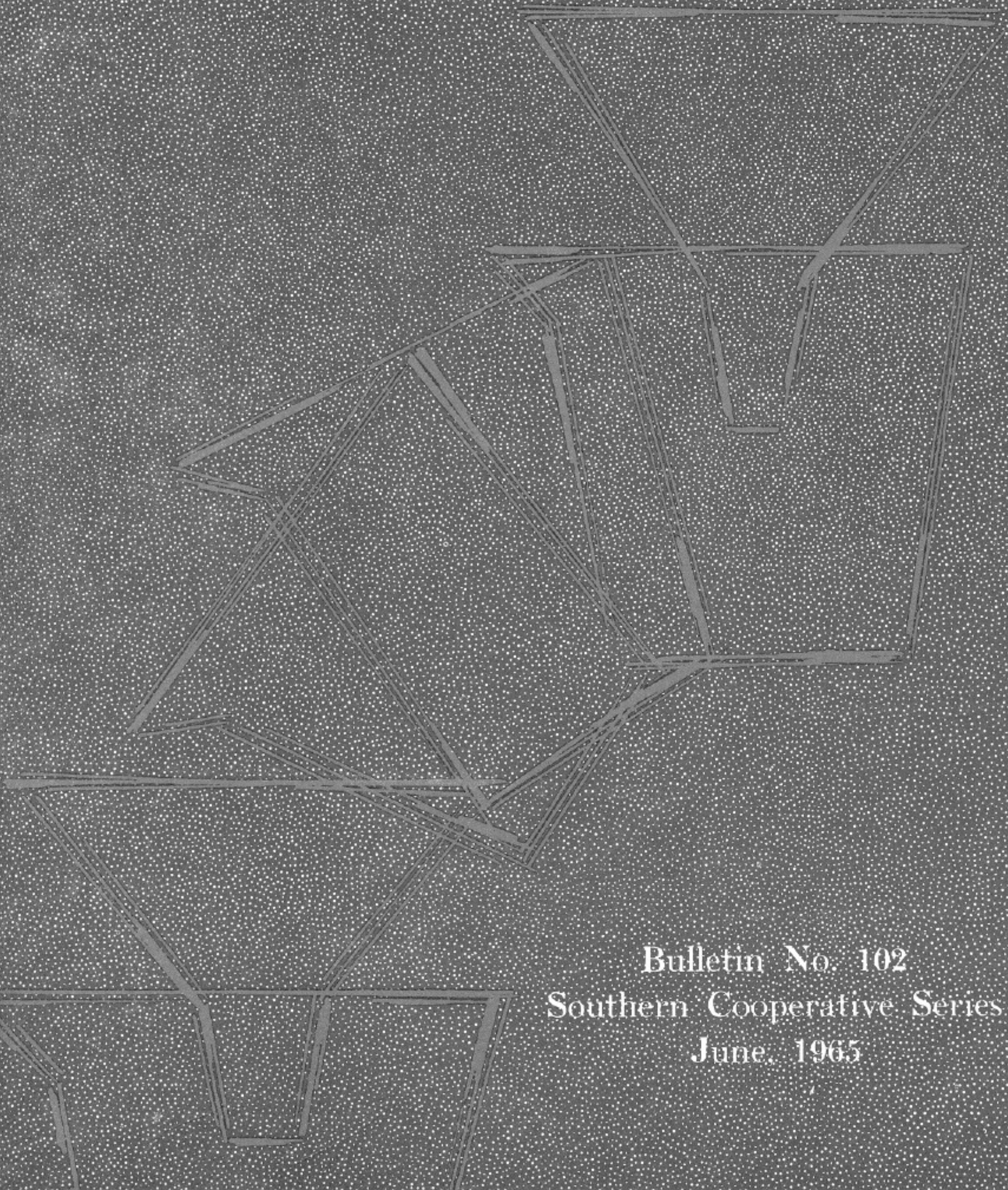
Procedures Used By State Soil-Testing Laboratories In The Southern Region Of The United States



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FOREWORD

Bulletin 102, like others in the Southern Cooperative Series, is in effect a separate publication by each of the cooperating Agricultural Experiment Stations, and as such may be mailed under the frank and indicia of each.

Since the bulletin is identical for all the cooperating stations listed below, it is suggested that copies be requested from only one source.

Requests for copies from areas outside the cooperating states should be addressed to the South Carolina Agricultural Experiment Station, Clemson, South Carolina.

E. V. Smith, Director, Alabama Agricultural Experiment Station, Auburn, Alabama.

E. M. Cralley, Director, Arkansas Agricultural Experiment Station, Fayetteville, Arkansas.

J. R. Beckenbach, Director, Florida Agricultural Experiment Station, Gainesville, Florida.

G. H. King, Director, Georgia Agricultural Experiment Stations, Athens, Georgia.

W. A. Seay, Director, Kentucky Agricultural Experiment Station, Lexington, Kentucky.

Doyle Chambers, Director, Louisiana Agricultural Experiment Station, Baton Rouge, Louisiana.

H. H. Leveck, Director, Mississippi Agricultural Experiment Station, State College, Mississippi.

R. L. Lovvorn, Director, North Carolina Agricultural Experiment Station, Raleigh, North Carolina.

L. E. Hawkins, Director, Oklahoma Agricultural Experiment Station, Stillwater, Oklahoma.

M. A. Lugo-Lopez, Acting Director, Puerto Rico Agricultural Experiment Station, Rio Piedras, Puerto Rico.

O. B. Garrison, Director, South Carolina Agricultural Experiment Station, Clemson, South Carolina.

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R. E. Patterson, Director, Texas Agricultural Experiment Station, College Station, Texas.

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C.S.R.S.	A. S. Newman
USDA-ARS	A. W. White L. F. Welch *

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* Former Members.

ACKNOWLEDGMENTS

Many individuals contributed to the development, adaptation, and correlation of the current soil-testing procedures used by the state soil-testing laboratories of the Southern Region. Sincere appreciation is extended to all experiment station and extension personnel who contributed to the development and standardization of these soil-test procedures.

Special appreciation is expressed to members of the former Soil-Test Work Group of the Southern Regional Soils Research Committee and to former and present members of the S-52 Technical Committee.

Present members of the Technical Committee submitted the procedures listed as being used by the soil-testing laboratories of their respective states and reviewed the manuscript for accuracy.

EDITORIAL COMMITTEE,

N. R. Page
G. W. Thomas
H. F. Perkins
R. D. Rouse

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PROCEDURES USED BY STATE SOIL-TESTING LABORATORIES IN THE SOUTHERN REGION OF THE UNITED STATES

Compiled by N. R. PAGE

INTRODUCTION*

The use of soil testing as an aid to efficient crop production has been accepted with enthusiasm. Nowhere else does this enthusiasm exceed that found in the South. The reasons for this are at once apparent. Southern soils, in general, are inherently deficient in at least two of the three major nutrient elements. Furthermore, they tend to be acid and to respond to the application of liming materials. After these soils are farmed and fertilized for some time, however, the status of their nutrient levels is by no means certain. Indeed, under intensive cultivation, levels of some nutrients may be excessively high, whereas others are depleted severely. This problem is complicated by the low cation-exchange capacities of many southern soils.

Soil testing is an effective instrument for informing the producer of crops where deficiencies and excesses exist. Use of this information strongly influences recommendations made by farm advisors as well as fertilizer and lime usage by farmers themselves.

Despite the progress which has been made, the candid observer must admit that there are troubles ahead for soil testing. One of these troubles is a lack of uniform recommendations when state lines are crossed. This is evident even when given soil types extend from one state to another. While it probably is idealistic to look for uniform recommendations on a given soil as one goes from state to state, it also is foolish for adjacent states to ignore the problem that exists.

Because of the interest in exchanging information on soil testing, the Southern Regional Soil-Testing Work Group was formed in 1954. After a number of years of very constructive exchanges of information among members of the Work Group, the Southern Soils Research Committee recommended to the Southern Experiment Station Directors that a regional project on soil testing should be initiated. Such a project was authorized and initiated in 1962 under the number S-52. One of the activities of the S-52 Technical Committee has been a compilation of the soil-testing methods now in use by the various states in the Southern Region.

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* Prepared by G. W. Thomas.

The compilation and publication of such information in this bulletin is designed to accomplish at least three objectives: first, research workers may become more fully aware of methods used in the region; second, it should increase exchange of methods in an effort to use the best ones available; and third, the bulletin records the progress that already has been achieved in the difficult task of soil analysis in the Southern Region.

SAMPLE PREPARATION

All of the states in the Southern Region except Puerto Rico have one or more soil-testing laboratories designed to analyze soil samples by quick-test procedures for the citizens of the state.

All except three of the soil-testing laboratories air-dry each soil sample at room temperature before it is processed. Alabama dries each sample at 55° C for 24 hours. Arkansas dries certain soils that are high in clay at 85° F for 48 hours. North Carolina uses a rack designed so that air, which has been heated by a large light bulb, passes over the open soil samples.

After crushing, the soil is screened through a seive (mesh size varies from 0.84 to 2.00 mm) to remove rocks, sticks, and plant debris. Georgia, Kentucky (University laboratory only), Louisiana, Mississippi, North Carolina, Texas, and Virginia use a mechanical soil crusher. The other laboratories crush the soil samples by hand.

It is assumed that each soil sample has been taken properly and that it is representative of the area to be evaluated.

SPECIALIZED AND AUTOMATIC EQUIPMENT

To reduce the time required for extraction, filtering, analysis, and washing, all of the laboratories use various types of racks that hold a multiple number of filtering tubes, bottles, or vials. In addition, automatic pipettes and shakers, washing and drying equipment, and electric stirrers are widely used. Since these innovations vary among laboratories and do not affect the accuracy of the analyses, they will not be described.

pH

Reagents (may be purchased or prepared as described)

1. **Buffer Solution, pH 4.00:**
    a. Dissolve 11.806 g of citric acid and either 10.9468 g of Na_2HPO_4 or 27.6152 g of $Na_2HPO_4 \cdot 12H_2O$ in 1 liter of distilled water.

b. Add 4.0 ml of 0.1N NaOH to 500 ml of 0.1M potassium acid phthlate and dilute to 1 liter with distilled water.

2. **Buffer Solution, pH 7.00:**

a. Dissolve 3.391 g of citric acid and either 23.3844 g of Na_2HPO_4 or 58.9913 g of $Na_2HPO_4 \cdot 12H_2O$ in 1 liter of distilled water.

b. Add 296.3 ml of 0.1N NaOH to 500 ml of 0.1M potassium acid phthlate and dilute to 1 liter with distilled water.

General Procedure

Weigh or measure a quantity of soil equivalent to 10 – 50 g into a beaker or cup (50 – 150 ml). Add sufficient distilled water or 1N KCl to give either a 1:1, 1:2, or 1:2.5 soil-solution ratio and mix thoroughly. After equilibrating the soil-solution mixture for a minimum period of time (15 minutes – 12 hours), either filter or stir the mixture thoroughly, and measure the pH value immediately on a pH meter by means of the glass electrode.

Before determining the pH of a soil sample, standardize the pH meter against two known buffer solutions having pH values of 4.00 and 7.00, respectively.

Variations From the General Procedure

1. **Soil-Solution Ratios:**

a. **1:1 Ratio (volume):** Alabama (20 ml soil – 20 ml water).

b. **1:1 Ratio (weight):** North Carolina (10 g soil – 10 ml water); Oklahoma (15 g soil – 15 ml water or 1N KCl); Arkansas and Georgia (20 g soil – 20 ml water); Louisiana (35 g soil – 35 ml water); and Virginia (37 g soil – 37 ml water).

c. **1:2 Ratio:** Mississippi, Puerto Rico, South Carolina, and Texas (10 g soil – 20 ml water) and Florida (50 g soil – 100 ml water).

d. **1:2.5 Ratio:** Kentucky (10 g soil – 25 ml water).

2. **Minimum Equilibration Period After Adding Water:**

Puerto Rico – shake 15 minutes and filter; Arkansas and Oklahoma – 20 minutes; Georgia, Kentucky, and Texas – 30 minutes; Alabama, Florida (except for peats and mucks), Mississippi, North Carolina, South Carolina, and Virginia – 1 hour; Florida (peats and mucks) – 2 hours; and Louisiana – 12 hours or overnight.

EXTRACTING SOLUTIONS

Currently 17 different extracting solutions are being used by the state soil-testing laboratories of the Southern Region to estimate the residual levels of cations and anions in the soil that are available to plants. The large number of extracting solutions is due partially to the wide diversity of soils in the region and to differences in soil-test development within each state. Each extracting solution has been calibrated locally with crop response so that it meets the needs of the state or region in which it is used. The major disadvantage to a large number of extracting solutions on a regional basis is that they hinder the exchange of calibration data among states.

