AOAC Official Method 994.16
pH Measurement of Mineral Soils

First Action 1994
Final Action 1997
Alternative I

(Applicable to pH measurement of mineral soils containing <17% organic carbon.)

See Table **994.16A** for the results of the interlaboratory study supporting acceptance of the method.

A. Principle

Air-dried soil test sample is mixed with H_2O (1 + 1). pH is measured potentiometrically.

B. Apparatus

(**a**) *pH meter.*—Equipped with glass electrodes (indicating and reference), or combination electrode.

(**b**) *Automatic pipet.*—Capable of accurately delivering 10 mL.

(**c**) *Stirrer.*—Glass rod or portable mechanical stirrer, capable of stirring at 1550 rpm. Small stirrer motor mounted on handle with short, slightly bent plastic or glass rod agitator is acceptable.

(**d**) *Glassware.*—Paper cups holding 28 g; or glass beakers, 50 mL.

C. Reagents

(**a**) *H_2O.*—Distilled or deionized.

(**b**) *Standard buffers.*—pH 4.00, 7.00, and 10.00. Use pH 4.00 and 7.00 for acid soils, and pH 7.00 and 10.00 for alkaline soils.

D. Calibration

Calibrate pH meter to appropriate setting using 2 standard buffers, **C**(**b**), depending on type of test sample. If reading of second standard buffer is not within 0.05 pH unit after adjusting to first standard buffer, follow manufacturer's instructions.

E. Preparation of Standard Soils

Use 3 different standard soils (air-dry) of known pH, covering pH range of test samples. Weigh 10 g air-dried soil into paper cup. (*Note*: Calibrated volume measurement of soil may be substituted for weighing.) Add 10 mL H_2O, **C**(**a**), to soil using automatic pipet. (*Note*: For fine-textured soils containing high level of organic matter it may be necessary to add additional 10 mL H_2O to make suspension.) Mix thoroughly 5 s with glass rod or mechanical stirrer. Let soil–H_2O suspension stand for 30 min. Measure pH of each standard as in **G**. pH values are acceptable within ±0.1 pH unit of known values. If pH values fall outside this range, recalibrate instrument with standard buffers and check pH of standard soils again. Follow manufacturer's instructions for recalibration. Replace electrodes if they cannot be calibrated within acceptable limits.

If pH values of all standard soils are 0.1 pH unit lower or higher than known pH of soil, recheck reference electrode.

Repeat pH measurements of standard soils just before analysis of test sample.

F. Preparation of Test Sample

Air dry soil at 20–40°C for 1–4 days depending on the relative humidity and soil test properties. Grind air-dried soil to pass 2 mm sieve and mix well. Prepare soil test sample–H_2O suspension as in **E**, beginning "Weigh 10 g air-dried soil into paper cup . . .".

G. Determination

Perform pH measurement at 20–25°C. Before analysis stir test solution from **F** with glass rod or mechanical stirrer. Insert electrode(s) of calibrated pH meter from **D** into container and swirl soil–H_2O suspension slightly. [*Note*: Position of reference electrode with respect to glass electrode and flow rate from reference electrode may affect pH determination; follow manufacturer's instructions for electrode(s).] Read pH immediately (after 30–60 s) to the nearest 0.1 pH unit. After removing electrode(s) from soil–H_2O suspension rinse them with H_2O; blot off excess H_2O with filter paper.

Follow manufacturer's instructions for storing and maintaining pH electrodes.

Alternative II

(Applicable to pH measurement of mineral soils with variable salt content.)

Table 994.16A Interlaboratory study results for pH measurements of mineral soils using alternative I

Soil	Overall mean of laboratory values (x)	Repeatability standard deviation (s_r)	Repeatability (r)	Reproducibility standard deviation (s_R)	Reproducibility (R)	Repeatability relative standard deviation (RSD_r), %	Reproducibility relative standard deviation (RSD_R), %
Malbis	5.62	0.30	0.86	0.40	1.14	5.28	7.20
Peck 90-9	6.13	0.19	0.54	0.23	0.66	3.04	3.69
Peck 90-12	4.88	0.18	0.51	0.28	0.80	3.60	5.73
Saline soil	9.74	0.16	0.46	0.26	0.74	1.67	2.67
Peck 90-11	7.86	0.39	1.11	0.44	1.25	4.92	5.54
Halii	4.55	0.18	0.51	0.29	0.83	3.94	6.35
Peck 90-10	5.87	0.18	0.51	0.24	0.68	3.14	4.00
Myakka	5.94	0.29	0.83	0.34	0.97	4.80	5.80
Laurentides	4.28	0.33	0.94	0.46	1.31	7.80	10.75
Houston	7.73	0.11	0.31	0.22	0.63	1.45	2.81

