

# Florida Method of Test For pH of Soil and Water

Designation: FM 5-550

## 1. SCOPE

- 1.1 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) and accuracies for mV: the greater of  $\pm 0.2$  mV or  $\pm 0.05\%$ , for pH:  $\pm 0.002$  pH units, and for temperature:  $\pm 0.3$  °C, or better, a display of calibration data including % slope and offset; and a refillable double-junction 3-in-1 combination electrode that includes a silver/silver chloride ( $\text{Ag}/\text{AgCl}_2$ ) reference electrode, glass bulb indicating electrode, and thermocouple with pH/ATC connectors that match the meter inputs, and capable of measuring 0 to 14 pH from -5 °C to 100 °C with a minimum resolution of 0.01 pH units (for example, Fisher Scientific pH/mV meter kit catalog #13-636-AB150).
- 2.2 Analytical Balance: An analytical balance with a capacity of 2,000 g or greater and a resolution of 0.01 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 electrode manual).
- 3.3 Deionized or distilled water (dilution water) with a resistivity equal or greater than 200,000 ohm-cm.

## 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 gathering samples. Excessive moisture should be avoided by sampling from an area that has been allowed to gravity drain for a short time. If the soil sample has excess free moisture, place 2.4 pounds (1,075 g)  $\pm$  5% 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.

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 where the moisture content appears to have stabilized. Avoid taking the sample from near a weathered soil surface. If sampling from ground level, remove top 12" (30 cm) to eliminate vegetation and debris before sampling. The soil sample may be taken from underneath standing water, but excess water should not be included with the sample. Soil samples should be placed in plastic or plastic-lined bags. Squeeze the bag down snugly around the sample and seal tightly to minimize contact with air.

- 4.2 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 be obtained from areas conducive to the capture of representative samples. Care should be observed not to sample from stagnant or pooled water, unless a structure will be placed in such an area. Sample the water just below the surface to alleviate introduction of floating debris, i.e. leaves, sticks, foam or trash. Fill the sample container to the top to eliminate introducing air into the sample and tightly seal the lid. The sample container shall be clean, at least 1 quart (1 liter) in size, and be either glass or plastic with an airtight lid. When possible, submerge the sample container below the surface of the water to completely fill and secure lid underwater.
- 4.3 Transporting the samples: Maintain test samples in a cool dark area after sampling and during transport to the test facility.
- 4.4 Storing Samples: If samples need to be stored, 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:

Allow the soil sample and dilution water to reach room temperature. Place 100 g of sample in a 250 ml beaker and then add 100 ml of dilution water. Break up any clumps of soil. Thoroughly stir the soil and water mixture for 20 seconds every 10 minutes over a 30-minute period.

### 5.3 Preparation of Coarse Aggregate:

Split the sample per AASHTO R 76 to obtain a 1,300 g  $\pm$  5% and place aggregate in a suitable container. Add 1,200 mL of dilution 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 1). Remove pH electrode from storage solution, rinse with dilution water and blot or dab dry with delicate glass tissue, uncover the fill hole, 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. When the calibration is complete, record the calibration slope in %, offset in mV, and buffer temperature in °C (Note 2). The slope must be within 95% to 102%, the offset within  $\pm$  10 mV, and the buffer temperatures within  $25 \pm 5$  °C; if otherwise, follow the instruction manual to clean, fill, equilibrate, re-calibrate, re-activate, or replace the electrode so that the slope, offset, and temperatures are within these values.
- 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 dilution water and blot or dab the electrode tip dry with a delicate glass tissue (Note 3).
- 6.3 Check of Calibration: Check the calibration by measuring the pH of each of the standard buffers. The measured pH should be within 0.05 pH units of the buffer pH at the test temperature. Buffer pH versus test temperature data are typically tabulated on the buffer bottle. 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 4).
- 6.4 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  $\frac{1}{2}$ " (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 into and contaminate the reference solution. Very gently stir the electrode in the sample (or alternately, gently move the sample beaker beneath the electrode) to assure a pH representative of the bulk solution (Note 5). Wait until the meter's stable reading indicator stays on for 10 seconds or longer (Note 6). 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. After the second reading, return the pH electrode to the pH 7 buffer and check that the electrode is still within calibration.

- 6.5 Storage of Electrode (Note 7): 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 dilution 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 1: Always use fresh buffers for electrode calibration (less than seven (7) days in an airtight container).

Note 2: 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.

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

Note 4: If after a sample pH measurement 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 5: 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 6: Disable the "hold" feature of the pH meter.

Note 7: Store the electrode in accordance with manufacturer's instructions if different from this section.

## 7. PRECISION AND BIAS

- 7.1. 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.

- 7.2. Reproducibility: 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.”

**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