The extracting solutions used by the soil-testing laboratories of the Southern Region and the element(s) that are estimated from the extract by each state are summarized in table 1.

The quantities of reagents required and the procedure for preparing 18 liters of each extracting solution are:

1. **0.15N H_2SO_4:** Dilute 72 ml of concentrated H_2SO_4 to 18 liters with distilled water. Determine the normality by titrating with a standard base and adjust the normality, if necessary.

2. **1 percent $(NH_4)_2SO_4$ in 0.05N H_2SO_4:** Add 25 ml of concentrated H_2SO_4 and 180 g of $(NH_4)_2SO_4$ to approximately 10 liters of distilled water. After the $(NH_4)_2SO_4$ has dissolved, dilute to 18 liters with distilled water and mix thoroughly.

3. **0.1N HCl:** Dilute 146 ml of concentrated HCl to 18 liters with distilled water.

4. **0.03N NH_4F in 0.1N HCl:** Add 146 ml of concentrated HCl and 20 g of NH_4F to approximately 5 liters of distilled water. When the NH_4F has dissolved, dilute to 18 liters with distilled water and mix thoroughly.

5. **0.03N NH_4F in 0.025N HCl:** Add 37.5 ml of concentrated HCl and 20 g of NH_4F to approximately 5 liters of distilled water. When the NH_4F has dissolved, dilute to 18 liters with distilled water and mix thoroughly.

6. **0.05N HCl and 0.025N H_2SO_4:** Add separately 73 ml of concentrated HCl and 12 ml of concentrated H_2SO_4 to approximately 10 liters of distilled water. Dilute to 18 liters with distilled water and mix thoroughly.

7. **0.018N CH_3COOH:** Dilute 18.5 ml of concentrated CH_3COOH to 18 liters with distilled water.

8. **0.7N CH_3COONH_4 in 0.54N CH_3COOH buffered at pH 4.8:** Add separately 1271 ml of concentrated CH_3COOH and 860 ml of

TABLE I.—THE EXTRACTING SOLUTIONS USED BY THE SOIL-TESTING LABORATORIES OF THE SOUTHERN REGION AND THE ELEMENTS(S) THAT ARE DETERMINED BY EACH LABORATORY FROM THE SOIL EXTRACT.

No.	Extracting Solution	State	Elements Determined
1	0.15N H_2SO_4	Ky.	K & P
2	1% $(NH_4)_2SO_4$ in 0.05N H_2SO_4	Tenn.	K & P
3	0.1N HCl	La.	K, Ca, Mg, Na, & NO_3
4	0.03N NH_4F in 0.1N HCl	La.	P
5	0.03N NH_4F in 0.025N HCl	Ark.	P
		Okla.	P
6	0.05N HCl + 0.025N H_2SO_4	Ala.	K, Mg & P (acid soils)
		Ga.	K, Ca, Mg, P & Zn
		N. C.	K, Ca, Mg, Na, NH_4, Mn, & P
		S. C.	K, Ca, Mg, & P
		Va.	K, Ca, Mg, & P
7	0.018N CH_3COOH	Ky.	NO_3
8	0.7N CH_3COONH_4 in 0.54N CH_3COOH buffered at pH 4.8	Fla.	K, Ca, Mg, Na, P & NO_3
9	1N CH_3COONH_4 buffered at pH 7.0	Ala.	Ca
		Ark.	K, Ca, Mg, & Na
		Miss.	Mn
		Okla.	K, Ca, Mg, & Na
		P. R.	K, Ca, Mg, & Mn
10	0.5M $NaHCO_3$ buffered at pH 8.5	Ala.	P (calcareous soils)
		Okla.	P (calcareous soils)
		P. R.	P (alkaline soils)
11	0.73N CH_3COONa in 0.5 N CH_3COOH buffered at pH 4.8	P. R.	P (acid soils)
12	1.4N CH_3COONH_4 in 1N HCl buffered at pH 4.2	Texas	K, Ca, Na, & P (nonsaline soils)
13	0.25N $CaCl_2 \cdot 2H_2O$ containing 1.5 ppm Mg	Miss.	Mg
14	0.5N CH_3COONH_4 in 0.25 N CH_3COOH	Miss.	SO_4
15	0.1 N $(NH_4)_2SO_4$ and 0.03N NH_4F in 0.15N lactic acid and 0.5N CH_3COOH and 0.25N NH_4OH buffered at pH 4.25	Miss.	K & P
16	Distilled H_2O	Fla.	Cl
		N. C.	NO_3
17	Distilled H_2O (saturated paste extract)	Okla.	Ca, Mg, K, Na, Cl, SO_4, HCO_3, CO_3 (saline soils)
		Texas	Ca, Mg, K, Na, Cl, SO_4, NO_3, B, HCO_3, CO_3 (saline soils)

concentrated NH_4OH to approximately 10 liters of distilled water. Dilute to 18 liters with distilled water and mix thoroughly. Adjust to pH 4.8 with either dilute CH_3COOH or NH_4OH as needed.

9. **1N CH_3COONH_4 buffered at pH 7.0:** (a) Add separately 1060 ml of concentrated CH_3COOH and 1260 ml of concentrated NH_4OH to approximately 10 liters of distilled water. Dilute to 18 liters with distilled water and mix thoroughly. (b) Dissolve 1387 g of CH_3COONH_4 in 18 liters of distilled water. Adjust to pH 7.0 with either dilute CH_3COOH or NH_4OH as needed.

10. **0.5M $NaHCO_3$ buffered at pH 8.5:** Dissolve 756 g of $NaHCO_3$ in 18 liters of distilled water. Adjust to pH 8.5 with 10N NaOH.

11. **0.73N CH_3COONa in 0.5 N CH_3COOH buffered at pH 4.8:** Dissolve 1800 g of $CH_3COONa \cdot 3H_2O$ in approximately 10 liters of distilled water. Then add 540 ml of glacial CH_3COOH, dilute to 18 liters with distilled water, and mix thoroughly.

12. **1.4N CH_3COONH_4 in 1N HCl buffered at pH 4.2:** Dissolve 1980 g of CH_3COONH_4 in approximately 10 liters of distilled water. Then add 1460 ml of concentrated HCl, mix, and dilute to 18 liters with distilled water. Adjust the solution to pH 4.2 with either dilute HCl or NH_4OH as needed.

13. **0.25N $CaCl_2 \cdot 2H_2O$ and 1.5 ppm Mg:** Dissolve 330.7 g of $CaCl_2 \cdot 2H_2O$ and 0.27 g of $MgSO_4 \cdot 7H_2O$ in 18 liters of distilled water.

14. **0.5N CH_3COONH_4 in 0.25N CH_3COOH:** Add 693 g of CH_3COONH_4 and 256 ml of glacial CH_3COOH to approximately 10 liters of distilled water. After the CH_3COONH_4 has dissolved, dilute to 18 liters with distilled water and mix thoroughly.

15. **0.1N $(NH_4)_2SO_4$ and 0.03N NH_4F in a mixture of 0.15N lactic acid, 0.5N CH_3COOH and 0.25N NH_4OH buffered at pH 4.25:** Add 515 ml of glacial CH_3COOH and 270 ml of 85% lactic acid to approximately 10 liters of distilled water. Then add 20 g of NH_4F, 120 g of $(NH_4)_2SO_4$, and 288 ml of concentrated NH_4OH. After the solid reagents have dissolved and the solution is cool, dilute to 18 liters with distilled water, and mix thoroughly. Adjust the pH to 4.25 with either CH_3COOH or NH_4OH as needed.

EXTRACTING PROCEDURES

Nonsaline Soils

Measure or weigh a quantity of air-dried screened soil (1 g – 20 g) into a container. Add a scoop of activated charcoal (0.1 g – 0.25 g) and then add a measured volume of extracting solution (10 ml – 250 ml). Shake the soil-extracting solution mixture (1:2 – 1:20 soil-solution ratio) in a mechanical shaker for a definite period of time (40 sec – 30 min), and filter immediately through retentive filter paper into an appropriate container. Use aliquots of the filtrate for the determination of one or more elements.

The weight of soil and volume of extracting solution along with the time of shaking that is used by each laboratory for each element are given in table 2. Unless otherwise indicated, the same soil-extracting solution ratio is used for all of the elements tested.

TABLE 2.—THE WEIGHT OF SOIL, VOLUME OF EXTRACTING SOLUTION, SOIL-SOLUTION RATIO, AND THE TIME OF SHAKING USED BY THE SOIL-TESTING LABORATORIES IN EXTRACTING NUTRIENTS FROM THE SOIL.

Laboratory	Soil	Extracting Solution	Soil:Solution Ratio	Shaking Time	Elements Determined
	g	ml		min	
Ala.	5.0	20	1:4	5	ALL
Ark.	3.0	15	1:5	5	K, Ca, Mg, & Na
	1.0	10	1:10	0.67	P
Fla.	5.0	25	1:5	30	ALL
Ga.	5.0	20	1:4	5	K & P
	5.0	20	1:4	15	Ca, Mg, & Zn
Ky.	5.0	20	1:4	2[*]	P & K
	5.0	26	1:5.2	1[*]	NO_3
La.	2.5	50	1:20	15	P
	5.0	100	1:20	15	K, Ca, Mg, Na, & NO_3
Miss.	6.25	25	1:4	15	K & P
	5.0	40	1:8	30	Mg
	10.0	25	1:2.5	30	SO_4
	10.0	20	1:2	20	Mn
N. C.	5.0	20	1:4	5	ALL
Okla.	1.0	20	1:20	1	P
	5.0	25	1:5	2[*]	K
	10.0	50	1:5	2[†]	K, Ca, Mg, & Na
P. R.	12.5	25	1:2	15	P
	20.0	250	1:12.5	15	K, Ca, Mg, & Mn
S. C.	5.0	20	1:4	5	ALL
Tenn.	5.0	20	1:4	3	ALL
Texas	2.5	25	1:10	30	ALL (Nonsaline)
Va.	5.0	20	1:4	5	ALL

[*] Shaken by hand. Oklahoma filters the following day.

[†] Shaken by hand, filtered the following day, and residue leached with an additional 50 ml of extracting solution.

Saline Soils. Oklahoma, Puerto Rico, and Texas

Slowly add distilled water to 100 – 150 g of soil until the soil is saturated (Oklahoma adds 100 ml of distilled water to 100 g soil). Constantly stir the mixture with a spatula while the water is being added. At saturation, the soil flows slightly when the container is tilted and flows freely from the spatula except for soils that are high in clay. Equilibrate the paste for a minimum of 1 hour. If free water collects on the surface or the paste markedly stiffens during this period, remix by adding more soil or distilled water as needed. After equilibration, transfer the paste to a Buchner funnel and remove the water by vacuum. Retain the filtrate for analysis.

POTASSIUM (K)

All laboratories determine the concentration of K in the soil extract with a flame photometer using either a Perkin-Elmer, Beckman Model B or a Beckman DU.

Potassium Stock Solution

Dissolve 0.3814 g of dried C.P. KCl in 1 liter of extracting solution. This solution contains 200 ppm of K.

Potassium Standard Solutions

Prepare a set of standard solutions (the range for the region is from 0 – 20 ppm K to 0 – 100 ppm K) from the stock solution. Dilute to volume with extracting solution.

General Procedure

Follow the warmup procedure outlined by the manufacturer for the type of flame photometer being used. Adjust the air or oxygen and gas to the recommended pressure and light the burner. With the wave length set for K (768 mu), adjust the instrument so that the recording needle (dial) reads 100 when the highest standard solution is atomized in the flame. Then atomize the intermediate standard solutions and prepare a standard curve by plotting the flame photometer readings vs K concentration.

When the instrument has been calibrated with known concentrations of K, atomize an aliquot of the soil extract and record the reading or soil-test rating. Recalibrate the flame photometer with one of the K standards after every 5 to 20 soil samples.

Instrument Settings

Variations in instrument settings and the range of K concentrations that are used for the determination of extractable K by each laboratory are summarized in table 3.

TABLE 3.—INSTRUMENT SETTINGS AND THE RANGE OF POTASSIUM CONCENTRATIONS DETERMINED BY THE SOIL-TESTING LABORATORIES.