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Table 994.16B Interlaboratory study results for pH measurements in mineral soils using alternative II

Soil	Overall mean of laboratory values (x)	Repeatability standard deviation (s_r)	Repeatability (r)	Reproducibility standard deviation (s_R)	Reproducibility (R)	Repeatability relative standard deviation (RSD_r), %	Reproducibility relative standard deviation (RSD_R), %
Malbis	5.09	0.07	0.20	0.13	0.37	1.40	2.47
Peck 90-9	5.53	0.12	0.34	0.17	0.48	2.21	3.13
Peck 90-12	4.39	0.14	0.40	0.20	0.57	3.09	4.51
Saline soil	9.25	0.09	0.26	0.19	0.54	0.95	2.03
Peck 90-11	7.36	0.18	0.51	0.33	0.94	2.41	4.47
Halii	4.34	0.08	0.23	0.18	0.51	1.75	4.16
Peck 90-10	5.40	0.06	0.17	0.13	0.37	1.13	2.41
Myakka	4.31	0.04	0.11	0.12	0.34	0.99	2.86
Laurentides	3.69	0.25	0.71	0.28	0.80	6.91	7.54
Houston	7.48	0.15	0.43	0.16	0.46	2.02	2.17

See Table **994.16B** for the results of the interlaboratory study supporting acceptance of the method.

H. Principle

Salt content in soil influences ionic activity, which affects pH value of soil-water suspension. H^+ anions are displaced by cations. Exchangeable Al is displaced, which increases concentration of H^+ in solution. pH is decreased by ca 0.5 pH unit if $CaCl_2$ is used instead of H_2O.

I. Apparatus

Same as in **B**.

J. Reagents

See **C**(**a**)–(**b**).

(**c**) *$CaCl_2$ solution.*—0.01M. Dissolve 14.7 g $CaCl_2 \cdot 2H_2O$ in 10 L H_2O. pH of solution should be 5.0–6.5. If required, adjust pH with $Ca(OH)_2$ or HCl. Specific conductivity should be 2.32 ± 0.08 mS cm^{-1} at 25°C.

K. Calibration

Perform as in **D**.

L. Preparation of Standard Soils

Use 3 different standard soils (air-dry) of known pH, covering pH range of test samples. Weigh 10 g air-dry soil into paper cup. (*Note*: Calibrated volume measurement of soil may be substituted for weighing.) Add 10 mL 0.01M $CaCl_2$, **J**(**c**), to soil using automatic pipet. (*Note*: For fine-textured soils containing high level of organic matter it may be necessary to add additional 10 mL 0.01M $CaCl_2$ to make suspension.) Mix thoroughly 5 s with glass rod or mechanical stirrer. Let soil–$CaCl_2$ suspension stand for 30 min. Measure pH of each standard as in **G**. pH values are acceptable within ±0.1 pH unit of known values. If pH values fall outside this range, recalibrate instrument and check pH of standard soils again. Follow manufacturer's instructions for recalibration. Replace electrodes if they cannot be calibrated within acceptable limits.

If pH values of all standard soils are 0.1 pH unit lower or higher than known pH of soil, recheck reference electrode.

Repeat pH measurements just before analysis of test sample.

M. Preparation of Test Sample

Air dry soil at 20–40°C 1–4 days depending on relative humidity and soil properties. Grind air-dried soil to pass 2 mm sieve and mix well. Prepare soil test sample–$CaCl_2$ suspension as in **L**, beginning "Weigh 10 g air-dried test soil into paper cup . . .".

N. Determination

Proceed as in **G**.

Reference: *J. AOAC Int.* **78**, 310(1995).

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