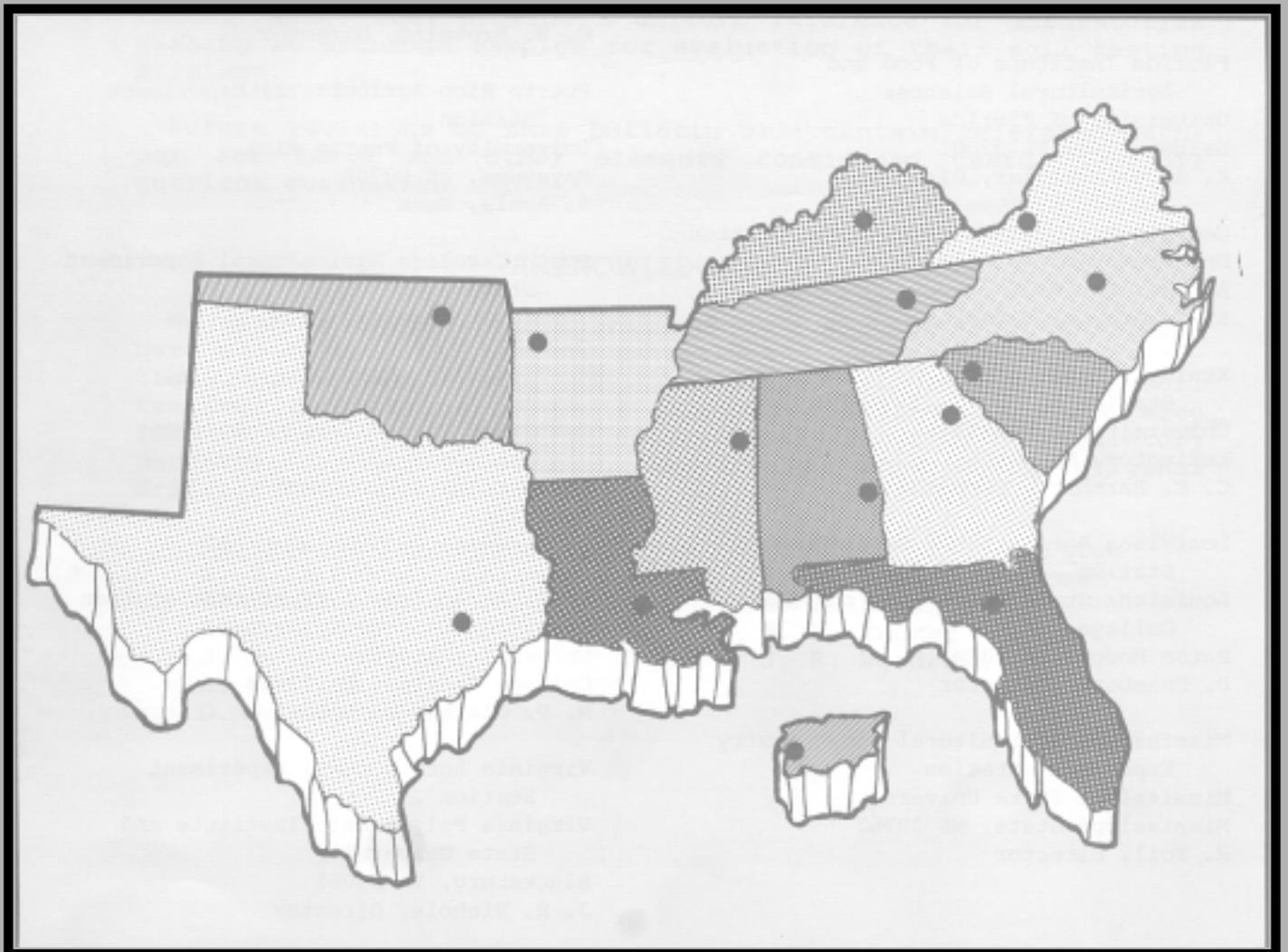




The University of Georgia  
College of Agriculture  
Experiment Stations

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# Reference Soil Test Methods for the Southern Region of the United States



Bulletin 289 is a publication in the Southern Cooperative Series and, as such, is in effect a separate publication by each of the cooperating Agricultural Experiment Stations listed below. Thus, it may be mailed under the frank and indicia of each. Requests for copies from outside the cooperating states may be addressed to the Georgia Agricultural Experiment Station, 125 Barrow Hall, University of Georgia, Athens, GA 30602.

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## FOREWORD

Over the past several years the Southern Regional Soil Testing and Plant Analysis Information Exchange Group (SRIEG-18) has worked towards developing standard procedures for use in evaluating the nutrient status and acidity level of soils in the southern United States. Standard procedures serve as a reference for those interested in including a new analysis in their program or who wish to employ a more suitable procedure for evaluation of a particular element.

This bulletin contains nine reference procedures for the analyses most commonly performed by soil testing laboratories in this region. Procedures were selected based on their accuracy in predicting crop response to applied nutrient as well as their popularity and general acceptance by workers in the soil testing field. Also, they provide a uniform reference for laboratories wishing to exchange samples for evaluation of their soil testing programs.

Future revisions of this bulletin will contain reference methods for trace and other elements considered useful in soil nutrient evaluation.

## ACKNOWLEDGMENTS

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for the Southern Region  
of the United States**

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# Determination of Soil Water pH

## 1. PRINCIPLE OF THE METHOD

1.1 This procedure is used to determine the pH of a soil in a water suspension. Soil pH is defined as the negative logarithm to base 10 of the H ion concentration, or the logarithm of the reciprocal of the H ion concentration in the soil solution. Therefore, since the pH is logarithmic, the H ion concentration in solution increases ten times when the pH is lowered one unit.

1.2 Most commercially available standard pH meters are adequate for measuring soil water pH through the range 3.5 to 8.5, which would include most soils encountered.

## 2. RANGE AND SENSITIVITY

2.1 Commercially available standard pH meters have an adequate range to measure the pH in water of usual soils between pH 3.5 and 8.0.

2.2 The sensitivity will depend on the instrument. In routine soil testing, it is only necessary to read the pH to the nearest 0.1 unit.

## 3. INTERFERENCES

3.1 Hydrogen ions may be displaced from the exchange sites, and additional H ions are formed in solution by other ions. This results in a lower pH (see 12.5).

3.2 Carbon dioxide ( $\text{CO}_2$ ) from the atmosphere or soil air dissolves in water forming carbonic acid ( $\text{H}_2\text{CO}_3$ ), which can lower the pH markedly. Only in soils which have a pH considerably above 7.0, i.e., very low H ion concentration, does the  $\text{CO}_2$  concentration of the air have an appreciable effect on the pH measurement.

## 4. PRECISION AND ACCURACY

4.1 Random variation of 0.1 to 0.2 pH units are allowable in replicate determinations, and this can be expected from one laboratory to another.

## 5. APPARATUS

5.1 No. 10 (2-mm opening) sieve.

5.2 Scoop, 10  $\text{cm}^3$  volume.

5.3 Cup, 50 ml, glass, plastic, or wax paper of similar size.

- 5.4 Pipette, 10-ml capacity.
  - 5.5 Stirring apparatus (mechanical shaker, stirrer, or glass rod).
  - 5.6 pH meter, line or battery operated with reproducibility to at least 0.05 pH units, and glass electrode paired with a calomel reference electrode.
  - 5.7 Glassware and dispensing apparatus for the preparation and dispensing of buffer solutions.
  - 5.8 Analytical balance.
6. REAGENTS
- 6.1 pH 7.0 Buffer Solution - Dissolve 3.3910 g citric acid ( $C_6H_8O_7$ ) and 23.3844 g disodium phosphate ( $Na_2HPO_4 \cdot 12H_2O$ ) in pure water and dilute to 1 liter (Commercially available buffer is acceptable).
  - 6.2 pH 4.0 Buffer Solution - Dissolve 11.8060 g citric acid and 10.9468 g disodium phosphate in pure water and dilute to 1 liter (Commercially available buffer is acceptable).
7. PROCEDURE
- 7.1 Scoop 10 cm<sup>3</sup> of air-dry, >10-mesh (2-mm) soil into a cup (see 5. 3). Pipette 10 ml pure water into the cup and mix for 5 seconds. Let stand for 30 minutes. Calibrate the pH meter according to instructions supplied with the specific meter. Stir the soil and water slurry (see 5.5). Lower the electrodes into the soil-water suspension so that the tip of the electrodes are at the soil-water interface. Stir the soil suspension by swirling the cup slightly just prior to reading the pH. Read the pH to the nearest tenth of a unit.
  - 7.2 Save the sample for the determination of the buffer pH.
8. CALIBRATION AND STANDARDS
- 8.1 The pH meter is calibrated using prepared (see 6.1 and 6.2) or commercially available buffer solutions of pH 7.0 and pH 4.0 according to the instrument instruction manual.
9. CALCULATION
- 9.1 The result is reported as pH<sub>w</sub> or pH in water suspension.
10. EFFECTS OF STORAGE

10.1 Air-dry soils may be stored several months in closed containers without affecting the  $pH_w$  measurement.

10.2 If the pH meter and electrodes are not to be used for extended periods of time, the instructions for storage published by the instrument manufacturer should be followed.