Instrument Settings	PERKIN – ELMER								BECKMAN – B		BECKMAN – DU				
	Ala.	Ga.	La.*	Miss.†	N. C.	S. C.	Tenn.	Va.	Fla.	Texas (2)	Ark.	Ky.	Okla.	Puerto Rico	Texas (1)
Acetylene – psi	5			0.5		3			5	4	4				3
Hydrogen – psi												5	4	4	
Propane – psi		4	8		4		5	3							
Air – psi	10	10	10	10	10	10	10	4							
Oxygen – psi									12	15	13	10	10	14	12.5
Phototube	Red	Red	Red	Red	Red	Red	Red	Red	Red	Red	Red	Red	Red	Red	Red
Slit Width – mm									0.3	0.1		0.2	0.19	.05–.1	0.03
Selector Switch											1.0	0.1	0.1	0.1	0.1
Zero Suppression											Off	0		Off	2
Load Resistor											3	2	1	3	3
Sensitivity									3		Off	8	Off	Off	Off
Resistor – ohms									10		3				
Procedure	D.R.	D.R.	I.S.	D.R.	D.R.	D.R.	D.R.	D.R.							
K Standards – ppm	0–50	0–50	0–20	0–80	0–29.3	0–29.3	0–50	0–39	0–60	0–19.5	0–100	0–100	0–80	0–50	0–50
Soil:Extract Ratio	1:4	1:4	1:20	1:4	1:4	1:4	1:4	1:4	1:5		1:5	1:4	1:5	1:12.5	1:10
K in Soil – lb/A	0–400	0–400	0–800	0–640	0–234	0–234	0–400	0–312	0–600		0–1000	0–800	0–800	0–1250	0–1000

D.R. = Direct Reading I.S. = Internal Standard (1) = Nonsaline Soils (2) = Saline Soils

* Louisiana adds 2 ml of a 6500 ppm Li solution to 15 ml of the soil extract and standard K solutions. This gives a Li concentration of 765 ppm in the atomized solutions.

† Since the Mississippi extracting solution is 0.03N to F, it is essential for health reasons that the fumes from the flame be removed from the laboratory. Pressure of gas (acetylene, hydrogen, or propane) and air or oxygen is dependent upon the type of burner being used.

Calculations

The concentration of K in the soil extract is obtained by converting the readings of the flame photometer to ppm K from the standard curve. Lb of K per acre = ppm K in the soil extract × dilution factor × 2. Texas reports K concentration of saline samples as me per ml of saturated extract or irrigation water.

PHOSPHORUS (P)

All of the laboratories determine the concentration of P in the extraction solution colorimetrically. Four states (Alabama, North Carolina, South Carolina, and Virginia) estimate P from the intensity of the yellow color formed by the unreduced vanadium phosphomolybdic complex. The other state laboratories estimate P from the intensity of the blue color developed by the selective reduction of the phosphomolybdic complex in an acid medium. Modifications in reducing agent and acid system of the general procedure used by the state laboratories are:

Reduction with $SnCl_2 \cdot 2H_2O$ in a H_2SO_4 system.

Reduction with $SnCl_2 \cdot 2H_2O$ in a HCl system.

Reduction with 1, 2, 4-aminonaphthol sulfonic acid in a HCl system.

Reduction with 1, 2, 4-aminonaphthol sulfonic acid in a H_2SO_4 system.

Since the sensitivity to concentrations of P varies with the method, reducing agent, and acid system, each of the methods is described separately.

Vanadate Method

Alabama, North Carolina, South Carolina, and Virginia

Phosphorus Stock Solution

Dissolve 0.1098 g of dried C.P. KH_2PO_4 in 1 liter of extracting solution. This solution contains 25 ppm of P.

Phosphorus Standard Solutions

Prepare a set of standard solutions ranging from 0 – 25 ppm P from the stock solution. Dilute to volume with extracting solution.

Reagents

1. **Ammonium Molybdate:** Dissolve 50 g of ammonium molybdate in 1 liter of distilled water.
2. **Ammonium Vanadate:** Dissolve 2.5 g of NH_4VO_3 in 1 liter of 1:1 HNO_3.

3. For color development, mix equal volumes of the ammonium molybdate and NH_4VO_3 solutions. Prepare a fresh mixture each week.

Procedure

Add 4 ml of the soil extract (standard solution) to a glass vial. Then add 1 ml of reagent #3 (ammonium molybdate-ammonium vanadate mixture), mix thoroughly, and allow the color to develop for a minimum of 20 minutes. Prepare a blank with each group of samples by adding 1 ml of reagent #3 to 4 ml of the extracting solution.

Adjust the spectrophotometer (colorimeter) to 100 percent transmission at 450 mu (420 mu filter) with the blank. Read the percent transmission of the soil extract and convert percent transmission to ppm P in the extract by means of the standard curve.

Reduction Method

STANNOUS CHLORIDE IN A SULFURIC ACID SYSTEM

Florida, Kentucky, and Louisiana

Phosphorus Stock Solution

Dissolve 0.4393 g of dried C.P. KH_2PO_4 in 1 liter of distilled water. This solution contains 100 ppm of P. Dilute 50 ml of the stock solution to 1 liter with distilled water for a working stock solution of 5 ppm P.

Phosphorus Standard Solutions

Measure 0, 1, 2, 4, 6, 8, and 10 ml of the 5 ppm P solution into 50 ml volumetric flasks, add 5 ml of extracting solution, and proceed as directed in the section on procedure.

FLORIDA

Reagents

1. **Stannous Chloride:** Dissolve 25 g of $SnCl_2 \cdot 2H_2O$ in 100 ml of concentrated HCl. Prepare daily a working solution by diluting 5 ml of the concentrated solution and 5 ml of concentrated HCl to 200 ml with distilled water.
2. **Ammonium Molybdate:** Dissolve 25 g of ammonium molybdate in warm distilled water. Dilute 280 ml of concentrated H_2SO_4 to 600 ml with distilled water. After the solutions have cooled, slowly add the ammonium molybdate solution, stirring constantly, to the H_2SO_4 solution. Dilute to 1 liter with distilled water.

Procedure

To a measured 5 ml of the soil extract add 25 ml of distilled water, 5 ml of the ammonium molybdate solution, and 0.5 ml of the dilute $SnCl_2 \cdot 2H_2O$ solution, mixing well after each addition. Make to a volume of 50 ml with distilled water and mix. Prepare a blank with each group of samples using 5 ml of extracting solution instead of 5 ml of the soil extract. Allow the samples to stand for 15 minutes before reading.

Adjust the colorimeter to 100 percent transmission with the blank using a 650 mu filter. Read the percent transmission of the soil extract and convert percent transmission to ppm P in the soil extract by means of the standard curve.

KENTUCKY

Reagents

1. **Stannous Chloride:** Dissolve 4 g of $SnCl_2 \cdot 2H_2O$ in 25 ml of concentrated HCl. Dilute 1 ml of the concentrated solution to 40 ml with distilled water.
2. **Ammonium Molybdate:** Dissolve 9.06 g of ammonium molybdate in approximately 250 ml of distilled warm water (not above 50° C). Add 175 ml of 10N H_2SO_4, cool to at least 30° C, and dilute to 1 liter with distilled water.

Procedure

To a measured 5 ml of the soil extract, add 10 ml of the ammonium molybdate solution and 2.5 ml of the dilute stannous chloride solution, mixing well after each addition. From 3-7 minutes after adding the stannous chloride, compare the intensity of the blue color developed with standard color slides.

LOUISIANA

Reagents

1. **Stannous Chloride:** Dissolve 25 g of $SnCl_2 \cdot 2H_2O$ in 100 ml of concentrated HCl and dilute to 1 liter with distilled water. Store in a dispensing bottle and cover with a layer of mineral oil to prevent contact with the air.
2. **Ammonium Molybdate:** Dissolve 5 g of ammonium molybdate in warm distilled water. Dilute 55 ml of concentrated H_2SO_4 to 800 ml with distilled water. After the two solutions have cooled, slowly add the ammonium molybdate solution to the H_2SO_4 solution with constant stirring. Dissolve 40 g of H_3BO_3 in the combined solutions and dilute to 1 liter with distilled water.

Procedure

To a measured 5 ml aliquot of the soil extract, add 35 ml of distilled water, 10 ml of the ammonium molybdate solution, and 3 drops of the $SnCl_2 \cdot 2H_2O$ solution, mixing thoroughly after each addition. Prepare a blank with each group of samples using 5 ml of the extracting solution instead of the soil extract.

Adjust the spectrophotometer to 0 optical density at 650 mu with the blank. Read the optical density of the soil extract and convert optical density to ppm P in the soil extract by means of the standard curve.

STANNOUS CHLORIDE IN A HYDROCHLORIC ACID SYSTEM

Georgia, Oklahoma, and Texas

Phosphorus Stock Solution

Dissolve 0.2225 g of $NaH_2PO_4 \cdot H_2O$ in approximately 400 ml of distilled water. Add 5 ml of concentrated H_2SO_4 and dilute to 1 liter with distilled water. This solution contains 50 ppm P.

Phosphorus Standard Solutions

1. **Georgia:** Prepare a set of standard solutions ranging from 0 – 2 ppm P from the stock solution.
2. **Texas:** Dilute 30 ml of the stock solution to 500 ml with distilled water for a working stock solution of 3 ppm P. Measure from 0 – 10 ml of the 3 ppm P solution into 25 ml volumetric flasks, add 2.5 ml of extracting solution, and proceed as directed in the section on procedure.

GEORGIA

Reagents

1. **Stannous Chloride:** Dissolve 25 g of $SnCl_2 \cdot 2H_2O$ in 100 ml of concentrated HCl and dilute to 1 liter with distilled water. Store in a dispensing bottle and cover with a layer of mineral oil to prevent contact with the air.
2. **Ammonium Molybdate:** Dissolve 100 g of ammonium molybdate in 850 ml of distilled water. Add 160 ml of distilled water to 1700 ml of concentrated HCl. Add the ammonium molybdate solution, while stirring constantly, to the HCl solution. Dilute 16.7 ml of the ammonium molybdate solution to 1 liter with extracting solution.

Procedure

Add 9 ml of the dilute ammonium molybdate solution to 1 ml of soil extract in a vial and mix thoroughly. Add 1 drop of the

$SnCl_2 \cdot 2H_2O$ solution and mix. Prepare a blank with each group of samples by using 1 ml of extracting solution instead of 1 ml of the soil extract.

Adjust the colorimeter to 100 percent transmission with the blank using a 650 mu filter. Read the percent transmission of the soil extract and convert to ppm P in the soil extract by means of the standard curve.

OKLAHOMA

Reagents

1. **Stannous Chloride:** Dissolve 10 g of $SnCl_2 \cdot 2H_2O$ in 25 ml of concentrated HCl. Store under a layer of xylene. Dilute 1 ml of the stock solution to 300 ml with distilled water each day.
2. **Ammonium Molybdate:** Dissolve 15 g of ammonium molybdate in approximately 300 ml of distilled water. Add 291 ml of concentrated HCl and dilute to 1 liter with distilled water. Store in brown bottle, in the dark.

Procedure

Measure 2 ml of the soil extract into a vial. Add 5 ml of distilled water, 2 ml of the ammonium molybdate solution, and 1 ml of the dilute $SnCl_2 \cdot 2H_2O$ solution. Mix thoroughly after each addition and allow the color to develop for 30 minutes. Prepare a blank with each group of samples by using 2 ml of extracting solution instead of 2 ml of the soil extract.

Adjust the spectrophotometer to 100 percent transmission at 700 mu with the blank. Read the percent transmission of the soil extract and convert to ppm P in the soil extract by means of the standard curve.

TEXAS

Reagents

1. **Stannous Chloride:** Dissolve 10 g of $SnCl_2 \cdot 2H_2O$ in 25 ml of concentrated HCl. Store under a layer of mineral oil. Dilute 1 ml of the stock solution to 250 ml with distilled water each day.
2. **Ammonium Molybdate:** Dissolve 60 g of ammonium molybdate in approximately 2 liters of distilled water. Add 1140 ml of concentrated HCl and dilute to 4 liters with distilled water.

Procedure

Add 12.5 ml of distilled water to 2.5 ml of the soil extract in a test tube. Add 5 ml of the ammonium molybdate solution and 5 ml of the dilute $SnCl_2 \cdot 2H_2O$ solution. Mix thoroughly after each addition and allow the color to develop for 5 minutes. Prepare a blank with each group of samples by using 2.5 ml of the extracting solution instead of the soil extract.

Adjust the spectrophotometer to 100 percent transmission at 665 mu with the blank. Read the percent transmission of the soil extract and convert to ppm P in the soil extract by means of the standard curve.

