Applied Microbiology
&
Biotechnology Laboratory

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Standard Operating Procedure

AMBL-205-C

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Revised:	
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Laboratory Determination of Soil pH by Extraction with a Calcium Chloride Ionic Strength-Stabilizing Solution

METHOD SUMMARY

This SOP describes a procedure for determining the pH of a soil using a soil-calcium chloride mixture and measuring the pH of the solution. This method is adapted from Section 4.3 (USDA-NRCS, 2014) and from "Determination of pH of Soils by Different Methods: Collaborative Study (Kalra, 1995).

ENVIRONMENTAL HEALTH AND SAFETY

Hazards Assessment: This method involves the use of pH buffer solutions having pH values of 4.0 and 10.0, and the handling of calcium chloride. Assess environmental soils samples for potential hazards before analysis, otherwise soil samples are assumed to contain unknown chemical or biological hazards. In any case, handle soil samples according to safe practice.

Buffers used for standardizing the pH meter are not considered to contain hazardous materials; however, they may be considered either acidic (pH 4.0) or alkaline (pH 10.0).

Skin Contact: Repeated exposure may dry and crack skin.

Eye Contact: Contact can cause severe irritation, redness, swelling and blurred vision.

Ingestion: Although, unlikely to occur during the performance of this method, ingestion can cause nausea.

Inhalation: Exposure by inhalation is not expected.

Calcium chloride is classified as an eye irritant and should be handled to avoid the formation of dusts and aerosols.

Skin Contact: Avoid skin contact with proper use of gloves. Dispose of gloves and thoroughly wash hands after handling.

Eye Contact: Can cause serious irritation. Wear eye protection. Do not rub your eyes.

Ingestion: Although, unlikely to occur during the performance of this method, ingestion can a burning sensation, nausea and vomiting.

Inhalation: Exposure by inhalation of dust or aerosols can cause respiratory tract irritation and coughing.

Safety Equipment and Engineering Controls: This method requires that an eye wash station and a shower be located nearby.

Personal Protective Equipment (PPE): This method requires the use of the following PPE.

Gloves (nitrile)

Safety goggles or glasses

Laboratory coat

Analysis-derived Wastes and Disposal:

Waste Generated	Hazardous (Y / N)	Disposal
This procedure generates small volumes of pH buffer solution.	N	May be disposed in a sink.
This procedure generates a saturated soil of 50 g (dry mass).	N	Disposal in a waste container destined for the landfill is considered acceptable.
This procedure may generate a saturated soil of 50 g (dry mass), having a known or suspected hazardous substance.	Y	If the soil contains a known or suspected hazardous substance, dry the soil to the extent possible and dispose of properly as hazardous waste.
This procedure generates about 50 mL of 0.01 M calcium chloride solution mixed with soul.	N (or Y)	May be disposed in the sink unless the soil has a known or suspected hazardous substance that is also soluble in water, in which case the solution is dispose as a hazardous waste.

METHOD DESCRIPTION

1.0 Introduction and Applicability

Soil pH is determined by suspending the soil in a 0.01 M calcium chloride solution and then after a designated amount of time the solution is separated from the soil and the pH of the solution is measured.

This method is intended for use in laboratory and is applicable to natural and contaminated soils having no available free water. The variability of a soil's pH due to seasonal or other effects is largely removed by this method and considered most applicable for the diagnostic analysis of soils from North America (3).

This method is not intended for saturated sediments or muds, liquid or semi-liquid waste samples.

2.0 Apparatus

a. pH meter, and a combination pH electrode with temperature compensation
b. Magnetic stirrer and micro stir bar
c. Glass stirring rod
d. Beaker, 100-mL
e. Small scoop or spoon
f. Analytical balance capable of weighing to the nearest 0.1 g or less.
g. Analytical balance capable of weighing to the nearest 0.0001 g or less.
h. Sieve, ¼ inch mesh (6.35 mm)
i. Mortar and pestle, ceramic
j. Centrifuge tubes, 50-mL polypropylene
k. Centrifuge, operated at 2500 rpm