11. INTERPRETATION - see 12.1, 12.2, 12.3, and 12.4.

## 12. REFERENCES

12.1 Schofield, R. K. and A. W. Taylor. 1955. The measurement of soil pH. *Soil Sci. Soc. Amer. Proc.* 19:164-167.

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12.3 Peech, M. 1965. Hydrogen-ion activity. In C. A. Black (ed), *Methods of soil analysis, Part II, chemical and microbiological properties.* Amer. Soc. Agron., Madison, Wis., pp 914-926.

12.4 McLean, E. O. 1973. Testing soils for pH and lime requirement. In L. M. Walsh and J. D. Beaton. (ed), *Soil testing and plant analysis, revised edition,* Soil Sci. Soc. Amer., Madison, Wis., pp 78-95.

12.5 Coleman, N. T. and G. W. Thomas. 1964. Buffer curves of acid clays as affected by the presence of ferric iron and aluminum. *Soil Sci. Soc. Amer. Proc.* 28:187-190.

# Determination of Soil Buffer pH by the Adams-Evans Lime Buffer

## 1. PRINCIPLE OF THE METHOD

1.1 This procedure describes the determination of the lime requirement of a soil by the Adams-Evans buffer method (see 12.1). The method was developed for non-montmorillonitic, low organic matter soils where amounts of lime needed are small and the possibility of damage from over-liming exists. The lime requirement of an acid soil is defined by this procedure as the amount of lime or other base required to change an acid condition to a less acid condition (a maximum pH of 6.5).

1.2 The Adams-Evans lime requirement method is based on separate measures of soil pH and buffer pH (see 12.1 and 12.2). Soil pH is used to estimate acid saturation of the soil ( $H\text{-sat}_1$ ) from the relationship

$$\text{Soil pH} = 7.79 - 5.55(H\text{-sat}_1) + 2.27(H\text{-sat}_1)^2$$

The same relationship is used to calculate the acid saturation at the desired soil pH ( $H\text{-sat}_2$ ). For example, at a pH of 6.5, the value for  $H\text{-sat}_2$  is 0.26. The buffered solution estimates exchange acidity (soil H). Each 0.008 meq of acid results in a pH change of 0.01 units in 20 ml of solution (10 ml water + 10 ml buffer). This change is linear between pH 7 and 8.

$$\text{Soil H (meq/100 cm}^3\text{)} = 8(8.00 - \text{buffer pH})$$

Acid to be neutralized is calculated from the desired change in H-saturation.

$$\text{Acid to be neutralized} = \frac{\text{Soil H} \times (H\text{-sat}_1 - H\text{-sat}_2)}{H\text{-sat}_1}$$

## 2. RANGE AND SENSITIVITY

2.1 The Adams-Evans buffer method is very reliable for soils with relatively small amounts of exchangeable acidity (max. = 8 meq/100 g). The procedure provides a fairly high degree of accuracy for estimating lime requirements to reach pH 6.5 or less.

2.2 A sensitivity for the lime requirement determination is within 500 lbs/A of lime.

## 3. INTERFERENCES

3.1 There are no significant interferences.

#### 4. SENSITIVITY

- 4.1 A sensitivity of 0.01 in pH of the buffer-soil slurry is needed for the interpretation of this analysis.

#### 5. APPARATUS

- 5.1 No. 10 (2-mm opening) sieve.
- 5.2 Scoop, 10 cm<sup>3</sup>, volumetric.
- 5.3 Cup, 50 ml, glass, plastic, or waxed paper of similar size.
- 5.4 Pipette, 10-ml capacity.
- 5.5 Mechanical shaker or stirrer.
- 5.6 pH meter, line or battery operated with reproducibility to at least 0.01 pH units and glass electrode paired with calomel reference electrode.
- 5.7 Glassware and dispensing apparatus for preparing and dispensing Adams-Evans buffer.
- 5.8 Analytical balance.

#### 6. REAGENTS

- 6.1 pH 7.0 Buffer Solution - Dissolve 3.3910 g citric acid (C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>) and 23.3844 g disodium phosphate (Na<sub>2</sub>HPO<sub>4</sub> • 12H<sub>2</sub>O) in pure water and dilute to 1 liter (Commercially available buffer is acceptable).
- 6.2 pH 4.0 Buffer Solution - Dissolve 11.8060 g citric acid and 10.9468 g disodium phosphate in pure water and dilute to 1 liter (Commercially available buffer is acceptable).
- 6.3 Adams-Evans Lime Buffer Solution - Dissolve 74 g potassium chloride (KCl) in 500 ml pure water. Add 10.5 g potassium hydroxide (KOH), and stir to bring into solution. Add 20 g p-nitrophenol (HO•C<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>) and continue to stir. Add 15 g boric acid (H<sub>3</sub>BO<sub>3</sub>). Stir and heat, if necessary, to bring into solution. Dilute to 1 liter with pure water when cool. Adjust pH to 8.00 with either KOH or HCl.

#### 7. DETERMINATION

- 7.1 Scoop 10 cm<sup>3</sup> of air-dry, <10-mesh (2-mm) soil into a 50-ml cup (see 5.3). Add 10 ml pure water and mix for 5 seconds. Wait for 30 minutes, stir and read the soil pH. Add 10 ml Adams-Evans buffer solution (see 6.1) to the cup. Shake at 250 oscillations per minute (OPM) on

an oscillating shaker for 10 minutes or stir intermittently. Let stand for 30 minutes. Stir and read the soil-buffer pH on a standard pH meter. Read the pH to the nearest 0.05 pH unit.

## 8. CALIBRATION AND STANDARD

8.1 The pH meter is calibrated using prepared (see 6.1 and 6.2) or commercially available buffer solutions of pH 7.0 and pH 4.0 according to the instrument instruction manual. The pH meter is then adjusted to read pH 8.00 in an equal volume solution of Adams-Evans buffer (see 6.3) and pure water.

## 9. CALCULATIONS

9.1 The Adams-Evans buffer method assumes that agricultural-grade limestone is about 2/3 effective in neutralizing acidity up to a soil pH of about 6.5 and allows for this by using a correction of 1.5. Thus, the lime requirement is the product of the following equation (see 1.2):

$$\frac{\text{Soil H}}{\text{H-sat}_1} \times (\text{H-sat}_1 - \text{H-sat}_2) \times 1.5,$$

or for 10 cm<sup>3</sup> soil in 10 ml water + 10 ml buffer, it is

$$\text{CaCO}_3(\text{T/A}) = \frac{8000(8.00 - \text{buffer pH})}{\text{H-sat}_1} \times (\text{H-sat}_1 - \text{H-sat}_2) \times 1.5$$

## 10. EFFECTS OF STORAGE

10.1 Air-dry soils may be stored several months in closed containers without affecting the  $\text{pH}_{\text{Adams}}$  measurement.

10.2 If the pH meter and electrodes are not to be used for extended periods of time, the instructions for storage published by the instrument manufacturer should be followed.

## 11. INTERPRETATION

11.1 The Adams-Evans buffer method was developed for soils that have a maximum soil H of 8.00 meq/100 g and which have  $\text{H-sat}_1$  of 1.00 at about pH 4.5. However, it readily extends to soils with more H by adding less than 10 g of soil to 10 ml water and 10 ml buffer and multiplying by the appropriate dilution factor. It also extends to soils that have pH values below 4.5 when  $\text{H-sat}_1$  is 1.00 by changing the intercept of the pH equation by the appropriate amount (see 1.2). For example, a soil that has a pH of 4.0 when H-sat is 1.00 has the following relationship between pH and H-saturation:

$$\text{Soil pH} = 7.29 - 5.55(\text{H-sat}) + 2.27(\text{H-sat})^2$$

11.2 The lime requirement for low CEC soils (with pH of about 4.5 when H-saturated) can be determined in Table I from values based on the soil pli, and the Adams-Evans buffer pH. The lime requirement is given in terms of lb/A agricultural ground limestone,  $\text{TNP}_{90}$  and a 6 2/3-inch plow depth to increase soil pH to 6.5 (see 9.1).