1, 2, 4-AMINONAPHTHOL SULFONIC ACID IN A HYDROCHLORIC ACID SYSTEM. Arkansas, Mississippi, and Tennessee

Phosphorus Stock Solution

Dissolve 0.4393 g of KH_2PO_4 in 1 liter of extracting solution. This solution contains 100 ppm P.

Phosphorus Standard Solutions

Prepare a set of standard solutions ranging from 0 – 20 ppm P from the stock solution. Make to volume with extracting solution.

ARKANSAS AND TENNESSEE

Reagents

1. **1, 2, 4-Aminonaphthol Sulfonic Acid:** Mix together 5 g of Na_2SO_3, 146.25 g of $Na_2S_2O_5$, and 2.5 g of 1, 2, 4-aminonaphthol sulfonic acid and grind to a fine powder. Dissolve 8 g of the mixture in 50 ml of warm distilled water and allow to stand for 12 hours. Prepare a fresh solution after 21 days.
2. **Ammonium Molybdate:**
    a. **Arkansas:** Dissolve 50 g of ammonium molybdate in 500 ml of distilled water. Add slowly 850 ml of concentrated HCl and dilute, when cool, to 2 liters with distilled water.
    b. **Tennessee:** Dissolve 15 g of ammonium molybdate in 300 ml of distilled water. Add slowly and with constant stirring 300 ml of concentrated HCl. After cooling, dilute to 1 liter with distilled water.

Procedure

Measure 5 ml of the soil extract into a vial. Add 5 drops of the ammonium molybdate solution and mix. Add 5 drops of the 1, 2, 4-aminonaphthol sulfonic acid reagent and mix immediately. Allow the color to develop for 10 minutes.

Prepare a blank with each group of samples by using 5 ml of the extracting solution instead of the soil extract. Adjust the spectrophotometer to 100 percent transmission at 625 mu with the blank. Read the percent transmission of the soil extract and convert to ppm P in the soil extract by means of the standard curve.

Reagents

1. **Boric Acid:** Dissolve 50 g of H_3BO_3 in 1 liter of distilled water.
2. **Ammonium Molybdate:** Dissolve 10 g of ammonium molybdate in 400 ml of distilled water. Add slowly 172 ml of concentrated HCl and 100 ml of H_3BO_3 solution. When cool, dilute to 1 liter with distilled water.
3. **1, 2, 4-Aminonaphthol Sulfonic Acid:** Mix together 1070 g of $Na_2S_2O_5$ (or 1170 g of $NaHSO_3$), 20 g of Na_2SO_3, and 20 g of 1, 2, 4-aminonaphthol sulfonic acid and grind to a fine powder. Dissolve 111 g of the mixture in 685 ml of warm (50-55° C) distilled water. Let stand for several hours, filter, and store in an amber-colored bottle. Prepare a new solution after 14 days.

Procedure

Add 6 ml of the ammonium molybdate solution to 3 ml of the soil extract in a 10 ml vial and mix. Add 0.5 ml of the 1, 2, 4-aminonaphthol sulfonic acid and mix. Prepare a blank and a standard (15 ppm P) with each set of samples in the same manner as the soil extracts.

Adjust the colorimeter to 100 percent transmission with the blank using a 660 mu filter. When the color intensity of the standard has developed so that the percent transmission is equal to that used for the standard curve (15-20 minutes), read the percent transmission of the soil extracts and convert to ppm P in the soil extract by means of the standard curve.

1, 2, 4-AMINONAPHTHOL SULFONIC ACID IN A SULFURIC ACID SYSTEM

PUERTO RICO

Phosphorus Stock Solution

Dissolve 0.4393 g of KH_2PO_4 in 1 liter of extracting solution. This solution contains 100 ppm P.

Phosphorus Standard Solutions

Prepare a set of standard solutions ranging from 0-20 ppm P from the stock solution. Make to volume with extracting solution.

Reagents

1. **1, 2, 4-Aminonaphthol Sulfonic Acid:** Dissolve 150 g of $NaHSO_3$ (or 137 g of $Na_2S_2O_5$) and 5 g of Na_2SO_3 in approximately 800 ml of distilled water. Dissolve 25 g of 1, 2, 4-amino-

naphthol sulfonic acid in the sulfite solution which has been heated to 50° C. Dilute to 1250 ml with distilled water, filter, and store in an amber-colored bottle in a refrigerator.

2. **Ammonium Molybdate:** Dissolve 7.5 g of ammonium molybdate in approximately 500 ml of distilled water. Add slowly and with constant stirring 53 ml of concentrated H_2SO_4. After cooling, dilute to 1 liter with distilled water.

Procedure

Phosphorus is determined with a Technicon Autoanalyzer. Pour the soil extract into a disposable polyethylene vial. The instrument automatically mixes a selected volume of soil extract and ammonium molybdate. The mixture is then injected into and mixed with a selected volume of 1, 2, 4-aminonaphthol sulfonic acid. The colored solution is heated to 95° C, passed through a colorimeter, and the percent transmission is registered on a recorder. Run a set of standard P solutions with each group of soil samples. Convert the percent transmission of the soil extract to ppm P in the soil by means of the standard curve.

Calculation

Lb of P per acre = ppm P in the soil extract × dilution factor × 2.

CALCIUM (Ca)

Kentucky, Mississippi, and Tennessee do not determine extractable Ca on routine soil samples. Georgia determines Ca upon request. Three of the state laboratories (North Carolina, South Carolina, and Virginia) precipitate Ca as the oxalate and measure photometrically the amount of turbidity produced. Georgia uses an atomic absorption spectrophotometer to measure Ca. The other state laboratories determine the concentration of Ca in the soil extract with a flame photometer. On saline soils and irrigation water, Texas determines the concentration of Ca by titrating with EDTA using ammonium purpurate as the indicator and Oklahoma removes sulfates with $BaCl_2$ before determining Ca with a flame photometer.

Calcium Stock Solution

Dissolve either 4.9946 g of $CaCO_3$ or 7.35 g of $CaCl_2 \cdot 2H_2O$ in 1 liter of extracting solution. This solution contains 2,000 ppm Ca.

Calcium Standard Solutions

Prepare a set of standard solutions (the range for the region is from 0 – 50 ppm Ca to 0 – 1,000 ppm Ca) from the stock solution. Make to volume with extracting solution.

Flame Photometer

Alabama, Arkansas, Florida, Louisiana, Oklahoma, Puerto Rico, and Texas—nonsaline

General Procedure

Follow the warmup procedure outlined by the manufacturer for the type of flame photometer being used. Adjust the air or oxygen and gas to the recommended pressure and light the burner. With the wave length set for Ca (622 mu, 554 mu, 420 mu or 5675 A°), adjust the instrument so that the recording needle (dial) reads 100 when the highest standard solution is atomized in the flame. Then atomize the intermediate standard solutions and prepare a standard curve by plotting the flame photometer readings vs Ca concentration.

After the instrument has been calibrated with known concentrations of Ca, atomize an aliquot of the soil extract and record the reading or soil-test rating. Recalibrate the flame photometer with one of the Ca standards after every 5 to 20 soil samples. Convert photometer readings to ppm Ca in the soil extract from the calcium standard curve.

Instrument Settings

Variations in instrument settings and the range of Ca concentrations that are used for the determination of extractable Ca by each laboratory using flame photometry are summarized in table 4.

Atomic Absorption Spectrophotometer

Georgia

Procedure

After installing the Ca cathode lamp, select the operating parameters (table 4) for the Ca lamp and set the instrument controls to the values suggested by the manufacturer.

Follow the warmup procedure outlined by the manufacturer. Adjust the air and acetylene to the recommended pressure and light the burner. Adjust the reference and sample beams to approximately 100 microamperes while atomizing extracting solution into the burner. Turn response switch of ratiometer on and adjust to 0 with the zero adjuster. With the wave length at 0.4227 microns, atomize the standard solutions and prepare a standard curve by plotting readings vs Ca concentration.

When the instrument has been calibrated with known concentrations of Ca, atomize an aliquot of the soil extract (containing 1% La as La_2O_3) and record the reading or soil-test rating. Recalibrate the instrument with one of the Ca standards after every tenth soil sample.

TABLE 4.—INSTRUMENT SETTINGS AND THE RANGE OF CALCIUM CONCENTRATIONS DETERMINED BY THE SOIL-TESTING LABORATORIES.

Instrument Settings	PERKIN – ELMER		BECK-MAN – B	BECKMAN – DU				PERKIN – ELMER ATOMIC ABSORPTION
	Ala.	La.*	Fla.	Ark.	Okla.	P. R.	Texas	Ga.
Acetylene – psi	5	4	5	4			3	8
Hydrogen – psi					4	4		
Air – psi	10	10						22
Oxygen – psi			12	13	10	14	12.5	
Phototube	Blue	Blue	Red	Blue	Blue		Red	Ca-cathode
Sensitivity			4	3	7–8	3	3	
Slit width – mm			3.6		0.06	0.1–0.2	0.1	0.14
Selector Switch				1.0	0.1	0.1	0.1	
Zero Suppression				Off		Off	2	
Resistor – ohm			10	2.0	2.0	2.0		
Procedure	D.R.	I.S.						
Ca Standards – ppm	0–240	0–200	0–400	0–1000	0–200	0–500	0–500	0–50
Soil: Extract ratio	1:4	1:20	1:5	1:5	1:5	1:12.5	1:10	1:4
Ca in Soil – lb/A	0–1920	0–8000	0–4000	1–10000	0–2000	1–12500	0–10000	0–200

D.R. = Direct Reading I.S. = Internal Standard

* Louisiana adds 2 ml of an 850 ppm Li standard to 15 ml of the soil extract and standard Ca solutions. This gives a Li concentration of 100 ppm in the atomized solutions.

Pressure of gas (acetylene or hydrogen) and air or oxygen is dependent upon the type of burner being used.

Calcium Oxalate

North Carolina, South Carolina, and Virginia

Reagents

1. **Oxalic Acid:** Dissolve 6 g of $H_2C_2O_4 \cdot 2H_2O$ and 2.5 g of CH_3COONH_4 in approximately 700 ml of distilled water. Add 50 ml of triethanolamine and dilute to 1 liter with distilled water.

Procedure

Add 1 ml of $H_2C_2O_4 \cdot 2H_2O$ solution to a glass vial. Then add 4 ml of the soil extract (standard solution) and mix. Prepare a blank with each group of samples by adding 1 ml of oxalic acid solution to 4 ml of the extracting solution.

Adjust the spectrophotometer (colorimeter) to 100 percent transmission at 420 mu with the blank. After 10 – 30 minutes and no longer than 2 hours, mix the suspension by inverting the vial and shaking vigorously. Measure the light transmission of the turbid suspension and convert percent transmission to ppm Ca in the soil extract from the standard curve.

EDTA

Texas, saline soils and irrigation water

Reagents

1. **4N Sodium Hydroxide:** Dissolve 160 g of NaOH in 1 liter of distilled water.
2. **Ammonium Purpurate (Murexide) Indicator:** Thoroughly mix 0.5 g of murexide with 100 g of powdered K_2SO_4.
3. **0.01N EDTA:** Dissolve 1.861 g of disodium dihydrogen ethylenediamine tetra-acetic acid and 0.05 g of $MgCl_2 \cdot 6H_2O$ in 1 liter of distilled water.

Procedure

Pipette an aliquot of the soil extract or water containing less than 0.1 me of Ca into an Erlenmeyer flask. Dilute to around 25 ml with distilled water, and add 5 drops of NaOH and 50 mg of ammonium purpurate indicator. Titrate with EDTA solution until the color changes from orange-red to purple.

Calculations

Lb of Ca per acre = ppm Ca in the soil extract × dilution factor × 2.
Me of Ca per ml = N of EDTA × ml of EDTA ÷ ml of aliquot.

MAGNESIUM (Mg)

Kentucky and Tennessee do not determine extractable Mg on routine soil samples. The other states determine Mg either upon request, by physiographic regions, or on all samples. Alabama and Georgia determine Mg with a Perkin-Elmer atomic absorption spectrophotometer. Florida, Oklahoma, and Puerto Rico determine the concentration of Mg in the soil extract with a flame photometer. On saline soils and irrigation water, Texas determines Mg by titrating with EDTA using eriochrome black T as the indicator. The other state laboratories determine Mg by the intensity of the colored lake formed between Mg and thiazol yellow in an alkaline medium.

