FLORIDA DEPARTMENT OF TRANSPORTATION
State Materials Office
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Florida Method of Test for pH of Soil and Water

Designation: FM 5-550

1. SCOPE

This method covers the determination of pH in soil and water using a pH/mV meter with automatic temperature compensation (ATC) and a combination electrode that includes a silver/silver chloride reference electrode, a glass bulb indicating electrode, and a thermocouple. This method can also be used for the determination of pH in coarse aggregate.

2. APPARATUS

2.1. pH Meter and Electrode System: Portable or benchtop pH/mV meter with automatic temperature compensation (ATC), accuracies for mV: the greater of ± 0.2 mV or ± 0.05%, for pH: ± 0.002 pH units, and for temperature: ± 0.3°C, or better, and the ability to display calibration data including % slope and offset. The electrode shall be a refillable double-junction 3-in-1 combination electrode that includes a silver/silver chloride ($Ag/AgCl_2$) reference electrode, glass bulb indicating electrode, and thermocouple with pH/ATC connectors that match the meter inputs, and capable of measuring pH from 0 to 14 at temperatures between 0°C and 100°C with a minimum resolution of 0.01 pH units (as examples, Fisher Scientific pH/mV meter catalog #13-636-AB150 and Fisherbrand Accumet electrode catalog # 13-620-631.

2.2. Analytical Balance: An analytical balance with a capacity of 2,000 g or greater and a resolution of 0.1 g or better.

2.3. 100 and 250-mL beakers, wash bottle, glass stirring rods, 100-mL scoop, and delicate glass cleaning tissues.

3. REAGENTS

3.1. Standard buffered solutions for pH 4, 7, and 10.

3.2. Electrode filling solution and electrode storage solution (refer to the manufacturer's instruction manual).

3.3. Distilled (or higher purity) water with a minimum resistivity of 200,000 ohm-cm (**Note 1**).

Note 1: Distilled (or higher purity) water stored in containers that are not airtight will absorb ions from acidic and basic gases in the atmosphere resulting in lower resistivity of the water over time.

4. SAMPLES

4.1. Soil Sampling: Every effort should be made to obtain a soil sample that is representative of the bulk material. Use clean tools for collecting samples. Excessive moisture should be avoided by sampling from areas that have been allowed to gravity drain. If the soil sample has excess free moisture, place 2.2 lb (1 kg) of the soil on top of a suitable sieve and cover with plastic. Allow the sample to drain for a minimum of one hour. This step may be performed in the lab prior to testing.

4.2. If the soil sample is obtained from a heap that has been sitting for a long time, take the sample from a depth below the weathered surface. If sampling from ground level, remove the top 12 in. (30 cm) to eliminate vegetation and debris before sampling. The soil sample may be taken from underneath standing water, but free-standing water should not be included with the sample. Soil samples should be placed in plastic (watertight) bags. The bag should be squeezed down snugly around the sample and sealed tightly to minimize contact with air.

4.3. Water Sampling: Water samples should be obtained from the main channel of rivers and streams. Sampling from other bodies of water such as lakes or ponds should occur in areas conducive to collecting a representative sample. Care should be observed to avoid sampling from stagnant or pooled water unless a structure will be placed in such an area.

4.4. The water sample container shall be clean, at least 1 qt (1 L) in size and be either glass or plastic with an airtight lid. Rinse the container several times with the water to be collected. When possible, submerge the sample container below the surface of the water to avoid introduction of floating debris such as leaves, sticks, foam, or trash. Fill the sample container to the top to eliminate introducing air into the sample and tightly seal the lid.

4.5. Transporting Samples: Maintain test samples in a cool dark area after sampling and during transport to the test facility.

4.6. Storing Samples: Store water and soil samples at or below 39°F (4°C). Care should be taken to prevent freezing of the samples. Analyze samples within seven (7) days of collection.

5. SAMPLE PREPARATION

5.1. Preparation of Water:

Allow test sample to reach room temperature.

5.2. Preparation of Soil:

5.2.1. Loose Granular Soils: Spread the sample in a thin layer on a clean tray and dry under ambient conditions until a constant mass is achieved, or dry in an oven at no higher than 140°F (60°C) for four hours or until a constant mass is achieved. Sieve through a No. 10 mesh (2 mm) sieve. Split the sample per **AASHTO R 76** to obtain 1,300 g ± 5%.

5.2.2. Muck and Soils with Clay: Spread the sample in a thin layer on a clean tray and dry under ambient conditions until a constant mass is achieved, or dry in an oven at no higher than 140°F (60°C) for four hours or until a constant mass is achieved. Using a rawhide mallet or other suitable device, pulverize the sample and sieve through a No. 10 mesh (2 mm) sieve. Split the sample per **AASHTO R 76** to obtain 1,300 g ± 5%.

5.2.3. After sieving is complete, allow the soil sample and distilled water to reach room temperature. Place 100 g of sample in a 250-mL beaker and then add 100 mL of room temperature distilled water. Break up any clumps of soil. Thoroughly stir the soil and water mixture 3 times for 20 seconds each time, waiting 10 minutes between each stirring event. (30-minutes total time).