Table 1. Lime requirement in pounds per acre to adjust furrow slice of soil (2 million pounds) to pH 6.5

Soil pH in water	Soil pH in buffered solution									
	7.90	7.80	7.70	7.60	7.50	7.40	7.30	7.20	7.10	7.00
6.3	183	366	549	732	915	1098	1281	1464	1647	1830
6.1	324	648	972	1295	1619	1943	2267	2591	2915	3238
5.9	436	872	1308	1744	2180	2616	3052	3489	3925	4361
5.7	528	1056	1584	2112	2641	3169	3697	4225	4753	5281
5.5	605	1211	1816	2422	3027	3633	4238	4844	5449	6055
5.3	672	1344	2016	2689	3361	4033	4705	5377	6049	6721
5.1	731	1462	2193	2924	3655	4386	5117	5848	6579	7310
4.9	785	1569	2354	3138	3923	4707	5492	6276	7061	7845
4.7	836	1672	2507	3343	4179	5015	5850	6686	7522	8358
4.5	891	1782	2674	3565	4456	5347	6239	7130	8021	8912

## 12. REFERENCES

- 12.1 Adams, F. and C. E. Evans. 1962. A rapid method for measuring lime requirement of red-yellow podzolic soils. Soil Sci. Soc. Amer. Proc. 26:355-357.
- 12.2 Hajek, B. F., F. Adams, and J. T. Cope. 1972. Rapid determination of exchangeable bases, acidity, and base saturation for soil characterization. Soil Sci. Soc. Amer. Proc. 36:436-438.

## Determination of Specific Conductance in Supernatant 1:2 Soil:Water Solution

### 1. PRINCIPLE OF THE METHOD

1.1 Although specific conductance measurements in saline soils are principally carried out on a soil-paste extract, research workers in the humid soil region have normally used a 1:2 soil:water extract, particularly in connection with highly fertilized greenhouse soils. Specific conductance values in the 1:2 extract were observed not to be comparable with those in the saturation extract. However, Jackson (see 12.1) concludes that specific conductance ranges of the widely contrasting alkaline and humid regions are quite similar.

1.2 Specific conductance measurements may be made on all greenhouse soils and on all field problem soils. The 1:2 soil:water ratio in the procedure is based on a soil volume rather than on a soil weight basis. This avoids the need for further dilution of low bulk density Histosols and potting materials. Guidelines for restoring fields flooded by salt water are included.

### 2. RANGE AND SENSITIVITY

2.1 The method is adapted to a wide range of salt concentrations, depending on the instrument, and it can be extended outside of the instrument range by suitable dilution of the sample.

### 3. INTERFERENCES

3.1 Specific conductance increases with increasing temperature, hence, compensation of temperature differences from the calibrated standard is required.

3.2 For reproducible results clean and well-platinized electrodes are essential.

### 4. PRECISION AND ACCURACY

4.1 Specific conductance values expressed as millimho per centimeter (mmho per cm) should be reported to the second decimal place (see 12.2).

4.2 Uniformity and interpretation of test results are impaired by reporting specific conductance in units other than mmho per cm.

### 5. APPARATUS

5.1 No. 10 (2-mm) sieve.

- 5.2 50-60 ml plastic, paper or glass beaker.
- 5.3 10 cm<sup>3</sup> capacity, volumetric soil measure (see 12.3).
- 5.4 Pipette, 20 ml transfer or pipetting machine.
- 5.5 Conductivity meter, Solu-Bridge, or equivalent.
- 5.6 Conductivity cell, pipette type, 2 to 3 ml capacity.
- 5.7 Thermometer, 1-100°C.

## 6. REAGENTS

- 6.1 0.01N Potassium Chloride (KCl) - Dissolve 0.7456 g of KCl in pure water made up to 1 liter at 25°C.

## 7. CALIBRATION AND STANDARDS

- 7.1 To determine the cell constant ( $\emptyset$ ), 0.01 N KCl (see 6.1) solution at 25°C will have a specific conductance (SC) of 0.0014118 mho per cm.
- 7.2 The cell constant ( $\emptyset$ ) of any commercially available conductivity cell, according to Willard, Merritt and Dean (see 12.4), is obtained from the relationship:

$$K = \frac{1(\emptyset)}{R A} = \frac{\emptyset}{R}$$

where K is the specific conductance, A is the electrode area and d is the distance (cm) between electrode plates. R is the resistance in ohms per cm. In the case of 0.01 N KCl (see 7.1), the cell constant  $\emptyset = 0.0014118$  (in mho per cm) x R (in ohm per cm). R = 708.5 ohm if the cell is 1.0 cm ( $\emptyset$ -1.0). Notice that mhos = ohm<sup>-1</sup>.

- 7.3 Some Solu-Bridge instrument dials read in SC (mhos x 10<sup>-5</sup>) as well as resistance (ohms). Before accepting the mhos x 10<sup>-5</sup> dial readings, the cell constant should be determined and the mho x 10<sup>-5</sup> dial readings substantiated as being correct for the cell constant used.

## 8. PROCEDURE

- 8.1 Measure 10 cm<sup>3</sup> (see 5.3) of 2 mm sieved soil into beaker (see 5.5), add 20 ml pure water, stir thoroughly and allow suspension to settle for at least 30 minutes or long enough for the solids to settle.
- 8.2 Draw supernatant into the conductivity pipette to slightly above the constricted part of pipette. Avoid drawing liquid into rubber bulb. If this occurs, rinse

bulb before continuing with the next sample.

## 9. CALCULATIONS

9.1 Specific conductance (SC) of the soil extract is calculated as follows:

$$\text{SC, mhos per cm at } 25^{\circ}\text{C} = \frac{0.0014118 \times R_{\text{std}}}{R_{\text{ext}}}$$

where the value of 0.0014118 is the specific conductance of the standard 0.01 N KCl solution in mho per cm at 25°C and  $R_{\text{std}}$  and  $R_{\text{ext}}$  refer to resistance in ohms of the standard (0.01 N KCl) solution and extract, respectively. Multiply the results by 1000 to obtain mmho per cm at 25°C. Report specific conductance values in mmho per cm.

9.2 Alternate method of calculation:

After the cell constant ( $\emptyset$ ) has been determined (as in 7.2 above), specific conductance of the soil extract can be obtained from the relationship,

$$\text{SC, mhos per cm at } 25^{\circ}\text{C} = \frac{\emptyset}{R}$$

where  $\emptyset$  is the determined cell constant and R is the resistance in ohms per cm of the soil extract.

## 10. INTERPRETATION

10.1 Results with various soils and crops, using a 1:2 soil:water ratio have been reported by Dunkle and Merkle (see 12.6) and Merkle and Dunkle (see 12.7). Jackson (see 12.1) summarized these and other studies giving the specific conductance in 1:2 soil:water extract (observed) to that in the saturation extract (calculated) for a silt loam of 40% and a clay loam high in organic matter at 100% saturation. The conductance ratios of the 1:2 saturation extract values of the 40 and 100% saturated soils were 0.2 and 0.5, respectively.

10.2 Using the 0.2 ratio values in relation to the Scofield salinity scale (see 12.5) together with published guides (see 12.8, 12.9, 12.10, 12.11) a general guide to plant effects associated with different ranges of specific conductance measured in a 1:2 soil:water ratio by volume follows:

Table 1. Soluble salt reading in mmho/cm and corresponding approximate ppm of salt in 1:2 air-dried soil (volume to volume) water extract and salinity effects

Reading mmho/cm*	Salt ppm	Salinity Effects
<0.40	<512	Salinity effects mostly negligible.
0.40-0.80	512-1024	<u>Very slightly saline.</u> Yield of crops of low salt tolerance may be reduced by 50% for all classes of crops (see Table 2).
0.81-1.60	1025-2048	<u>Slightly saline.</u> Yields of fruit and vegetable crops of medium salt tolerance may be reduced by 50%. Similar yield reductions may occur in the more sensitive forage and field crops of medium salt tolerance. Lower half (0.81-1.20) of range satisfactory for well-drained mineral greenhouse soils. Upper half (1.21-1.60) of range higher than desirable for greenhouse soils except for peat and lightweight mixes.
1.61-2.40	2049-3072	<u>Moderately saline.</u> Yields of virtually all fruit crops significantly reduced. Yield reductions of 50% may occur in the more sensitive forage and field crops of high salt tolerance. Similar yield reductions occur in the more highly salt-tolerant vegetable crops. For greenhouse crops (>2.0) leach soil with enough water so that 2-4 quarts pass through each square foot of bench area or one pint of water per 6-inch pot; repeat after about one hour. Repeat again if readings are still in the high range.
2.41-3.20	3073-4096	<u>Strongly saline.</u> Only highly salt-tolerant forage and field crops will yield satisfactorily.
>3.20	>4096	<u>Very strongly saline.</u> Only a few highly salt-tolerant grasses, herbaceous plants and certain shrubs and trees will grow.