Magnesium Stock Solutions

1. **Alabama and Louisiana:** Dissolve 1 g of Mg ribbon in 400 ml of distilled water containing 20 ml of concentrated HCl and dilute to 1 liter with distilled water. Dilute 50 ml of the 1,000 ppm Mg solution to 1 liter with extracting solution. The stock solution contains 50 ppm Mg.
2. **Arkansas:** Dissolve 0.2205 g of $Mg(C_2H_3O_2)_2 \cdot 4H_2O$ in 1 liter of extracting solution. This solution contains 25 ppm Mg.
3. **Florida:** Dissolve 1.6579 g of MgO in 1 liter of extracting solution. This solution contains 1,000 ppm Mg.
4. **Georgia, North Carolina, Oklahoma, South Carolina, and Virginia:** Dissolve 1.2163 g of $MgSO_4 \cdot 7H_2O$ in 1 liter of extracting solution. This solution contains 120 ppm Mg.
5. **Mississippi:** Dissolve 1.0136 g of $MgSO_4 \cdot 7H_2O$ in 1 liter of extracting solution. This solution contains 100 ppm Mg.
6. **Puerto Rico:** Dissolve 10.544 g of $Mg(NO_3)_2 \cdot 6H_2O$ in 1 liter of extracting solution. This solution contains 1,000 ppm Mg.
7. **Texas:** Dissolve 0.0953 g of $MgCl_2$ in 1 liter of distilled water. This solution contains 15 ppm Mg.

Magnesium Standard Solutions

Prepare a set of standard solutions (the range for the region is from 0 – 3 ppm Mg to 0 – 200 ppm Mg) from the stock solution. Dilute to volume with extracting solution.

Flame Photometer

Florida, Oklahoma, and Puerto Rico

Instrument Settings (Beckman-DU)	**Fla. & Okla.**	**P. R.**
oxygen	10 psi	14 psi
hydrogen	5 psi	4 psi
sensitivity	5	3
photomultiplier	full	full
resistor	2	2
slit width	0.03 – 0.06 mm	2 – 3 mm

General Procedure

Follow the warmup procedure outlined by the manufacturer for the type of flame photometer being used. Adjust the oxygen and gas to the recommended pressure and light the burner. With the wave length set for Mg (285.2 or 371 mu), adjust the instrument so that the recording needle (dial) reads 100 when the highest standard solution is atomized in the flame. Then atomize the intermediate standard solutions and prepare a standard curve by plotting the flame photometer readings vs Mg concentration.

When the instrument has been calibrated with known concentrations of Mg, atomize an aliquot of the soil extract and record the reading or soil-test rating. Recalibrate the flame photometer with one of the Mg standards after every 5 to 10 soil samples. Convert photometer readings to ppm Mg in the soil extract from the Mg standard curve.

Thiazol Yellow

Arkansas, Louisiana, Mississippi, North Carolina, South Carolina, and Virginia

Reagents

1. **Compensating Solution:** The quantity of reagents used by each state to prepare 1 liter of compensating solution is:

Reagents	La.	Miss.	N. C.	S. C.	Va.
$CaCl_2 \cdot 2H_2O$	1.5 g		2.0 g	3.4 g	0.4 g
$NH_2OH \cdot HCl$	2.0 g	2.0 g	1.0 g	2.0 g	1.0 g
$AlCl_3 \cdot 6H_2O$	0.5 g		0.5 g	1.0 g	0.2 g
$MnCl_2 \cdot 4H_2O$				0.6 g	
$MgSO_4 \cdot 7H_2O$ (120 ppm)			50 ml	50 ml	
Triethanolamine	100 ml	60 ml	400 ml	400 ml	50 ml
NH_4Cl		5.4 g			
EDTA		21.9 g			

Dissolve the reagents in distilled water. Add the triethanolamine, and, after mixing, dilute the solution to 1 liter with distilled water.

2. **Thiazol Yellow:**
   a. **Arkansas:** Dissolve 0.1 g of thiazol yellow in 100 ml of distilled water. Filter and store in an amber-colored bottle.

b. **Louisiana:** Dissolve 0.75 g of thiazol yellow and 0.5 g of krilium in 1 liter of distilled water.

c. **Mississippi:** Dissolve 0.225 g of thiazol yellow, 1.0 g of sodium polyacrylate, 0.75 g of gum arabic, and 2.0 g of $NH_2OH \cdot HCl$ in approximately 500 ml of distilled water. Add 20 ml of triethanolamine, mix, and dilute to 1 liter with distilled water.

d. **North Carolina and South Carolina:** Dissolve 5 g of polyvinyl alcohol in approximately 500 ml of distilled water by heating on a steam bath. When cool, add 0.1 g of thiazol yellow and dilute to 1 liter with distilled water.

e. **Virginia:** Dissolve 0.3 g of thiazol yellow in 1 liter of distilled water. Dilute 20 ml to 500 ml with distilled water.

3. **Sodium Hydroxide:**

a. **Arkansas:** Dissolve 100 g of NaOH in 1 liter of distilled water.

b. **Louisiana:** Dissolve 80 g of NaOH in 1 liter of distilled water.

c. **Mississippi:** Dissolve separately 100 g of NaOH and 5 g of $Na_2S_2O_5 \cdot 5H_2O$ in distilled water. Mix and add 1 g of NaCN. Dilute to 1 liter with distilled water.

d. **North Carolina and South Carolina:** Dissolve 50 g of NaOH in 1 liter of distilled water.

e. **Virginia:** Dissolve 300 g of NaOH in 1 liter of distilled water. Dilute 50 ml to 500 ml with distilled water.

4. **Lactic Acid Solution:**

a. **Mississippi:** 1:1 solution of lactic acid in distilled water.

5. **Hydroxylamine Hydrochloride:**

a. **Arkansas:** Dissolve 25 g of $NH_2OH \cdot HCl$ in 500 ml of distilled water.

6. **Gum Arabic:**

a. **Arkansas:** Dissolve 4 g of gum arabic in warm distilled water. Dilute to 200 ml with distilled water and add a crystal of thymol.

Procedure

1. **Arkansas:** Measure 2 ml of the soil extract (standard solution) into a glass vial. Add 2 drops of $NH_2OH \cdot HCl$, 2 drops of gum arabic, 5 drops of thiazol yellow, and 5 ml of NaOH in the order listed. Mix well after each addition. Allow the color to develop for 10 minutes and compare with a series of stand-

ards (0, 5, 10, 15, 20, and 25 ppm Mg) which have been treated similar to the soil extract.

2. **Louisiana:** Add 21 ml of distilled water to 15 ml of the soil extract. Then add 2 ml of compensating solution, 2 ml of thiazol yellow, and 10 ml of NaOH solution in the order listed. Mix thoroughly after each addition. Prepare a blank with each group of samples by treating 15 ml of extracting solution in a manner similar to the soil extract.
3. **Mississippi:** Measure 3 ml of the soil extract (standard solution) into a glass vial. Add 1 drop of lactic acid, 5 ml of compensating solution and 5 ml of thiazol yellow, mix, and add dropwise 2 ml of the NaOH solution. Allow the color to develop for 15 – 20 minutes. Prepare a blank with each group of samples by treating 3 ml of the extracting solution in a manner similar to the soil extract.
4. **North Carolina and South Carolina:** Measure 1 ml of the soil extract (standard solution) into a glass vial. Add 4 ml of a 50-50 mixture of compensating solution and thiazol yellow (mixed just prior to use). Then add 1 ml of NaOH, mix well, and allow the mixture to stand for 1 hour. Prepare a blank with each group of samples by adding 4 ml of the 50-50 mixture of compensating solution and thiazol yellow and 1 ml of NaOH to 1 ml of extracting solution.
5. **Virginia:** Measure 1 ml of the soil extract (standard solution) into a glass vial. Add 2 ml of compensating solution and 2 ml of a 50-50 mixture of the diluted thiazol yellow and NaOH solutions. Mix and read the color developed within 5 minutes. Prepare a blank with each group of samples by adding 2 ml of compensating solution and 2 ml of the 50-50 mixture of thiazol yellow and NaOH to 1 ml of extracting solution.
6. **General** (all states): If a precipitate forms when the NaOH is added, dilute the soil extract with extracting solution, and repeat the test using the same volume of the diluted soil extract as that used originally.

   Adjust the spectrophotometer (colorimeter) to 100 percent transmission at 540-550 mu (530 mu filter) with the blank. Just prior to reading, remix the solution thoroughly, read the percent transmission, and convert percent transmission to ppm Mg in the extract by means of the standard curve.

EDTA

Puerto Rico and Texas, saline soils and irrigation water

Reagents

1. **Ammonium Chloride – Ammonium Hydroxide Buffer:** Dissolve 67.5 g of NH_4Cl in 570 ml of concentrated NH_4OH and dilute to 1 liter with distilled water.
2. **Eriochrome Black T Indicator:** Dissolve 0.5 g of indicator and 4.5 g of $NH_2OH \cdot HCl$ in 100 ml of methanol.
3. **0.01N EDTA:** Dissolve 1.8619 g of disodium dihydrogen ethylenediamine tetra-acetic acid in 1 liter of distilled water.

Procedure

Pipette an aliquot of the soil extract containing less than 0.1 me of Ca plus Mg into an Erlenmeyer flask. Dilute to around 25 ml with distilled water, add 10 drops of the $NH_4Cl - NH_4OH$ buffer and 4 drops of eriochrome black T indicator. Titrate with EDTA until the color changes from wine-red to blue.

Atomic Absorption Spectrophotometer

Alabama and Georgia

Procedure

After inserting the Mg cathode lamp into the instrument according to the manufacturer's instructions, adjust the wavelength to 0.2852 microns and the slit width to 1.0 mm.

Follow the warmup procedure outlined by the manufacturer. Adjust the air pressure to 22 psi and acetylene pressure to 8 psi and light the burner. Adjust reference and sample beams to approximately 100 microamperes while atomizing extracting solution into the burner. Turn response switch of ratiometer to on and adjust to 0 with the zero adjuster. Atomize the standard solutions (0 – 3 ppm, Alabama and 0 – 4 ppm, Georgia) and prepare a standard curve by plotting readings vs Mg concentration.

When the instrument has been calibrated with known concentrations of Mg, atomize an aliquot of the soil extract and record the reading or soil-test rating. Recalibrate the instrument with one of the Mg standards after every tenth soil sample.

Calculations

Lb of Mg per acre = ppm Mg in the soil extract × dilution factor × 2.

Me of Mg per ml = (N of EDTA × ml of EDTA ÷ ml of aliquot) – me of Ca per ml.

SODIUM (Na)

Arkansas, Florida, Louisiana, North Carolina, Oklahoma, and Texas determine extractable Na. Oklahoma and Texas also determine Na on saline soils and irrigation water for salinity interpretation. All of these laboratories determine the concentration of Na in the extract with a flame photometer.

Sodium Stock Solution

Dissolve 5.0836 g of dried C.P. NaCl in 1 liter of extracting solution. This solution contains 2,000 ppm Na.

Sodium Standard Solutions

Prepare a set of standard solutions from the stock solution. Dilute to volume with extracting solution.

Procedure

Follow the warmup procedure outlined by the manufacturer for the type of flame photometer being used. Adjust the air or oxygen and gas to the recommended pressure and light the burner. With the wave length set for Na (589 mu), adjust the instrument so that the recording needle (dial) reads 100 when the highest standard solution is atomized in the flame. Then atomize the intermediate standard solutions and prepare a standard curve by plotting the flame photometer readings vs Na concentration.

When the instrument has been calibrated with known concentrations of Na, atomize an aliquot of the soil extract and record the reading or soil-test rating. Recalibrate the flame photometer with one of the Na standards after every 5 to 20 soil samples. Convert photometer readings to ppm Na in the soil extract by means of the standard curve.

Instrument Settings

Variations in instrument settings and the range of Na concentrations that are used to determine extractable Na by each laboratory are summarized in table 5.

Calculations

Lb of Na per acre = ppm Na in soil extract × dilution factor × 2.

Texas reports Na concentration of saline samples as me per ml of saturated extract or irrigation water.

TABLE 5.—INSTRUMENT SETTINGS AND THE RANGE IN SODIUM CONCENTRATIONS DETERMINED BY THE SOIL-TESTING LABORATORIES.