5.3. Preparation of Coarse Aggregate:

Split the sample per **AASHTO R 76** to obtain 1,300 g ± 5% and place aggregate in a suitable container. Add 1,200 mL of distilled water, cover, and let stand for 24 hours at room temperature. Collect the leachate using clean equipment to avoid contamination.

6. TEST PROCEDURE

6.1. Calibration of pH Meter and Electrode System: Pour 50 mL of pH 4, pH 7, and pH 10 standard buffer solutions into separate 100-mL beakers and allow solutions to reach room temperature prior to calibration (**Note 2**). Remove pH electrode from storage solution, rejuvenate following the manufacturer's instructions. Rinse with distilled water and blot or dab dry with delicate glass tissue, make certain the fill hole is uncovered, then place in pH 7 buffer solution for 10 minutes prior to calibration. Calibrate the pH/mV meter and 3-in-1 combination electrode with three standard buffers according to the directions

in the manufacturer's instruction manual. Check that the pH for each of the buffer solutions is within 0.05 pH units for the buffer pH at the test temperature. The pH versus temperature tables is typically tabulated on the buffer solution bottles. If the measured pH is outside 0.05 pH units of the buffer pH at test temperature, take corrective steps and re-calibrate the electrode (**Note 3**). When the calibration is complete, record the calibration slope in %, the offset in mV, and the buffer temperature in °C (**Note 4**). The slope must be within 95% to 102%, the offset within ± 10 mV, and the buffer temperatures within 25 ± 5°C; if not, follow steps in the instruction manual to clean, fill, re-calibrate, or replace the electrode so that the slope, offset, and temperatures are within these ranges.

Note 2: Always use fresh buffers for electrode calibration (less than seven days in an airtight container).

Note 3: If the calibration check fails, suspect a partially clogged liquid junction on the electrode. Add electrode filling solution to flush the liquid junction, and then re-check calibration.

Note 4: For some meters, the slope and offset are presented separately for two buffers (for example, pH 4 and pH 7, or pH 7 and pH 10). Record the slope and offset for the range that includes the sample pH.

6.2. Care of Electrode: Check the electrode filling solution and keep filled to the fill hole. Between any two pH measurements, rinse electrode with distilled water and blot or dab the electrode tip dry with a delicate glass tissue (**Note 5**).

Note 5: With some 3-in-1 combination electrodes, the narrow space between the glass bulb and the temperature sensor traps liquid, whether buffer, sample, or distilled water. This trapped liquid can be transferred into the sample and may affect the pH reading.

6.3. Determination of pH: Insert the pH electrode in the buffer, water, or soil sample such that the portion of the electrode that contains the glass bulb, thermocouple, and liquid junction is submersed, typically about ½" (1.3 cm) of the electrode tip. Do not immerse the electrode to a depth greater than the electrode's internal reference solution, as the sample may penetrate and contaminate the reference solution. Very gently stir the electrode in the sample (alternately, gently move the sample beaker beneath the electrode, or use a magnetic stir plate and stir bar) to assure a pH representative of the bulk solution (**Note 6**). Wait until the meter's stable reading indicator stays on for 10 seconds or longer (**Note 7**). Record the pH to the nearest hundredth pH units and sample temperature to the nearest tenth °C, for example, pH 7.00 @ 25.1°C. Rinse the electrode with distilled water between all measurements.

After every other sample measurement, return the pH electrode to the pH 7 buffer and check that the electrode is still within calibration.

Note 6: Ideally, once the pH reading has stabilized, the reading will not change whether gently stirred or unstirred. For low ionic strength solutions, however, the pH may not stabilize if stirred. If the pH reading does not stabilize after 5 min with gentle stirring, obtain and record a stable reading with no stirring.

Note 7: Disable the "hold" feature of the pH meter.

6.4. Storage of Electrode (**Note 8**): Store the pH electrode overnight or longer in its storage bottle either in the electrode storage solution with the fill hole open or in pH 4 or 7 buffer with the fill hole closed. Do not store in distilled water as this will have an adverse effect on electrode performance. Tap water or standard buffer solutions are appropriate for short-term electrode storage.

Note 8: Store the electrode in accordance with manufacturer's instructions if different from this section.

7. REPORT

7.1. The following information should be reported.

7.1.1 pH of the sample.

7.1.2 Temperature of the sample when tested in °C.

8. PRECISION AND BIAS

8.1. Precision: For two test materials, multi-laboratory standard deviations of a single test result were dependent on soil pH (**Table 1**). For materials of similar pH, the results of two properly conducted tests in different laboratories on the same material are not expected to differ by more than the pH units shown in column labeled "Acceptable Range of Two Results."

8.2. Bias: Single-operator, single laboratory bias for this method using an Accumet meter/electrode AB150/13-620-631 was evaluated at 20° C with repeated measurements of each six aqueous standards ranging from pH 5.00 to pH 9.00. The average bias was -0.01 pH units.

Table 1 Multi-Laboratory Precision for FM 5-550 pH

Material	Average, pH units	Standard Deviation, pH units	Acceptable Range of Two Results
Sand (A-3)	7.76	0.32	0.91
Sand (A-3)	5.11	0.15	0.42