\*mmho/cm x 1000 = mg salt/dm<sup>3</sup>

Table 2. Range of salt tolerance of crops

SC, mmho/cm	Rating	Salt Tolerance
----- Forage Crops -----		
0.5-0.8	Low*	Ladino clover, burnet, red clover, alsike clover, meadow foxtail, white dutch clover.
0.81-2.6	Medium	Milkvetch, sourclover, tall meadow oatgrass, smooth brome, big trefoil, reed canary, meadow fescue, blue grama, orchardgrass, oats (hay), wheat (hay), rye (hay), alfalfa, hubam clover, sudan grass, dallis grass, strawberry clover, mountain brome, beardless wildrye, birdsfoot trefoil, perennial ryegrass, yellow sweetclover, white sweetclover.
2.61-3.6	High	Hardinggrass, barley (hay), tall fescue, crested wheatgrass, canada wildrye, tall wheatgrass, rescue grass, rhodes grass, bermuda grass, nutall alkaligrass, saltgrass, alkali sacaton.
----- Field Crops -----		
0.5-0.8	Low	Field peas, soybeans, field beans.
0.81-2.6	Medium	Castorbeans, sunflower, flax, broadbean - lima, pinto, etc., corn (field), rice, sesbania, soybean, sorghum (grain), oats (grain), wheat (grain), rye (grain), safflower.
2.61-3.6	High	Cotton, rape, sugarbeet, barley.
----- Vegetable Crops -----		
0.5-0.8	Low	Beans, celery, radish.
0.81-1.6	Medium	Cucumber, squash, pea, onion, carrot, lettuce, cauliflower, bell pepper, potato, sweet potato, sweet corn, cabbage, broccoli, tomato.
1.61-2.4	High	Spinach, asparagus, kale, beets.

\* No crop injury at low end of range to as much as 50% yield reduction at high end of range.

Table 2. (continued)

SC, mmho/cm	Rating	Salt Tolerance
		----- Fruit Crops -----
0.5-0.8	Low	Strawberry, avocado, blackberry, boysenberry, raspberry, peach, apricot, almond, plum, prune, apple, pear, grapefruit, orange, lemon.
0.81-1.6	Medium	Cantaloupe, grape, olive, fig, pomegrante, date palm.

## 11. GUIDELINES FOR RESTORING FIELDS FLOODED BY SALT WATER

- 11.1 Plants growing on saltwater flooded soil exhibit greatest damage when soil moisture is limiting growth. When the soil is relatively dry, the salt concentration of the soil solution around the plant roots is the greatest and prevents uptake of moisture. On the other hand, saltwater may wash across fields doing little or no damage if the soil has been previously saturated by rain or fresh-water flooding.
- 11.2 Treatments in returning land to a productive level is based on the salt content from properly collected samples of the suspected salt-damaged area. Collect core samples 18 inches deep from each field. Divide cores into four parts as follows: (a) 0-2 inches; (b) 2-6 inches; (c) 6-12 inches; and (d) 12-18 inches.
- 11.3 Reclamation - Apply calcium sulfate (landplaster, gypsum) to the fields if mmho/cm is above 1.00 in the top 6 inches of the soil. The application rate is as follows: (a) less than 2% organic matter, 2,000 lbs/a; (b) 2 to 5%, 3,000 lbs/a; and (c) above 5%, 4,000 lbs/a.
- 11.4 Resample in 3 to 6 months to determine progress of treatments. Since calcium sulfate contributes to the specific conductance, it is essential to determine calcium and sodium in the extract.

## 12. REFERENCES

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## Determination of Phosphorus by Mehlich I (0.05N HCl in 0.025N H<sub>2</sub>SO<sub>4</sub>) Extraction

### 1. PRINCIPLE OF THE METHOD

- 1.1 This method is primarily for determining phosphorus in sandy soils which have exchange capacities of less than 10 meq/100 g and are acid in reaction (pH less than 6.5). The method is not suited for alkaline soils.
- 1.2 The method was first published by Mehlich (see 12.1) and then by Nelson, Mehlich, and Winters (see 12.2) as the North Carolina Double-Acid Method and is adaptable to the Coastal Plain soils of eastern United States. It is currently being used by a number of state soil testing laboratories in the United States (Alabama, Delaware, Florida, Georgia, Maryland, New Jersey, South Carolina, and Virginia).

### 2. RANGE AND SENSITIVITY

- 2.1 Phosphorus can be extracted and determined in soil concentrations from 2-200 kg P/ha without dilution. The upper limit may be extended by diluting the extract prior to calorimetric determination.
- 2.2 The sensitivity varies depending on the method of color development. Greater sensitivity can be obtained with the molybdophosphoric acid blue color method (see 12.3) as compared to the vanadomolybdophosphoric acid color method (see 12.4). The estimated sensitivity of the method is  $\pm 1$  ppm P.

### 3. INTERFERENCES

- 3.1 With some soils, the extract may be colored, varying from light to dark yellow. If the vanadomolybdophosphoric acid method (see 12.4) is employed, as originally prescribed for the double-acid method (see 12.1), decolorizing is necessary to avoid obtaining high results. Decolorization can be accomplished by including activated charcoal in the extraction procedure. Decolorization is not necessary if color development is by the molybdophosphoric blue color procedure (see 12.3). A description of the method is given by Watanabe and Olsen (see 12.5).
- 3.2 Arsenate present in the extractant will produce a blue color with the molybdophosphoric blue color procedure unless the arsenate is reduced. A reduction procedure is given by Jackson (see 12.6).

## 4. PRECISION AND ACCURACY

4.1 Repeated analyses of two standard soil samples over 30 days in the Georgia Soil Testing and Plant Analysis Laboratory gave variance of 6.4 to 9.0%, respectively. Each soil tested 32 and 40 kg P/ha, respectively. The variance is essentially a factor related to the homogeneity of the soil rather than the extraction or colorimetric procedures.

## 5. APPARATUS

5.1 No. 10 (2-mm opening) sieve.

5.2 Scoop, 5 cm<sup>3</sup> volumetric.

5.3 Extraction bottle or flask, 50 ml with stoppers.

5.4 Mechanical reciprocating shaker, 180 oscillations/minute.

5.5 Filter funnel, 11 cm.

5.6 Whatman No. 1 filter paper (or equivalent), 11 cm.

5.7 Photoelectric calorimeter suitable for measurement in the 880 nm range.

5.8 Colorimetric tube or cuvet.

5.9 Funnel racks.

5.10 Analytical balance.

5.11 Volumetric flasks and pipettes as required for preparation of reagents, standard solutions and color development.

## 6. REAGENTS

6.1 All reagents are ACS analytical grade unless otherwise noted.

6.2 Extracting Reagent (0.05N HCl in 0.025N H<sub>2</sub>SO<sub>4</sub>)- Dilute 4 ml concentrated HCl and 0.7 ml concentrated H<sub>2</sub>SO<sub>4</sub> to 1 liter with pure water.

6.3 Ascorbic Acid Solution - Dissolve 176.0 g ascorbic acid in pure water and dilute to 2 liters with pure water. Store in dark glass bottle in a refrigerated compartment.

6.4 Sulfuric-Molybdate Solution - Dissolve 100 g ammonium molybdate [(NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>•4H<sub>2</sub>O] in 500 ml of pure water. Dissolve 2.425 g antimony potassium tartrate

[K(SbO)C<sub>4</sub>H<sub>4</sub>O<sub>6</sub>•1/2H<sub>2</sub>O] in molybdate solution. Add slowly 1400 ml concentrated H<sub>2</sub>SO<sub>4</sub> and mix well. Let cool and dilute to 2 liters with pure water. Store in polyethylene or Pyrex bottle in a dark, refrigerated compartment.

- 6.5 Working Solution - Dilute 10 ml of the ascorbic acid solution (see 6.3) plus 20 ml of the sulfuric-molybdate solution (see 6.4), with extracting reagent (see 6.2) to 1 liter. Prepare fresh daily. Allow to stand at least 2 hours before using.
- 6.6 Phosphorus Standard (1000 ppm) - Weigh 3.85 g ammonium dihydrogen phosphate (NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>) into a 1-liter volumetric flask and bring to volume with extracting solution (see 6.2). Prepare standards containing 1, 2, 5, 10, 15, and 20 µg P/ml diluting aliquots of the 1000 µg P/ml standard with extraction solution (see 6.2).

## 7. PROCEDURE

- 7.1 Extraction - Measure 5 cm<sup>3</sup> of air-dry, <10-mesh (2-mm) soil into a 50-ml extraction bottle (see 5.1). Add 25 ml of the extraction solution (see 6.3) and shake for 5 minutes on a reciprocating shaker at a minimum of 180 oscillations per minute (see 5.2). Filter and collect the extract.
- 7.2 Color Development - Pipette 1 ml of the extractant into a spectrophotometer cuvet. Add 24 ml of the working solution (see 6.5). Mix well and let stand 20 minutes. Read the absorbance at 882 nm. The spectrophotometer should be zeroed against a blank consisting of extraction reagent (see 6.2).

## 8. CALIBRATION AND STANDARDS

- 8.1 Working Phosphorus Standards - With the standard phosphorus solution (see 6.6), prepare 6 working standard solutions containing from 1 to 20 µg phosphorus per ml in the final volume. Make all dilutions with the extracting reagent (see 6.2). Use a 1.0 ml aliquot of each standard and carry through the color development (see 7.2).
- 8.2 Calibration Curve - On semilog graph paper, plot the percent transmittance on the logarithmic scale versus ppm phosphorus on the linear scale.
- 8.3 The color intensity reaches a maximum in approximately 20 minutes and will remain constant for about 6 hours.