Instrument Settings	PERKIN – ELMER		BECKMAN – B		BECKMAN – DU		
	La.*	N. C.	Fla.	Texas–Saline	Ark.	Okla.	Texas
Hydrogen – psi						4	
Acetylene – psi			5	4	2		3
Propane – psi	8	4					
Air – psi	10	10					
Oxygen – psi			10	15	12	10	12.5
Procedure	I.S.	D.R.					
Sensitivity			4		Full	6	
Phototube	Red	Red	Red	Red	Blue	Blue	Red
Slit Width – mm			1:5	4		0.01	0.04
Zero Suppression					1		2
Selector Switch					1	0.1	0.1
Na Standards – ppm	0–20	0–17.25	0–560	0–1380		0–200	0–250
Soil: Extract Ratio	1:20	1:4	1:5		1:5	1:5	1:10
Na in Soil – lb/A	0–800	0–138	0–5600			0–2000	0–5000

D.R. = Direct Reading I.S. = Internal Standard

[*] Louisiana adds 2 ml of a 6500 ppm Li solution to 15 ml of the soil extract and standard Na solutions. This gives a Li concentration of 765 ppm in the atomized solutions.

Pressure of gas (acetylene, hydrogen, or propane) and air or oxygen is dependent upon the type of burner being used.

ORGANIC MATTER

Alabama and Tennessee do not determine organic matter on routine soil samples. Florida and South Carolina estimate organic matter visually by color. The other laboratories determine the organic matter content by modifications of the Walkley-Black wet oxidation procedure; however, Georgia, Kentucky, and Louisiana determine it only upon request.

Reagents

1. **Dichromate:**
   (a) **4N:** Dissolve 198.72 g of $Na_2Cr_2O_7 \cdot 2H_2O$ or 196.29 g of $K_2Cr_2O_7$ in 1 liter of distilled water.
   (b) **1N:** Dissolve 49.68 g of $Na_2Cr_2O_7 \cdot 2H_2O$ or 49.05 g of $K_2Cr_2O_7$ in 1 liter of distilled water.
   (c) **0.4N:** Dissolve 19.87 g of $Na_2Cr_2O_7 \cdot 2H_2O$ or 19.62 g of $K_2Cr_2O_7$ in 1 liter of distilled water.
2. **Reducing Agent:**
   (a) **0.2N Ferrous Ammonium Sulfate:** Dissolve 78.43 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in approximately 500 ml of distilled water containing 15 ml of concentrated H_2SO_4. Dilute to 1 liter with distilled water.
   (b) **0.4N Ferrous Ammonium Sulfate:** Dissolve 156.86 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in approximately 500 ml of distilled water containing 15 ml of concentrated H_2SO_4. Dilute to 1 liter with distilled water.
   (c) **0.35N Ferrous Sulfate:** Dissolve 97.3 g of $FeSO_4 \cdot 7H_2O$ in approximately 500 ml of distilled water containing 15 ml of concentrated H_2SO_4. Dilute to 1 liter with distilled water.
   (d) **0.5N Ferrous Sulfate:** Dissolve 139 g of $FeSO_4 \cdot 7H_2O$ in approximately 500 ml of distilled water containing 15 ml of concentrated H_2SO_4. Dilute to 1 liter with distilled water.
   (e) **1N Ferrous Sulfate:** Dissolve 278 g of $FeSO_4 \cdot 7H_2O$ in approximately 500 ml of distilled water containing 15 ml of concentrated H_2SO_4. Dilute to 1 liter with distilled water.
3. **Indicator:**
   (a) **0.25M o-Phenanthroline:** Dissolve 0.45 g of o-phenanthroline in 100 ml of distilled water.
   (b) **Barium Diphenylamine Sulfonate:** Dissolve 0.16 g of barium diphenylamine sulfonate in 100 ml of distilled water.
   (c) **Ferroin:** Dissolve 14.05 g of o-phenanthroline monohydrate and 6.95 g of $FeSO_4 \cdot 7H_2O$ in distilled water and dilute to 1 liter.

TABLE 6.—THE QUANTITIES OF SOIL, OXIDIZING, AND REDUCING AGENTS USED TO ESTIMATE ORGANIC MATTER CONTENT.

	Ark.*	Ga.	Ky.	Miss.	N. C.	Okla.	P. R.	Texas †	Va.
Soil	1.0 g	1.0 g	1.0 g	1.0 g	1.5 g	0.5 g	1.0 g	1.0 g	1.5 g
4N Dichromate									20 ml
1N Dichromate		10 ml	15 ml	10 ml			10 ml	10 ml	
0.4N Dichromate	20 ml				25 ml	10 ml			
H_2SO_4	20 ml	20 ml	30 ml	20 ml	25 ml	15 ml	20 ml	10 ml	20 ml
Distilled H_2O	60 ml	60–100 ml	100 ml	100 ml	100 ml	100 ml	200 ml	60–200 ml	100 ml
0.35N $FeSO_4 \cdot 7H_2O$			yes						
0.5N $FeSO_4 \cdot 7H_2O$							yes		
1N $FeSO_4 \cdot 7H_2O$								yes	
0.2N $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$						yes			
0.4N $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$	yes	yes			yes				
o-phenanthroline	0.15 ml	0.25 ml	0.25 ml		0.25 ml	0.25 ml			
Ferroin							0.25 ml		
Barium diphenyl sulfonate								1.0 ml	
Spectrophotometer	yes			yes				yes	yes

* Determines O. M. colorimetrically and by titration.

† Texas dilutes with 60 ml of distilled H_2O when determining the O. M. content colorimetrically.

General Procedure

Weigh or measure a quantity of soil (0.5 – 1.5 g) into an Erlenmeyer flask. Add a volume (10 – 25 ml) of a standard dichromate solution (0.4N – 4.0N) to the soil. Add concentrated H_2SO_4 (10 – 30 ml) rapidly to the dichromate, shake, either heat to 150-163° C or use only the heat of dilution of H_2SO_4, and allow the mixture to cool (10 minutes – overnight). Dilute with distilled water (60 – 200 ml).

Determine the organic matter content by either:

(1) Titrating the unreacted dichromate with a ferrous salt using either o-phenanthroline, ferroin, or barium diphenylamine sulfonate as the indicator.

(2) Measuring the percent transmission of a filtered aliquot in a spectrophotometer at 620 mu. Adjust the instrument to 100 percent transmission with a blank that is prepared by mixing together all reagents in the same order and quantity as used to determine the organic matter content of soils.

The specific quantities of soil and reagents used by each state in determining the organic matter content are summarized in table 6.

Calculations

The percentage of organic matter is calculated by:

1. Converting the percent transmission to percent O.M. from a standard curve prepared from samples of known organic matter content.
2. (Me of dichromate) – (Me of ferrous salt) × 0.67 ÷ wt. of sample.

LIME REQUIREMENT

Alabama, Louisiana, Mississippi, and Puerto Rico estimate the lime requirement of soils. Kentucky and Virginia estimate lime requirement only upon request and on ASC samples, respectively. Since each state uses a different procedure, each procedure will be described separately.

ALABAMA

Reagent

1. **Buffer Solution:** Dissolve separately 360 g of p-nitrophenol and 270 g of H_3BO_3 in 3 liters each of hot distilled water. Dissolve 189 g of KOH in approximately 200 ml of distilled water. Add 1332 g of KCl to 6 liters of distilled water in a 20-liter bottle. Most of the KCl should go into solution. Then add the p-nitrophenol, H_3BO_3, and KOH to the HCl solution, shaking thoroughly after each addition. Make to 18 liters with distilled water and adjust to pH 8.00 with either KOH or HCl.

Procedure

To the soil from the pH determination (20 ml soil – 20 ml H_2O), add 20 ml of the buffer solution, stir thoroughly, and let stand for a minimum of 10 minutes. Standardize the pH meter to read 8.00 with a 1:1 buffer-water mixture. Stir thoroughly and measure the pH of the soil-buffer suspension immediately with a glass electrode to the nearest 0.05 unit.

To raise the soil pH to 6.5, add the pounds of limestone per acre listed in table 7 at the intersection of soil pH and buffer pH values.

KENTUCKY

Reagent

1. **Buffer Solution:** Dissolve 32.4 g of p-nitrophenol, 45 ml of triethanolamine, 54 g of K_2CrO_4, 36 g of $Ca(C_2H_3O_2)_2$, and 955.8 g of $CaCl_2 \cdot 2H_2O$ in approximately 9 liters of distilled water. Add the reagents in the order listed. Dilute to 18 liters with distilled water and adjust to pH 7.5 with either HCl or NaOH.

Procedure

To 5 g of soil in a 50 ml beaker add 5 ml of distilled water, 10 ml of the buffer solution, and stir thoroughly. After 1 hour, stir the soil-buffer suspension and measure the pH immediately by means of a glass electrode.

Determine the quantity of limestone required to raise the soil pH to the desired value (pH 6.0 or pH 6.4) from the pH of the soil-buffer suspension (table 8).

LOUISIANA

Reagents

1. **Calcium Hydroxide Solution:** Add an excess of $Ca(OH)_2$ to distilled water. Filter the saturated solution and determine the normality. Calculate the volume equivalent to 1 ton of limestone per acre for 10 g of soil.
2. **0.01N Calcium Chloride:** Dissolve 0.555 g of $CaCl_2$ in 1 liter of distilled water.

Procedure

Weigh five 10 g samples of each soil into 100 ml beakers. Add 25 ml of 0.01N $CaCl_2$ and stir. Then add increments of the saturated $Ca(OH)_2$ solution equivalent to 0, 1, 2, 3, and 4 tons of limestone per acre. Stir the mixture thoroughly and after 25 hours determine the pH with a glass electrode. From the buffer curve, estimate the quantity of lime required to raise the soil pH to the desired range.

TABLE 7.—THE QUANTITY OF LIMESTONE IN HUNDRED POUNDS REQUIRED TO RAISE THE SOIL pH TO 6.5.

pH of Soil-Buffer Suspension	Soil pH Values															
	6.0	5.9	5.8	5.7	5.6	5.5	5.4	5.3	5.2	5.1	5.0	4.9	4.8	4.7	4.6	4.5
7.85	6	7	7	8	9	9	10	10	11	11	12	12	12	13	13	13
7.80	8	9	10	11	11	12	13	13	14	15	15	16	16	17	17	18
7.75	10	11	12	13	14	15	16	17	18	18	19	20	20	21	21	22
7.70	11	13	15	16	17	18	19	20	21	22	23	24	24	25	26	27
7.65	13	15	17	18	20	21	22	23	25	26	27	27	28	29	30	31
7.60	15	17	19	21	23	24	26	27	28	29	30	31	32	33	34	36
7.55	17	20	22	24	26	27	29	30	32	33	34	35	36	38	39	40
7.50	19	22	24	26	28	30	32	34	35	37	38	39	41	42	43	45
7.45	21	24	27	29	31	33	35	37	38	40	41	43	45	46	47	49
7.40	23	26	29	32	34	36	38	40	42	44	46	47	49	50	52	53
7.35	25	28	31	34	37	39	41	44	45	48	51	51	53	54	56	58
7.30	27	31	34	37	40	42	45	47	49	51	53	55	57	59	60	62
7.25	29	33	36	40	43	45	48	50	53	55	57	59	61	63	65	67
7.20	31	35	39	42	45	48	51	54	56	58	61	63	65	67	69	71
7.15	33	37	41	45	48	51	54	57	60	62	64	67	69	71	73	76
7.10	34	39	44	48	51	54	58	60	63	66	68	71	73	75	78	80
7.05	36	41	46	50	54	58	61	64	67	69	72	75	77	79	82	85
7.00	38	44	48	53	57	61	64	67	70	73	76	78	81	84	86	89

TABLE 8.—QUANTITY OF LIMESTONE REQUIRED TO RAISE THE SOIL REACTION TO pH 6.0 AND pH 6.4.

pH of Soil-Buffer Suspension	Limestone Tons per Acre[*]	
	pH 6.0	pH 6.4
6.7	1.5	1.5
6.6	2.0	2.5
6.5	2.5	3.0
6.4	3.0	4.0
6.3	4.0	4.5
6.2	4.5	5.0
6.1	5.0	6.0
6.0	5.5	6.5
5.9	6.0	7.5
5.8	6.5	8.0
5.7	7.5	8.5
5.6	8.0	9.5
5.5	8.5	10.0
5.4	9.0	10.5
5.3	9.5	11.5
5.2	10.0	12.0
5.1	11.0	12.5
5.0	11.5	13.5
4.9	12.0	14.0
4.8	12.5	14.5

[*] 90 percent neutralizing value of $CaCO_3$.

MISSISSIPPI

Reagent

Buffer Solution: Dissolve 178 g of p-nitrophenol in approximately 5 liters of hot distilled water. Dissolve 450 g of $CaCl_2 \cdot 2H_2O$ in approximately 5 liters of distilled water. Mix together the p-nitrophenol and $CaCl_2 \cdot 2H_2O$ solutions and dissolve 34.5 g of NaOH in the mixture. Dilute to 18 liters with distilled water and adjust the pH to exactly 7.2 with either NaOH or HCl.