3.0 Reagents

a. Buffer solutions suitably prepared with known pH value, 4.0, 7.0, and 10.0, or commercially purchased solutions.
b. Electrode storage solution prepared as a saturated potassium chloride for a combination electrode, or the solution recommended by the electrode manufacturer.
e. Calcium chloride ($CaCl_2$), calcium chloride dihydrate ($CaCl_2$-$2H_2O$) or calcium chloride hexahydrate ($CaCl_2$-$6H_2O$).
d. Reagent water, distilled water

4.0 Procedure

a. Read AMBL SOP 205A and be certain that you have calibrated the pH meter according to manufacturer's instructions and that you know how to perform a pH measurement. All measurements should be done with buffer and sample solutions at room temperature.

b. Prepare the 0.01 M CaCl2 solution using one of the following three methods of preparation.

   1. Weigh and dissolve 1.1098 g of $CaCl_2$ in 800 mL of reagent water. Dilute to 1.0 liter with reagent water.
   2. Weigh and dissolve 1.4701 g of $CaCl_2$-$2H_2O$ in 800 mL of reagent water. Dilute to 1.0 liter with reagent water.
   3. Weigh and dissolve 2.1908 g of $CaCl_2$-$6H_2O$ in 800 mL of reagent water. Dilute to 1.0 liter with reagent water.

   Store in a tightly capped, amber glass container in a cool dark location.

c. Prepare the soil sample (air-dried) by sieving to separate and remove rocks larger than 0.25 inch (6.35 mm).

d. Remove and either by hand (with gloves) or if necessary with a mortar and pestle, break-up aggregated soil particles larger than 0.25 inch (6.35 mm) and re-sieve.

e. Thoroughly homogenize the soil passing the 0.25 inch (6.35 mm) sieve.

f. Scoop or spoon 50 ± 0.1 g of the homogenized soil sample into a 100-mL beaker and then add 50 ± 0.1 g of 0.01 M $CaCl_2$ solution.

g. Using a clean glass rod, stir the soil-0.01 M $CaCl_2$ solution mixture until the soil is fully suspended and continue to stir for another 60 seconds. Allow the mixture to settle for 10 minutes. Repeat this stirring and settling cycle three more times.

h. Transfer the supernatant into a 50-mL centrifuge tube, close the cap tightly and centrifuge at 2,500 rpm for 15 minutes. Alternately, allow the suspension to settle for 1 hour after it has been stirred for the last time in 4.0g above.

i. Without re-suspending the soil, transfer the centrate (or supernatant) into a second 50-mL tube. This may be done by either carefully pouring from one tube into the second tube, or by using a disposable pasteur pipet.

j. Place a micro sir bar into the tube and secure the tube on the sir plate.

k. While stirring, insert the pH probe and measure the pH according to SOP 205A.

l. Remove the electrode from the sample, rinse with deionized or distilled water, and dry with a soft tissue.

l. When finished, return the pH electrode to the storage solution bottle by twisting the bottle counterclockwise and gently moving it upward on the electrode body.

m. Turn off the pH meter.

5.0 Quality Control

Always standardize the pH meter after it has been turned on and before it is used to measure pH. Follow manufacturer's instructions on care and troubleshooting the electrode.

Analyze at least one sample in duplicate for each batch of 20 or fewer samples. The soil sample used for the duplicate analysis must be well homogenized and split before weighed and suspended in regent water. If a single sample is being analyzed, this sample must be analyzed in duplicate. Acceptance criteria for duplicate analysis of soil pH using this method has not yet been established.

6.0 Bibliography

1. Soil Survey Staff (2014) Section 4.3 Soil pH in Soil Survey Field and Laboratory Methods Manual. Soil Survey Investigations Report No. 51, Version 2.0. R. Burt and Soil Survey Staff (ed.). U.S. Department of Agriculture, Natural Resources Conservation Service.
2. Kalra, Yash P (1995) Determination of pH of Soils by Different Methods: Collaborative Study. Journal of AOAC International, v78, n2, pp. 310-324.
3. Miller, Robert O. and Kissel, David E. (2010) Comparison of Soil pH Methods on Soils of North America. Soil Sci. Soc. Am. J., v74, pp. 310-316.