## 9. CALCULATIONS

9.1 The results are reported as kg P/ha for a 20 cm depth of soil. Kg P/ha =  $\mu\text{g P/ml}$  of extract  $\times 10$ .

## 10. EFFECTS OF STORAGE

10.1 Soils may be stored in an air-dry condition for several months with no effect on extractable P.

10.2 After extraction, the extraction solution containing P should not be stored any longer than 24 hours.

## 11. INTERPRETATION

11.1 Accurate fertilizer recommendations for phosphorus must be based on field response data conducted under local soil-climate-crop conditions (see 12.7). For most soils and crops, the amount of P extracted is to be interpreted as follows:

<u>Category</u>	(to 20-cm depth) <u>kg P/ha in soil</u>
very low	< 16
low	17-38
medium	39-76
high	77-150
very high	> 150

11.2 Interpretations may vary somewhat depending on soil characteristics and different crops.

## 12. REFERENCES

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# Determination of Phosphorus by Bray P1 Extraction

## 1. PRINCIPLE OF THE METHOD

1.1 The extraction of phosphorus by this procedure is based upon the solubilization effect of the  $H^+$  on soil phosphorus and the ability of the  $F^-$  to lower the activity of  $Al^{+3}$  and to a lesser extent that of  $Ca^{+2}$  and  $Fe^{+2}$  in the extraction system. As described in this section, clay soils with a moderately high degree of base saturation or silty clay loam soils that are calcareous or have a very high degree of base saturation will lessen the solubilizing ability of the extractant. Consequently, the method should normally be limited to soils with  $pH_w$  values less than 6.8 when the texture is silty clay loam or finer. Calcareous soils, or high pH, fine textured soils may be tested by this method, but higher ratios of extractant-to-soil are often used for such soils (see 12.6); another alternative is the Olsen P procedure, (see 12.4).

1.2 The extractant was developed and first described by Bray and Kurtz (12.1). The extraction time and the solution-to-soil ratio in their procedure were 1 minute and 20 ml extractant to 2.85 g soil, respectively. To simplify adaptation to routine laboratory work and to extend the range of soils for which the extractant was suitable, both the extraction time and the solution-to-soil ratio have been altered by various laboratories. The extractant at varying extraction times and ratios is currently used in a majority of the mid-east, mid-south, and north central areas of the United States.

## 2. RANGE AND SENSITIVITY

2.1 This procedure yields a standard curve that is essentially linear to 10 ppm of phosphorus in the soil extract (approximately 200 kg P/ha or 178 lbs P/A of extractable phosphorus).

2.2 The sensitivity is approximately 0.15 ppm in the extract (3.0 kg/ha or 2.7 lbs/A phosphorus in the soil).

## 3. INTERFERENCES

3.1 Arsenic - concentrations of 1 ppm As in the extract do not interfere (see 12.3). Techniques for excessive As removal are given by Jackson (see 12.5).

3.2 Silica - Silica is tolerated up to 10  $\mu g$  of Si/ml extract. (see 12.3).

3.3 Fluoride - The fluoride in the extract normally will not interfere in the formation of the molybdenum blue color with phosphorus. Any interference may be eliminated upon addition of boric acid (see 12.2). Maximum color development is slower in the presence of the fluoride ion.

#### 4. PRECISION AND ACCURACY

The reproducibility of determinations by this procedure depends upon the extent to which the times of extraction and filtration, and color development are controlled. Reasonable control and thorough sample preparation should give a coefficient of variation of about 5%.

#### 5. APPARATUS

- 5.1 No. 10 (2-mm opening) sieve.
- 5.2 Scoop, 2.5 cm<sup>3</sup> volumetric.
- 5.3 Extraction bottle or flask - 50 ml with stoppers.
- 5.4 Mechanical reciprocating shaker, minimum of 200 oscillations per minute.
- 5.5 Filter funnel, 11 cm.
- 5.6 Whatman No. 2 filter paper or equivalent, 11 cm.
- 5.7 Photoelectric calorimeter suitable for measurement in the 880 nm range.
- 5.8 Colorimeter tube or cuvet.
- 5.9 Funnel racks.
- 5.10 Volumetric flasks and pipettes as required for preparation of reagents, standard solutions, and color development.

#### 6. REAGENTS

##### 6.1 Extracting Reagent (0.03N NH<sub>4</sub>F in 0.025N HCl):

- 6.1.1 1N NH<sub>4</sub>F - Dissolve 37 g ammonium fluoride in 400 ml pure water and dilute the solution to 1 liter. Store in a polyethylene container and avoid prolonged contact with glass.
- 6.1.2 0.5N HCl - Dilute 20.4 ml conc. HCl to 500 ml with pure water.
- 6.1.3 Extracting Reagent - Mix 30 ml of 1N NH<sub>4</sub>F (see 6.11) with 50 ml of 0.5 N HCl (see 6.12) and

dilute to 1 liter with pure water. This solution is 0.03 N in  $\text{NH}_4\text{F}$  and 0.025 N in  $\text{HCl}$  and has a pH of 2.6. Stored in polyethylene, it is stable for more than 1 year.

- 6.2 Sulfuric-Molybdate Solution - Dissolve 40 g ammonium molybdate  $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}]$  in 500 ml of pure water. Dissolve 0.972 g antimony potassium tartrate  $[\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6\cdot 1/2\text{H}_2\text{O}]$  in the molybdate solution. Add slowly 492 ml conc.  $\text{H}_2\text{SO}_4$  and mix well. Let cool and dilute to 2 liters with pure water. Store in polyethylene or Pyrex bottle in a dark, refrigerated compartment.
- 6.3 Working Solution - Add 60 ml of sulfuric-molybdate solution (see 6.2) to 800 ml of pure water and dissolve 1.056 g of 1-ascorbic acid in this solution. Dilute to 1 liter with pure water. Prepare fresh daily.
- 6.4 Phosphorus Standard (100 ppm) - Weigh 0.4394 g monobasic potassium phosphate ( $\text{KH}_2\text{PO}_4$ ) which has been oven-dried at  $100^\circ\text{C}$  into a 1-liter volumetric flask and bring to volume with extracting reagent (see 6.1.3).

## 7. PROCEDURE

- 7.1 Extraction - Scoop 2.0  $\text{cm}^3$  of air-dry <10-mesh soil (2-mm) into a 50-ml extraction bottle or flask, add 25 ml extracting reagent (see 6.1.3) and shake for 5 minutes on a reciprocating shaker at a minimum of 200 oscillations per minute (see 5.4). Filter through Whatman No. 2 filter paper, limiting the filtration time to 10 minutes and save the extract.
- 7.2 Color Development - Transfer exactly 1.0 ml of extract or standard solution to a calorimeter tube. Add 8 ml of working solution (see 6.3) and mix the contents of the tube thoroughly. After 10 minutes, measure the color intensity at 882 nm. The color intensity is stable for 4 hours.

## 8. CALIBRATION AND STANDARDS

- 8.1 Working Phosphorus Standards - With the standard phosphorus solution (see 6.4), prepare 6 working standard solutions containing from 0.2 to 10 ppm of phosphorus in the final volume. Make all dilutions with extracting reagent (see 6.1.3).
- 8.2 Calibration Curve - On semilog graph paper, plot the percent transmittance on the logarithmic scale versus ppm phosphorus in the standard solutions on the linear scale.

## 9. CALCULATIONS

9.1 The results are reported as kg P/ha for a 20 cm depth of soil. Kg/ha of phosphorus in the soil = ppm in the extract x 25. (This assumes a uniform 1.0 ml aliquot is used for standards and unknowns in 7.2.)

## 10. EFFECTS OF STORAGE

10.1 After air drying, the extractable phosphorus levels in soils remain stable for several months.

10.2 After extraction, the phosphorus in the extract should be measured within 12 hours.

## 11. INTERPRETATION

11.1 Accurate fertilizer recommendations for phosphorus must be based on field response data conducted under local soil-climate-crop conditions (see 12.7), but in general the extractable phosphorus levels may be categorized as follows:

<u>Category</u>	Extractable P, <u>kg/ha</u>
Low	<34
Medium	34-68
High	>68

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## Determination of Potassium, Calcium, Magnesium, and Sodium by Mehlich I (0.05N HCl in 0.024N H<sub>2</sub>SO<sub>4</sub>) Extraction

### 1. PRINCIPLE OF THE METHOD

1.1 This method is primarily for determining potassium, calcium, and magnesium in soils which have exchange capacities of less than 10 meq/100 g, are acid in reaction (pH less than 6.5), and are relatively low in organic matter content (less than 5%). The method is not suited for alkaline soils.

