shall have readability without estimation of 0.1 g or better. The capacity of this balance will need to exceed the mass of the container plus soil. In general, a balance with a minimum capacity of 300 g is sufficient.

7.3 *Separation Sieve*—2.00 mm (No. 10) sieve used to separate the coarse material from the representative sample. This sieve is subjected to rough operation and shall not be used for quantitative testing.

7.4 *Specimen-Mixing Container*—A glass beaker or equivalent inert container with enough capacity to hold the specimen and the test water. Usually, a 100 mL to 250 mL glass beaker is large enough.

7.5 Drying Oven (Optional)—Vented, thermostatically controlled oven capable of maintaining uniform temperatures of $105 \pm 5^{\circ}$ C and $130 \pm 5^{\circ}$ C as needed throughout the drying chamber. These requirements typically require the use of a forced-draft oven.

7.6 Volumetric Flask or Cylinder—Two, glass or plastic; one each with capacities of 1 L and 2 L. Required for making calcium chloride hydrate solutions, otherwise, they are optional.

7.7 *Thermometric Device*—A thermometric device capable of measuring the temperature range within which the test is being performed readable to 0.5° C or better and having an accuracy of at least $\pm 0.5^{\circ}$ C.

7.8 *Miscellaneous Items*—Items such as a wash/rinse bottle (squirt bottle), rubber scraper, spatula, plastic or glass containers to hold reagents, and stirring rod may be useful.

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.⁴ 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 *Test Water*—Type III water in accordance with Specification D1193 or distilled/deionized water with a pH between 6.5 and 7.5.

8.3 *Buffer Solutions*—Buffer solutions are commercially available and providing they meet the purity of reagents as described above, they are an acceptable alternative to making buffer solutions. The following NIST traceable buffer solutions are typically needed: pH of 4.0, 7.0, and 10.0. When making buffer solutions, refer to Annex A1 for the necessary chemicals and instructions.

8.4 Calcium Chloride Hydrate Stock Solution (CaCl₂· $2H_2O$)—Using the 1 L volumetric flask or cylinder, dissolve 147 g of CaCl₂· $2H_2O$ in test water and allow it to come to room temperature. Then, dilute by adding test water to the 1 L mark on the volumetric flask or cylinder. Mix the solution thoroughly. The molarity of this solution is 1.0 M (Note 3).

8.5 Calcium Chloride Hydrate Solution (CaCl₂• $2H_2O$) Using the 2 L volumetric flask or cylinder, add 20.0 mL of the calcium chloride hydrate stock solution and test water by adding test water to the 2 L mark on the volumetric flask or cylinder. Mix the solution thoroughly. The pH of this solution should be between 5 and 7 and the molarity is 0.01 M.

Note 3—It is acceptable to purchase the calcium chloride solutions provided they have the same molarity and pH as described above.

9. Hazards

9.1 These solutions may cause eye and skin irritation. Care should be taken to avoid contact with skin and eyes and solutions should not be ingested. The use of latex or rubber gloves, and possibly safety glasses, when working with the solutions is recommended. Wipe up spills immediately. Rinse skin coming into contact with the solutions with water quickly.

10. Sampling and Test Specimens

10.1 *General*—These test methods do not address, in any detail, procurement of the sample. It is assumed the sample is obtained using appropriate methods and is representative of the soil under evaluation. However, the testing agency shall preserve all samples in accordance with Practice D4220/ D4220M, Group B, except if the as-received sample does not meet those requirements. In which case, the water content of the material does not have to be maintained.

10.2 *Sampling*—Obtain a representative sample from the bulk sample. Follow guidance given in Test Method D6913, Annex A2, for specimen splitting/reducing. The moist mass of the representative sample must be large enough such that, after air drying and screening over the 2.00 mm (No. 10) sieve, the air-dried mass will be large enough to yield two test specimens.

10.2.1 Place the representative sample on a tray, pan, or flat surface to facilitate uniform air-drying. Be careful not to lose fines during this process. After drying, if the sample appears to have particles larger than 2.0 mm, the sample must be screened over the 2.00 mm (No. 10) sieve. If there are no coarse particles present, screening is not necessary. See 5.3 for soils having coarse particles and tested using these methods.

10.2.2 Air drying is necessary to accomplish sieving and to control the amount of water present at the time of measurement.

10.3 Test Specimens—Since the soil is in the air-dried state, segregation and loss of fines are common problems and care should be taken to make sure the test specimens accurately represent the sample. From the soil passing the 2.00 mm (No. 10) sieve, obtain two similar specimens for testing. Typically, a mass of 10 ± 0.1 g is adequate, however, 20 ± 0.1 g or 40 ± 0.1 g specimens may be necessary to allow for the pH electrode to be fully submerged.

10.3.1 For both methods, determine and record the mass of the test specimens to the nearest 0.1 g.

11. Standardization

11.1 Before each use standardize the pH meter using the buffer solutions in 8.3 or Annex A1 following the manufacturer's instructions of the meter. Select buffers so that the expected pH value of the tested soil is bracketed. Typically buffers of 4, 7, and 10 meet this requirement for most soils; however, in some applications buffers with higher or lower pH values are needed. Follow the manufacturer's instructions to adjust the pH meter accordingly. Record the date, time, and name or initials of person completing the standardization. If other data in association with the standardization is determined, record this information (Note 4).

11.1.1 After every 10 specimens or at the end of testing, whichever occurs first, verify the pH meter's standardization by inserting the probe into the pH 7.0 buffer solution. The value should be between 6.9 and 7.1. Read and record the pH value to the nearest 0.1 pH unit. If the value falls outside of this range, the pH meter must be re-standardized and the previous specimens must be retested to verify the results.

11.2 Check the pH sensitive paper by inserting a small portion of the paper into the pH 4.0, 7.0, and 10.0 buffer

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.