Procedure

Place 10 g of soil in a 50 ml beaker, add 20 ml of the buffer solution, and stir. Stir again after 30 minutes. Standardize the pH meter with the buffer solution. Stir the buffer-soil suspension after 1 hour and measure the pH immediately. Each decrease of 0.1 of a pH unit below 7.2 indicates the need of 1,000 pounds of limestone per acre.

VIRGINIA

Reagent

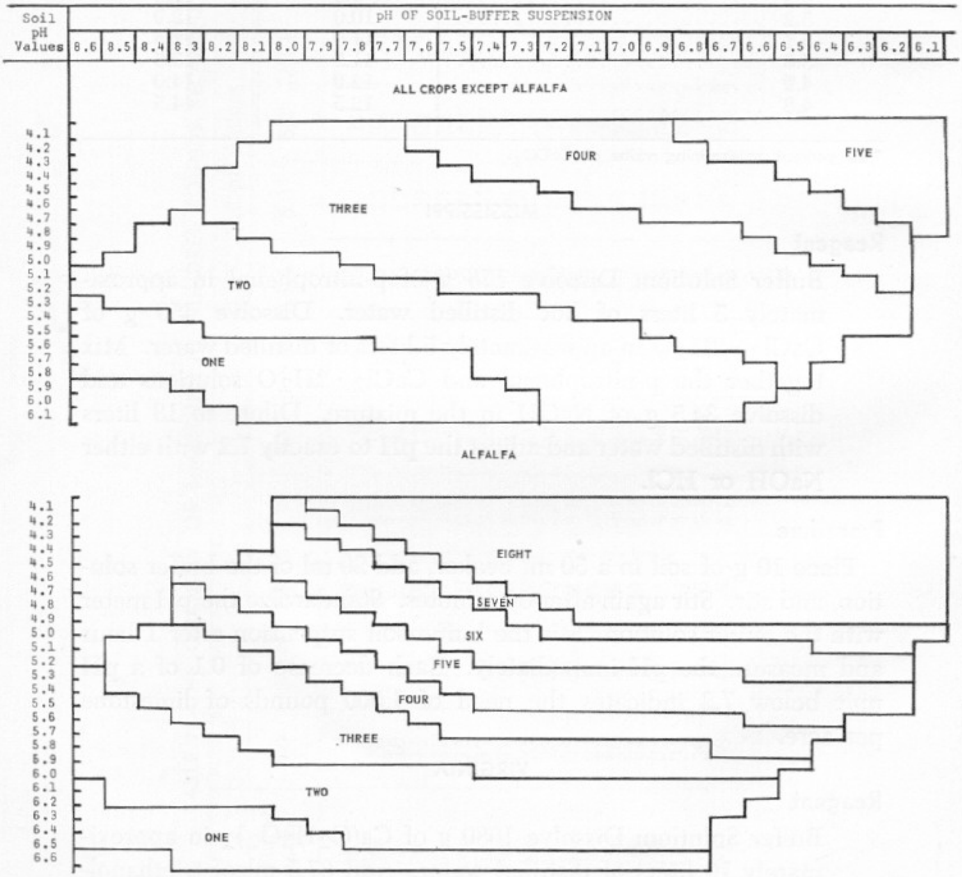
Buffer Solution: Dissolve 1080 g of $Ca(C_2H_3O_2)_2$ in approximately 10 liters of distilled water. Add 67.5 ml of triethanolamine, dilute to 18 liters with distilled water, and mix thoroughly. The pH of the solution should be 9.6. Fit the bottle with a delivery tube and protect the solution from atmospheric CO_2.

Procedure

Place 10 g of soil in a 50 ml beaker and add 20 ml of the buffer solution. Stir intermittently until the pH of the blank is 9.3 (approximately 4 hours). Stir thoroughly and measure the pH of the soil-buffer suspension with a glass electrode.

Determine the lime requirement from the values listed in table 9 at the intersection of soil pH and buffer pH values.

TABLE 9.—LIME RECOMMENDATION IN TONS PER ACRE.



Reagent

C. P. Calcium Carbonate

Procedure

Weigh five 20 g samples of each soil into 250 ml Erlenmeyer flasks. To the series of soil samples add $CaCO_3$ in 20 mg increments (20 – 100 mg), equivalent to 1, 2, 3, 4, and 5 tons of limestone per acre. Add 150 ml of distilled water to each flask, stopper, shake mechanically for 3 hours, and determine the pH with a glass electrode. From the buffer curve (pH vs tons of $CaCO_3$ added), estimate the quantity of lime required to raise the soil pH to the desired range.

SOLUBLE SALTS

Alabama, Arkansas, Florida, Georgia, Kentucky, Louisiana, North Carolina, Oklahoma, South Carolina, and Virginia determine the total soluble salts on special soils upon request. Texas determines total soluble salts on all samples. Louisiana determines gravimetrically the total solids that dissolve in water. The other states determine total soluble salts by either the specific conductance or resistance of the water extract.

Gravimetric

Louisiana

Shake 100 g of soil with 250 ml of distilled water for 15 minutes on a mechanical shaker. Allow to stand overnight before filtering. Evaporate a 50-ml aliquot to dryness in an evaporating dish, ignite at low heat to destroy the organic matter, and weigh. From the weight of salts calculate the percent of soluble salts in the soil.

Conductivity or Resistance

Alabama, Arkansas, Florida, Georgia, Kentucky, North Carolina, Oklahoma, South Carolina, Texas, and Virginia

Add sufficient distilled water to a weighed or measured quantity of soil so as to provide either a 1:1 or a 1:2 soil-water ratio (varies from 10 g soil – 20 ml H_2O to 100 g soil – 100 ml H_2O). Shake the suspension (10 minutes – 1 hour) or stir thoroughly. Allow the suspension to set (10 minutes – overnight) before filtering. Determine the temperature of the filtered extract and measure either the specific conductance with the Solu-Bridge or the resistance with a wheatstone bridge. Convert the readings to ppm of soluble salts from a calibration curve of known solutions of the salts under consideration.

In Texas, the soluble salts are determined directly in the soil-water suspension after pH has been determined.

CHLORIDES (Cl)

North Carolina, Oklahoma, and Texas determine the Cl content of irrigation water. Oklahoma and Texas, on saline soils, and Florida are the only states determining the Cl content of soils. North Carolina precipitates Cl as AgCl and measures photometrically the amount of turbidity produced. Oklahoma, Texas, and Florida titrate with $AgNO_3$ using the chromate ion to indicate the end point.

Turbidimetric Method

North Carolina

Chloride Stock Solution

Dissolve 1.6479 g of dry C.P. NaCl in 1 liter of distilled water. This solution contains 1,000 ppm of Cl.

Chloride Standard Solutions

Prepare a set of standard solutions ranging from 0 to 50 ppm Cl from the stock solution. Make to volume with distilled water.

Reagent

1. **Silver Nitrate:** Dissolve 85.0 g of C.P. $AgNO_3$ in 500 ml of distilled water. Add 35 ml of concentrated HNO_3 and dilute to 1 liter with distilled water.

Procedure

Add 4 ml of filtered water (standard solution) to a glass vial and add 1 ml of the $AgNO_3$ solution. Prepare a blank with each group of samples by adding 1 ml of $AgNO_3$ solution to 4 ml of distilled water. Adjust the spectrophotometer (colorimeter) to 100 percent transmission at 420 mu with the vial containing the blank. After 10 minutes, mix thoroughly and measure the light transmission of the turbid suspension. Convert percent transmission to ppm Cl in the water from the Cl standard curve.

Titrimetric Method

Florida, Oklahoma, and Texas, saline soils and irrigation water

Reagents

1. **0.1N Silver Nitrate:** Dissolve 16.988 g of C.P. $AgNO_3$ in 1 liter of distilled water. Add 3 drops of concentrated HNO_3

to prevent the formation of deposits. Store in a brown bottle. Texas uses a 0.05N $AgNO_3$ solution and Oklahoma uses a 0.01N $AgNO_3$ solution.

2. **1M Potassium Chromate:** Dissolve 1.942 g of K_2CrO_4 in 50 ml of distilled water. Add $AgNO_3$ solution dropwise until a slight red color persists. Filter and dilute to 100 ml. Both Oklahoma and Texas use a 5-percent solution of K_2CrO_4.
3. **0.1N Nitric Acid:** Dilute 6.5 ml of concentrated HNO_3 to 1 liter with distilled water.
4. **0.1N Sodium Hydroxide:** Dissolve 4.09 g of NaOH in 1 liter of distilled water.
5. **C.P. Calcium Carbonate**
6. **Phenolphthalein Indicator:** Dissolve 0.1 g of phenolphthalein in 50 ml of ethyl alcohol and dilute to 100 ml with boiled distilled water.

Procedure

Add 0.1N NaOH to an aliquot of the soil extract until the solution is basic to phenolphthalein. Then titrate with 0.1N HNO_3 until the solution becomes colorless. Add small amounts of solid $CaCO_3$ until a slight excess remains undissolved. Add from 3 to 6 drops of K_2CrO_4 solution and titrate with the $AgNO_3$ solution until the suspension has a brownish-red color.

Since Texas uses the solution that was used to determine the concentration of carbonates and bicarbonates, they omit the pH adjustment with NaOH, HNO_3 and $CaCO_3$.

Calculations

$$\text{ppm Cl} = \frac{\text{ml } AgNO_3 \times \text{N } AgNO_3 \times 0.0355 \times \text{dilution} \times 10{,}000}{\text{wt. of sample in g}}$$

Me of Cl per ml = ml of $AgNO_3$ × N of $AgNO_3$ × dilution factor.

MANGANESE (Mn)

North Carolina, Mississippi, and Puerto Rico determine extractable Mn. Each state oxidizes Mn^{2+} to MnO_4^- and measures the color intensity developed.

Manganese Stock Solution

1. **Mississippi and North Carolina:** Dissolve 0.1535 g of $MnSO_4 \cdot H_2O$ in 1 liter of extracting solution. This solution contains 50 ppm of Mn.
2. **Puerto Rico:** Add approximately 50 ml of distilled water and 0.5 ml of concentrated H_2SO_4 to 22.8 ml of a 0.1N $KMnO_4$

solution. Heat to boiling and reduce the MnO_4^- to Mn^{2+} with Na_2SO_3. Boil off the excess SO_2 and dilute to 1 liter. This solution contains 25 ppm Mn.

Manganese Standard Solutions

1. **Mississippi and North Carolina:** Prepare a set of standard solutions ranging from 0 – 25 ppm Mn from the stock solution. Make to volume with extracting solution.
2. **Puerto Rico:** Add increments of the stock solution ranging from 0.005 to 0.2 mg of Mn to graduated test tubes. Add 1 ml of 85 percent H_3PO_4 and continue as directed under procedure.

MISSISSIPPI

Reagents

1. **Potassium Periodate**
2. **85 percent Orthophosphoric Acid**

Procedure

Add 5 – 10 ml of H_3PO_4 and 0.25 g of KIO_4 to 10 ml of soil extract (standard solution) and digest near the boiling point for 30 minutes. Dilute to 100 ml with distilled water.

Prepare a blank with each set of samples using 10 ml of extracting solution instead of the soil extract. Adjust the spectrophotometer to 100 percent transmission at 525 mu with the blank. Measure the percent transmission of the soil extract and convert to ppm Mn in the extract from the standard curve.

NORTH CAROLINA

Reagents

1. **Trisodium Para-Periodate**
2. **Sodium Hydroxide Solution:** Dissolve 8 g of NaOH in 100 ml of distilled water.
3. **85 percent Orthophosphoric Acid**

Procedure

Measure 4 ml of soil extract (standard solution) into a glass vial. Add 0.5 ml of H_3PO_4, about 0.01 g of sodium para-periodate, and 2.0 ml of NaOH solution, mixing well after each addition.

Prepare a blank with each set of samples using 4 ml of extracting solution in place of the soil extract. Adjust the spectrophotometer to 100 percent transmission at 525 mu with the blank. After 30 minutes, measure the percent transmission of the soil extract and convert percent transmission to ppm Mn in the extract from the standard curve.