1.2 The use of the double acid as an extracting reagent was first published by Mehlich (see 12.1) and then specifically as a phosphorus extraction reagent by Nelson, Mehlich, and Winters (see 12.2) as the North Carolina Double-Acid Method and is adaptable to the Coastal Plain soils of eastern United States. It is currently being used by a number of state soil testing laboratories in the United States [Alabama, Delaware, Florida, Georgia, Maryland, New Jersey, and South Carolina, and Virginia (see 12.3)].

### 2. RANGE AND SENSITIVITY

2.1 Potassium, calcium, and magnesium can be extracted and determined in soil concentrations from 50 to 400 K, 120 to 1200 Ca, and 40 to 360 Mg kg/ha without dilution. The range and upper limits may be extended by diluting the extracting filtrate prior to analysis.

2.2 The sensitivity will vary with the type of instrument used, wave length selected, and method of excitation.

2.3 The commonly used methods of analysis are flame emission and atomic absorption spectroscopy. A more complete description of these methods is given by Isaac and Kerber (see 12.4). The use of an auto analyzer for this analysis is given by Flannery and Markus (see 12.5) and Isaac and Jones (see 12.6).

### 3. INTERFERENCES

3.1 Known interferences and compensation for the changing characteristics of the extract to be analyzed must be acknowledged. The use of internal standards such as lithium and compensating elements such as lanthanum are essential in most flame methods of excitation (see 12.4 and 12.5).

## 4. PRECISION AND ACCURACY

- 4.1 Repeated analysis of the same soil with medium concentration ranges of potassium, calcium, and magnesium will give variances of from 5 to 10 percent. A major portion of the variance is related to the homogeneity of the soil rather than the extraction or method of analysis.
- 4.2 The level of exchangeable potassium will increase upon the air drying of some soils (see 12.7). Soil samples can be extracted in the moist state; however, the difficulty in handling and storage of moist soil makes this method difficult for easy adaptation to a routine method analysis. Compensation can be made based on the expected release of potassium by the particular soil being tested.

## 5. APPARATUS

- 5.1 No. 10 (2-mm opening) sieve.
- 5.2 Scoop, 5 cm<sup>3</sup> volumetric.
- 5.3 Extraction bottle or flask, 50 ml with stoppers.
- 5.4 Mechanical reciprocating shaker, 180 oscillations per minute.
- 5.5 Filter funnel, 11 cm.
- 5.6 Whatman No. 1 filter paper (or equivalent), 11 cm.
- 5.7 Flame emission, atomic absorption spectrophotometer and/or AutoAnalyzer.
- 5.8 Funnel racks.
- 5.9 Analytical balance.
- 5.10 Volumetric flasks and pipettes as required for preparation of reagents and standard solutions.

## 6. REAGENTS

- 6.1 Extracting Reagent (0.05N HCl in 0.025N H<sub>2</sub>SO<sub>4</sub>) - Dilute 4 ml conc. HCl and 0.7 ml conc. H<sub>2</sub>SO<sub>4</sub> to 1 liter with pure water.
- 6.2 Potassium Standard (1000 ppm) - Weigh 1.9080 g potassium chloride (KCl) into a 1-liter volumetric flask and bring to volume with extracting reagent (see 6.1). Prepare working standards by diluting aliquots of the stock solution standard with extracting reagent (see 6.1) to cover the anticipated range in concentration to

be found in the soil extraction filtrate. Working standards from 5 to 50 ppm K should be sufficient for most soils.

- 6.3 Calcium Standard (1000 ppm) - Weigh 2.498 g calcium carbonate ( $\text{CaCO}_3$ ) into a 1-liter volumetric flask, add 50 ml of pure water, and add dropwise a minimum volume conc. HCl (approximately 20 ml) to effect complete solution of the calcium carbonate. Dilute to the mark with extracting reagent (see 6.1). Prepare working standards by diluting aliquots of the stock solution standard with extracting reagent (see 6.1) to cover the anticipated range in concentration to be found in the soil extraction filtrate. Working standards from 15 to 150 ppm Ca should be sufficient for most soils.
- 6.4 Magnesium Standard (1000 ppm) - Weigh 1.000 g magnesium ribbon (Mg) into a 1-liter volumetric flask and dissolve in minimum volume of (1+1) HCl and dilute in one liter of extracting reagent (see 6.1). Prepare working standards by diluting aliquots of the stock solution standard with extracting reagent (see 6.1) to cover the anticipated range in concentration to be found in the soil extraction filtrate. Working standards from 5 to 50 ppm Mg should be sufficient for most soils.
- 6.5 Sodium Standard (1000 ppm) - Weigh 2.542 g sodium chloride (NaCl) into a 1-liter volumetric flask and bring to volume with extracting reagent (see 6.1). Prepare working standards by diluting aliquots of the stock solution standards with extracting reagent (see 6.1) to cover the anticipated range in concentration to be found in the soil extraction filtrate. Working standards from 1 to 10 ppm Na should be sufficient for most soils.

## 7. PROCEDURE

- 7.1 Extraction - Scoop 5 cm<sup>3</sup> (see 5.2) of air-dry, <10-mesh (2-mm) soil into a 50-ml extraction bottle (see 5.3). Add 25 ml of the extracting reagent (see 6.1) and shake for 5 minutes on a reciprocating shaker at a minimum of 180 oscillations per minute (see 5.4). Filter and collect the filtrate.
- 7.2 Analysis - The elements potassium, calcium, magnesium, and sodium in the filtrate can be determined by either flame emission or atomic absorption spectroscopy or by an auto analyzer. Since instruments do vary in their operating conditions, no specific details are given here. It is recommended that the procedures described by Isaac and Kerber (see 12.4), Flannery and Markus (12.5), and Isaac and Jones (12.6) be followed.

## 8. CALIBRATION AND STANDARDS

8.1 Working Standards - Working standards should be prepared as described in section 6. If element concentrations are found outside the range of the instrument or standards, suitable dilutions should be prepared starting with a 1:1 soil extract to extracting reagent (see 6.1) dilution.

8.2 Calibration - Calibration procedures vary with instrument techniques and type of instrument. Every precaution should be taken to ensure that the proper procedures are taken and manufacturer recommendations followed in the operation and calibration of the instrument used.

## 9. CALCULATIONS

9.1 The results are reported as kg/ha for a 20 cm depth of soil. Kg of element/ha =  $\mu\text{g/ml}$  of element in extraction filtrate  $\times 10$ . If extraction filtrate is diluted, the dilution factor must be applied.

9.2 To convert to other units for comparison, see Mehlich (12.8).

## 10. EFFECTS OF STORAGE

10.1 Soils may be stored in an air-dry condition for several months with no effects on the exchangeable potassium, magnesium, and calcium content.

10.2 After extraction, the filtrate containing potassium, calcium, and magnesium should not be stored any longer than 24 hours unless refrigerated or treated to prevent bacterial growth.

## 11. INTERPRETATION

11.1 An evaluation of the analysis results as well as accurate fertilizer recommendations, particularly for the elements potassium and magnesium, must be based on field response data conducted under local soil-climate-crop conditions (see 12.9). Interpretative data used in the Southeast is also available (see 12.10).

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# Determination of Potassium, Calcium, Magnesium, and Sodium by Neutral Normal Ammonium Acetate Extraction

## 1. PRINCIPLE OF THE METHOD

- 1.1 This method uses a neutral salt solution to replace the cations present on the soil exchange complex; therefore, the cation concentrations determined by this method are referred to as "exchangeable."
- 1.2 The use of neutral normal ammonium acetate to determine exchangeable potassium was first described by Prianischnikov (see 12.1). Schollenberger and Simon (see 12.2) describe the advantages of this extracting reagent as to its effectiveness in wetting soil, replacing exchangeable cations, ease of volatility during analysis, and suitability for use with flame emission. More recent descriptions of this method are given by Jackson (see 12.3), Chapman (see 12.4) and Hesse (see 12.5). The neutral normal ammonium acetate extraction procedure is the most commonly used extraction reagent for determining potassium, magnesium, calcium, and sodium in soil testing laboratories in the United States (see 12.6).

## 2. RANGE AND SENSITIVITY

- 2.1 The range of detection will depend on the particular instrument setup. The range can be extended by the dilution of the extract.
- 2.2 The sensitivity will vary with the type of instrument used, wave length selected and method of excitation.
- 2.3 The commonly used methods of analysis are flame emission and atomic absorption spectroscopy. A more complete description of these methods is given by Isaac and Kerber (see 12.7).

## 3. INTERFERENCES

- 3.1 Under certain conditions, the extracting reagent (see 6.2) will extract more than those elements which exist in exchangeable form such as those elements released by weathering action during the period of extraction and the dissolution of carbonates of calcium and magnesium if present in the soil. However, these contributions will not normally significantly alter the analysis when used to assess the soil's fertility status.
- 3.2 Known interferences and compensation for the changing characteristics of the extract to be analyzed must be acknowledged. The use of internal standards such as

lithium and compensating elements such as lanthanum are essential in most flame methods of excitation (see 12.7).

#### 4. PRECISION AND ACCURACY

- 4.1 Repeated analysis of the same soil with medium concentration ranges of potassium, calcium, magnesium, and sodium will give variances of from 5 to 10 percent. A major portion of the variance is related to the homogeneity of the soil rather than the extraction or method of analysis.
- 4.2 The level of exchangeable potassium will increase upon the air drying of some soils (see 12.8). Soil samples can be extracted in the moist state; however, the difficulty in handling and storage of moist soil makes this method difficult for easy adaptation to a routine method of analysis. Compensation can be made based on the expected release of potassium by the particular soil being tested.

#### 5. APPARATUS

- 5.1 No. 10 (2-mm opening) sieve.
- 5.2 Scoop, 5 cm<sup>3</sup> volumetric.
- 5.3 Extraction bottle or flask, 50 ml with stoppers.
- 5.4 Mechanical reciprocating shaker, 180 oscillations per minute.
- 5.5 Filter funnel, 11 cm.
- 5.6 Whatman No. 1 filter paper (or equivalent), 11 cm.
- 5.7 Flame emission and/or atomic absorption spectrophotometer.
- 5.8 Funnel racks.
- 5.9 Analytical balance.
- 5.10 Volumetric flasks and pipettes as required for preparation of reagents and standard solutions.

#### 6. REAGENTS

- 6.1 Extracting Reagent (1N ammonium acetate adjusted to pH 7.0) - Dilute 57 ml of glacial acetic acid (99.5%) with pure water to a volume of approximately 500 ml. Then add 69 ml conc. ammonium hydroxide. CAUTION: Use fume hood. Add sufficient pure water to obtain volume of 990 ml. After thoroughly mixing the solution, adjust the

pH to 7.0 using ammonium hydroxide or acetic acid. Dilute to a final volume of 1000 ml with pure water. Alternate method: dissolve 77.1 g ammonium acetate in about 900 ml pure water. After thoroughly mixing the solution, adjust the pH to 7.0 using 3N acetic acid or approximately 3N ammonium hydroxide. Dilute to final volume of 1000 ml with pure water.

- 6.2 Potassium Standard (1000 ppm) - Weigh 1.9080 g potassium chloride (KCl) into 1-liter volumetric flask and bring to volume with extracting reagent (see 6.1). Prepare working standards by diluting aliquots of the stock solution standard with extracting reagent (see 6.1) to cover the anticipated range in concentrations to be found in the soil extraction filtrate. Working standards from 5 to 100 ppm K should be sufficient for most soils.
- 6.3 Magnesium Standard (1000 ppm) - Weigh 1.000 g magnesium ribbon (Mg) into 1-liter volumetric flask and dissolve in minimum volume of (1+1) HCl and dilute in one liter of extracting reagent (see 6.1). Prepare working standards by diluting aliquots of the stock solution standard with extracting reagent (see 6.1) to cover the anticipated range in concentration to be found in the soil extraction filtrate. Working standards from 5 to 50 ppm Mg should be sufficient for most soils.
- 6.4 Calcium Standard (1000 ppm) - Weigh 2.498 g calcium carbonate (CaCO<sub>3</sub>) into a 1-liter volumetric flask, add 50 ml of pure water, and add dropwise a minimum volume of concentrated HCl (approximately 20 ml) to effect complete solution of the calcium carbonate. Dilute to mark with extracting reagent (see 6.1). Prepare working standards by diluting aliquots of the stock solution standard with extracting reagent (see 6.1) to cover the anticipated range in concentration to be found in the soil extraction filtrate. Working standards from 50 to 200 ppm Ca should be sufficient for most soils.
- 6.5 Sodium Standard (1000 ppm) - Weigh 2.542 g sodium chloride (NaCl) into a 1-liter volumetric flask and bring to volume with extracting reagent (see 6.1). Prepare working standards by diluting aliquots of the stock solution standards with extracting reagent (see 6.1) to cover the anticipated range in concentration to be found in the soil extraction filtrate. Working standards from 1 to 10 ppm Na should be sufficient for most soils.

## 7. PROCEDURE

7.1 Extraction - Scoop 5 cm<sup>3</sup> (see 5.2) of air-dry <10-mesh (2-mm) soil into a 50-ml extraction bottle (see 5.3). Add 25 ml of the extracting reagent (see 6.1) and shake for 5 minutes on a reciprocating shaker (see 5.4) at a minimum of 180 oscillations per minute. Filter and collect the filtrate.

7.2 Analysis - The elements potassium, magnesium, calcium, and sodium in the filtrate can be determined by either flame emission or atomic absorption spectroscopy. Since instruments do vary in their operating conditions, no specific details are given here. It is recommended that the procedures described by Isaac and Kerber (see 12.7) be followed.

## 8. CALIBRATION AND STANDARDS

8.1 Working Standards - Working standards should be prepared as described in section 6. If element concentrations are found outside the range of the instrument or standards, suitable dilutions should be prepared starting with a 1:1 soil extract to extracting reagent (see 6.1) dilution.

8.2 Calibration - Calibration procedures vary with instrument techniques and type of instrument. Every precaution should be taken to ensure that the proper procedures are taken and manufacturer recommendations followed in the operation and calibration of the instrument used.

## 9. CALCULATIONS

9.1 The results are reported as kg/ha for a 20 cm depth of soil. Kg of element/ha =  $\mu\text{g/ml}$  of element in extraction filtrate  $\times 10$ . If extraction filtrate is diluted, the dilution factor must be applied.

9.2 To convert to other units for comparison, see Mehlich (12.9).

## 10. EFFECTS OF STORAGE

10.1 Soils may be stored in an air-dry condition for several months with no effects on the exchangeable potassium, magnesium, calcium, and sodium content. Potassium may be released on drying for some soils (see 12.8).

10.2 After extraction, the filtrate containing potassium, calcium, magnesium, and sodium should not be stored any longer than 24 hours unless refrigerated or treated to prevent bacterial growth.

## 11. INTERPRETATION

- 11.1 An evaluation of the analytical results in relation to crop response and accurate fertilizer recommendations, particularly for the elements potassium and magnesium, must be based on field response data conducted under local soil-climate-crop conditions (see 12.9).

## 12. REFERENCES

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# Method for Determination of Organic Matter Using the Dichromic Titrimetric Procedure

## 1. PRINCIPLE OF METHOD

1.1 Dichromic acid reacts with carbon as follows (see 9.1):  
$$2\text{H}_2\text{Cr}_2\text{O}_7 + 3\text{C} + 6\text{H}_2\text{SO}_4 \rightarrow 2\text{Cr}_2(\text{SO}_4)_3 + 3\text{CO}_2 + 8\text{H}_2\text{O}$$
In measuring soil organic matter, the conditions of the reaction as well as the nature of the carbon compounds determine the true electron change equivalent for the above reaction and it may range between 3 and 4 electron change (see 9.1). The titrimetric method described here gives a quantitative measurement of the reaction.

1.2 The procedure described here is the WALKLEY-BLACK heat of dilution method (see 9.2), which is based on the Schollenberger external heat method (see 9.3).

## 2. RANGE

2.1 The range of the method is 0.05 - 6.70% organic matter.

## 3. INTERFERENCES

3.1 Charcoal, graphite, and other carbonaceous materials give a positive interference. The effect of these materials is greatly reduced by using the heat of dilution method because of less heating (see 9.1).

3.2 Chloride interferes positively with the chromic acid reaction, but it takes approximately 12 units by weight of Cl to equal 1 unit of organic matter, so normally this reaction is of no consequence (see 9.1).

3.3 Higher oxides of manganese ( $\text{MnO}_2$ ) when present in large quantities in finely divided, reactive form may interfere negatively (see 9.1). Normal soils would not contain sufficient active  $\text{MnO}_2$  to interfere significantly.

3.4 Ferrous iron may produce high results for the method. However, air dried soils contain very little ferrous iron (see 9.1).

## 4. APPARATUS

4.1 No. 10 (2-mm opening) sieve.

4.2 Drying oven 100° F.

4.3 Analytical balance.

4.4 500 ml erlenmeyer flasks.

4.5 Two 10 ml automatic pipets.

- 4.6 50 ml automatic pipet.
- 4.7 50 ml burette.
- 4.8 Volumetric flasks and pipets as required for preparation of reagents.

## 5. REAGENTS

- 5.1 Concentrated  $H_3PO_4$ , 85%
- 5.2 Concentrated  $H_2SO_4$ , 96%
- 5.3 Sodium Fluoride (NaF)
- 5.4 Potassium Dichromate Solution (1N) - Dissolve 49.04 g of potassium dichromate, previously dried at 100°C for two hours, in deionized water and dilute to 1 liter.
- 5.5 Diphenylamine Indicator - Dissolve 0.50 g reagent grade diphenylamine in 20 ml of water and add 100 ml of concentrated  $H_2SO_4$ .
- 5.6 Ferrous Ammonium Sulfate Solution (0.5N) - Dissolve 196.1 g of  $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$  in 800 ml of deionized water containing 20 ml of concentrated  $H_2SO_4$  and dilute to 1 liter. Make fresh daily.

## 6. PROCEDURE

- 6.1 Weigh 1.0 g soil into a 500 ml erlenmeyer flask and pipet 10 ml dichromate solution into the flask. Under a hood, add 20 ml concentrated  $H_2SO_4$  and mix by gentle rotation, taking care not to throw soil onto the sides of the flask. Let stand for 30 minutes and dilute to 200 ml with water. Add 10 ml of concentrated  $H_3PO_4$ , 0.2 g NaF and 10 drops of diphenylamine indicator. Titrate the solution with 0.5N ferrous ammonium sulfate solution. Prepare a blank with each set of samples.

## 7. CALCULATIONS

7.1  $\% \text{ O.M.} = 10(1 - T/S) \times 0.67$

T = sample titration

S = blank titration

This formula involves two important assumptions. The first is that the procedure oxidizes only 76% of the organic C to  $CO_2$ ; the second assumption is that soil organic matter contains 58% carbon. Either factor may vary considerably depending on the nature of the soil organic matter.

8. EFFECTS OF STORAGE

8.1 Soil may be stored in an air-dry condition with no effect on percent organic matter.

9. REFERENCES

9.1 Jackson, M. L. 1958. Soil Chemical Analysis, p. 214-221. Prentice-Hall, Inc., Englewood Cliffs, N. J.

9.2 Walkley, Allen. 1947. A critical examination of a rapid method for determination of organic carbon in soils - effect of variations in digestion conditions and of inorganic soil constituents. Soil Sci. 63:251-257.

9.3 Schollenberger, C.J. 1927. A rapid approximate method for determining soil organic matter. Soil Sci. 24:65-68.

# Colorimetric Determination of Organic Matter Content

## 1. PRINCIPLE OF THE METHOD

1.1 Dichromic acid reacts with carbon as follows (10.1):  
$$2\text{H}_2\text{Cr}_2\text{O}_7 + 3\text{C} + 6\text{H}_2\text{SO}_4 \rightarrow 2\text{Cr}_2(\text{SO}_4)_3 + 3\text{CO}_2 + 8\text{H}_2\text{O}.$$
In measuring soil organic matter, the conditions of the reaction as well as the nature of the carbon compounds determine the true electron change equivalent for the above reaction, and it may range between 3 and 4 electron change (10.1). The quantitative measurement of the reaction may be made titrimetrically or colorimetrically. The method described here is based upon measurement of the green color produced by  $\text{Cr}^{+3}$  in acid solution.

1.2 The basic procedure of oxidizing soil organic matter with chromic acid was developed by Schollenberger (10.2) which was then modified to use heat of dilution from  $\text{H}_2\text{SO}_4$  (10.3) and to use calorimetric determination (10.4). The modifications permit adaptation of the original method to routine laboratory measurements at the expense of some precision and extent of organic matter oxidation. To overcome the latter, the method is calibrated against the Walkley-Black method on soil samples with a range of organic matter contents.

## 2. RANGE AND SENSITIVITY

The range of the method is from 0.2 to 15.0% organic matter with an estimated sensitivity of 0.3 to 0.5% organic matter.

## 3. INTERFERENCES

3.1 Interferences in the calorimetric procedure have not been extensively investigated. In the basic procedure, charcoal, graphite, and other carbonaceous materials can cause positive errors. However, as heating is lessened, interference from extraneous carbon sources becomes less (10.1).

3.2 Chloride interferes positively in the chromic acid reaction (10.3) but 1.2 units of  $\text{Cl}^-$  are required to equal 1 unit of organic matter by weight, so normally the reaction is of no consequence.

3.3 Ferrous iron may produce high results for the method (10.1) but air-dried soils should not contain significant reactive  $\text{Fe}^{+2}$ .

3.4 Higher oxides of manganese ( $\text{MnO}_2$ ) when present in large

quantities in finely divided, reactive form may interfere negatively. Normal soils would not contain sufficient active  $\text{MnO}_2$  to interfere significantly.

#### 4. PRECISION AND ACCURACY

- 4.1 Repeated analyses should give results with a coefficient of variation of no greater than 5%.
- 4.2 Soil samples should be thoroughly ground and mixed before subsampling because heterogeneity is a serious problem in organic matter distribution within samples.

#### 5. APPARATUS

- 5.1 No. 10 (2-mm opening) sieve.
- 5.2 Scoop, 1.5  $\text{cm}^3$  volumetric.
- 5.3 Test tube, 200 ml.
- 5.4 Delivery burette, or 20-ml automatic pipette.
- 5.5 Colorimeter (or spectrophotometer) for measuring absorbance at 645 nm (red filter).
- 5.6 Analytical balance.
- 5.7 Volumetric flasks and pipettes as required for preparation of reagents and standard solutions.

#### 6. REAGENTS

- 6.1 Sodium dichromate solution (0.67M) - Dissolve 4,000 g of reagent grade sodium dichromate in distilled water and dilute to 20 liters.
- 6.2 Technical grade sulfuric acid.

#### 7. PROCEDURE

- 7.1 Scoop 1.5  $\text{cm}^3$  (see 5.2) (or weigh 2 g) of air-dry, <10 mesh soil into a 200-ml test tube. Under a hood, add 20 ml of dichromate solution (see 6. 1) and then 20 ml of sulfuric acid (see 6.2). The sample is mixed thoroughly (CAUTION) and allowed to cool at least 40 min. After cooling, 100 ml of water is added, the solution is mixed and allowed to stand at least 8 hours. A volume of the clarified solution is transferred to a colorimeter vial using a syringe pipette. Measure absorbance at 645 nm (or with red filter).

## 8. CALIBRATION AND STANDARDS

8.1 A standard curve is established with several soils having an adequate range of organic matter contents. The percent organic matter is determined by a standardized method; absorbance values are determined for each soil by this method. A curve is then constructed by plotting percent organic matter versus absorbance. Including a reference sample with daily runs of the method aids in verifying equivalent conditions between standard curve and daily runs.

## 9. EFFECTS OF STORAGE

9.1 Soils may be stored in an air-dry condition with no effects on percent organic matter.

## 10. REFERENCES

- 10.1 Jackson, M. L. 1958. Soil Chemical Analysis. p 216. Prentice-Hall, Inc. Englewood Cliffs, N.J.
- 10.2 Schollenberger, C.J. 1927. A rapid approximate method for determining soil organic matter. Soil Sci. 24:65-68.
- 10.3 Walkley, Allen. 1947. A critical examination of a rapid method for determination of organic carbon in soils - effect of variations in digestion conditions and of inorganic soil constituents. Soil Sci. 63:251-257.
- 10.4 Graham, E. R. 1948. Determination of soil organic matter by means of a photoelectric colorimeter. Soil Sci. 65:181-183.

## Conversion Table

### Weights and Measures

U.S. abbr	Length unit	Approximate Metric Equivalent
mi	mile	1.609 kilometers
yd	yard	0.9144 meters
ft or '	foot	30.48 centimeters
in or "	inch	2.54 centimeters
<b>Area</b>		
sq mi or mi <sup>2</sup>	square mile	2.59 square kilometers
acre	acre	0.405 hectares or 4047 square meters
sq ft or ft <sup>2</sup>	square foot	0.093 square meters
<b>Volume/capacity</b>		
gal	gallon	3.785 liters
qt	quart	0.946 liters
pt	pint	0.473 liters
fl oz	fluidounce	29.573 milliliters or 28.416 cubic centimeters
bu	bushel	35.238 liters
cu ft or ft <sup>3</sup>	cubic foot	0.028 cubic meters
<b>Mass/weight</b>		
ton	ton	0.907 metric tons
lb	pound	0.453 kilograms
oz	ounce	28.349 grams
gr	grain	0.0648 grams
<b>Metric</b>		
abbr	Length unit	Approximate U.S. Equivalent
km	kilometer	0.62 miles
m	meter	39.37 inches or 1.09 yards
cm	centimeter	0.39 inches
mm	millimeter	0.04 inches
<b>Area</b>		
ha	hectare	2.47 acres
<b>Volume/capacity</b>		
l*	liter	61.02 cubic inches or 1.057 quarts
ml	milliliter	0.06 cubic inches or 0.034 fluid ounces
cc	cubic centimeter	0.061 cubic inches or 0.035 fluid ounces
<b>Mass/weight</b>		
MT or t	metric ton	1.1 tons
kg	kilogram	2.205 pounds
g	gram	0.035 ounces
mg	milligram	0.015 grains or 3.5 x 10 <sup>-5</sup> ounces

\* (spell out if abbreviation of lower case l would be confused with numeral 1.)