Reagents

1. **1:1 Nitric Acid**
2. **60 percent Perchloric Acid**
3. **1N Nitric Acid:** Dilute 65 ml of concentrated HNO_3 to 1 liter with distilled water.
4. **85 percent Orthophosphoric Acid**
5. **Potassium Periodate**

Procedure

Evaporate 25 ml of the soil extract to dryness. Digest with 1 ml of 1:1 HNO_3 and 1 ml of $HClO_4$ and evaporate to dryness. Dissolve the residue in 25 ml of 1N HNO_3. Transfer 10 ml of the 1N HNO_3 solution to a graduated test tube. Add 1 ml of H_3PO_4 and 50 mg of KIO_4 and heat in a bath of boiling water for 1 hour. Dilute to 11 ml with distilled water.

Prepare a blank with each set of samples, using 25 ml of extracting solution in place of the soil extract. Adjust the spectrophotometer to 100 percent transmission (525 mu and slit width of 0.06 mm) with the blank. Measure the percent transmission of the soil extract and convert to ppm Mn in the extract from the standard curve.

Calculation

Lb of Mn per acre = ppm Mn in the soil extract × dilution factor × 2.

NITRATES (NO_3)

Nitrates are determined by Florida, Kentucky (greenhouse samples), Louisiana, North Carolina, and Texas, saline soils only. Florida estimates NO_3 with diphenylamine; Kentucky with brucine sulfate; and Louisiana, North Carolina, and Texas use phenoldisulfonic acid.

Diphenylamine

Florida

NO_3 Stock Solution

Dissolve 1.3709 g dried C.P. $NaNO_3$ in 1 liter of extracting solution. This solution contains 1,000 ppm of NO_3–N.

NO_3 Standard Solution

Transfer 4, 13, and 44 ml of stock solution into 200 ml volumetric flasks and make to volume with extracting solution. The solutions contain 20, 65 and 220 ppm of NO_3–N, respectively.

Reagent

1. **Diphenylamine:** Dissolve 0.20 g of diphenylamine in 100 ml of concentrated H_2SO_4.

Procedure

Place one drop of each standard on a spot plate along with one drop of soil extract from each sample. Add 4 drops of diphenylamine solution, let stand for 2 minutes, and then stir with a glass rod. Compare the intensity of the blue color developed in the soil extract to that of the color developed in the three standards. Report the results as low, medium, or high for the 20, 65, and 220 ppm NO_3 standards, respectively.

Phenoldisulfonic Acid

Louisiana, North Carolina, and Texas, saline soils and irrigation water

Nitrate Stock Solution

Dissolve 3.608 g of dried KNO_3 in 1 liter of distilled water. This solution contains 500 ppm of NO_3–N.

Nitrate Standard Solutions

Prepare a set of standard solutions ranging from 0 – 250 ppm NO_3–N from the stock solution. Dilute the standards with distilled water.

Reagents

1. **Phenoldisulfonic Acid:** Dissolve 25 g of phenol in 150 ml of concentrated H_2SO_4. Add 75 ml of fuming H_2SO_4 and heat in a bath of boiling water for 2 hours.
2. **Silver Sulfate:** Dissolve 3.29 g of Ag_2SO_4 in 1 liter of distilled water.

Extraction

1. **North Carolina:** Add 0.2 g of CaO and 20 ml of distilled water to 10 g of soil. Shake for 10 minutes and filter.
2. **Texas:** To a 25-ml aliquot of saturated paste soil extract or irrigation water, add Ag_2SO_4 equivalent to chlorides present and 0.1 g of CaO. Dilute to 50 ml and filter.

Procedure

Evaporate 2 ml of soil extract (standard solution) to dryness on a steam bath or hot plate (110° C). When dry, remove and cool. Add 1 ml of phenoldisulfonic acid and rotate to dissolve the residue. After 10 minutes, add 10 to 15 ml of distilled water, 7 ml of 1:1 NH_4OH, and dilute to 50 ml with distilled water.

Adjust the spectrophotometer (colorimeter) to 100 percent transmission of the soil extract and convert percent transmission to ppm NO_3–N in the soil extract from the standard curve.

Brucine Sulfate

Kentucky

NO_3 Stock Solution

Dissolve 0.7221 g of oven-dried KNO_3 in 1 liter of distilled water. This solution contains 100 ppm of NO_3–N.

NO_3 Standard Solution

Prepare a set of standard solutions ranging from 0 – 12.5 ppm NO_3–N from the stock solution. Make to volume with distilled water.

Reagents

1. **Brucine Sulfate:** Dissolve 2 g of brucine sulfate in 10 ml of glacial CH_3COOH.
2. **0.018N Acetic Acid:** Dilute 1 ml of glacial CH_3COOH to 1 liter with distilled water.

Procedure

To 0.5 ml of soil extract, add 0.5 ml of 0.018N CH_3COOH, 6 drops of brucine sulfate, and 2 ml of concentrated H_2SO_4 in the order listed. After 5 minutes compare the intensity of the color developed to standard color slides (W. A. Taylor & Co., 7300 York Rd., Baltimore, Md., "Soil Nitrate N" slides) or determine the percent transmission in a spectrophotometer at 475 mu which has been adjusted to 100 percent transmission with the blank.

Calculations

Lb per acre NO_3–N = ppm NO_3–N in soil extract × dilution factor × 2.

AMMONIUM NITROGEN (NH_4)

The concentration of NH_4–N is determined only by North Carolina.

Ammonium-Nitrogen Stock Solution

Dissolve 0.0588 g of $(NH_4)_2SO_4$ in 500 ml of extracting solution. This solution contains 25 ppm of NH_4–N.

Ammonium-Nitrogen Standard Solutions

Prepare a set of standard solutions ranging from 0 – 20 ppm of NH_4–N from the stock solution. Dilute the standards to volume with extracting solution.

Reagents

1. **Sodium Tartrate:** Dissolve 50 g of sodium tartrate and 1 g of sodium polyacrylate in 1 liter of distilled water.
2. **Nessler's Reagent, Concentrated:** Dissolve 45.5 g of HgI_2 and 35 g of KI in 50-75 ml of distilled water. Add 112 g of KOH, mix and cool. Dilute to 1 liter with distilled water. After several days decant the clear, supernatant liquid and store in a brown bottle.
3. **Nessler's Reagent, Dilute:** Dilute concentrated Nessler's reagent 1:4 with distilled water and keep in a brown glass bottle.

Procedure

To 1 ml of the soil extract (standard solution), add 2 ml of sodium tartrate solution and 2 ml of the dilute Nessler's reagent. Mix and let stand for 15 minutes.

Adjust the colorimeter to 100 percent transmission at 420 mu with the blank. Read the percent transmission of the soil extract and convert percent transmission to ppm NH_4–N in the soil extract from the standard curve.

Calculations

Lb of NH_4–N per acre = ppm NH_4–N in the soil extract × dilution factor ×2.

SULFATES (SO_4)

Mississippi determines soluble SO_4 in all soils. Oklahoma and Texas determine soluble SO_4 in alkaline soils and irrigation water. Mississippi and Oklahoma precipitate the SO_4 with Ba and measure photometrically the amount of turbidity produced. Texas titrates the soluble SO_4 with a $BaCl_2$ solution using tetrahydroxyquinone as the indicator.

Sulfur Stock Solution

1. **Mississippi:** Dissolve 0.7703 g of $MgSO_4 \cdot 7H_2O$ in 1 liter of extracting solution. This solution contains 100 ppm S.
2. **Oklahoma:** Dissolve 10.1353 g of $MgSO_4 \cdot 7H_2O$ in 1 liter of extracting solution. This solution contains 1000 ppm SO_4.

MISSISSIPPI

Sulfur Standard Solution

Prepare a set of standards ranging from 0 – 40 ppm S from the stock solution. Dilute to volume with extracting solution.

Reagents

1. **Acid Seed Solution:** 1:1 HCl solution containing 20 ppm of S as the SO_4.
2. **Barium Chloride:** Crystals (20 – 60 mesh).

Procedure

To 10 ml of soil extract (standard solution) add 1 ml of acid seed solution and 0.5 g of $BaCl_2$. After 1 minute, swirl the mixture until the $BaCl_2$ dissolves. Prepare a blank with each set of samples by using 10 ml of the extracting solution.

Adjust the spectrophotometer to 100 percent transmission at 420 mu with the blank. Within 2 to 8 minutes read the percent transmission of the soil extract and convert percent transmission to ppm S in the soil extract from the standard curve.

OKLAHOMA

Reagents

1. **pH 4.8 Buffer:** Dissolve 34.46 g of $CH_3COONa \cdot 3H_2O$ in distilled water. Add 13 ml of glacial acetic acid and dilute to 7 liters.
2. **Gum Arabic:** 1 percent solution (preserved with Thymol)
3. **Barium Chloride:** Crystals (20-60 mesh)

Procedure

Pipette an aliquot of the soil extract into a 50-ml volumetric flask. Add 25 ml of the pH 4.8 buffer and dilute to volume with distilled water. Pour the solution into an Erlenmeyer flask, add 0.5 ml of gum arabic, 0.5 g of $BaCl_2$ crystals (¼ teaspoon), and shake mechanically until the $BaCl_2$ crystals are dissolved. Include a set of standards (0, 2, 5, and 10 ml of a 1,000 ppm SO_4 solution) with each series of samples.

Adjust the colorimeter to 100 percent transmission at 700 mu with the blank. Read the percent transmission of the soil extract and convert percent transmission to ppm SO_4 in the soil extract from the standard curve.

Calculations

Lb of $S(SO_4)$ per acre = ppm $S(SO_4)$ in the soil extract × dilution factor × 2.

TEXAS, SALINE SOILS AND IRRIGATION WATER

Reagents

1. **0.02N Barium Chloride:** Dissolve 2.6 g of $BaCl_2$ in 1 liter of distilled water.
2. **Tetrahydroxyquinone indicator (THQ)**
3. **0.1N Sodium Hydroxide:** Dissolve 4 g of NaOH in 1 liter of distilled water.
4. **0.05N Hydrochloric Acid:** Dissolve 4.3 ml of concentrated HCl in 1 liter of distilled water.
5. **Isopropyl Alcohol**

Procedure

Pipette an aliquot of the soil extract or water into an Erlenmeyer flask. Add 1 drop of phenolphthalein indicator. If acid, add 0.1N NaOH until the color is pink. Then add 0.05N HCl until the solution turns colorless. Add 5 mg of THQ indicator and 25 ml of isopropyl alcohol. Titrate with 0.02N $BaCl_2$ solution until the color changes from organge-red to rose-red.

The results are reported as me per ml of saturated extract or water.

BORON (B)

Texas determines the concentration of B on saline soils and irrigation water.

Boron Stock Solution

Dissolve 0.5716 g of H_3BO_3 in 1 liter of distilled water. This solution contains 100 ppm of B.

Boron Standard Solutions

Prepare a set of standard solutions ranging from 0 – 10 ppm B from the stock solution. Dilute to volume with distilled water.

Reagents

1. **Carmine:** Dissolve 0.92 g of carmine in 1 liter of concentrated sulfuric acid.
2. **Concentrated Hydrochloric Acid** (B free)
3. **Concentrated Sulfuric Acid** (B free)

Procedure

Pipette a 2-ml aliquot of soil extract or water into a B free flask. Add 2 drops of concentrated HCl and 10 ml of concentrated H_2SO_4 and cool. Add 10 ml of the carmine solution and let stand for 45 minutes. Prepare a blank with each set of samples using 2 ml of distilled water instead of the soil extract.

Adjust the colorimeter to 100 percent transmission at 585 mu with the blank. Read the percent transmission of the soil extract and convert percent transmission to ppm B in the soil extract or irrigation water from the standard curve.

ZINC (Zn)

Alabama and Georgia determine Zn on special samples upon request.

Zinc Stock Solution

Dissolve 0.4420 g of $ZnSO_4 \cdot 7H_2O$ in 1 liter of extracting solution. This solution contains 100 ppm Zn.

Zinc Standard Solutions

Prepare a set of standard solutions ranging from 0 – 5 ppm Zn from the stock solution. Make to volume with extracting solution.

Procedure

After inserting the Zn cathode lamp into the Perkin-Elmer atomic absorption spectrophotometer according to the manufacturer's instructions, adjust the wavelength to 0.2138 microns and the slit width to 0.40 mm.

Follow the warmup instruction of the manufacturer. Adjust air pressure to 22 psi. Turn on acetylene, light burner, and adjust gas pressure to 8 psi. Adjust reference and sample beams to approximately 100 microamperes while atomizing distilled water or blank reagents into the burner. Turn response switch of ratiometer on and adjust to 0 with zero adjuster.

When the instrument has been calibrated with known concentrations of Zn, atomize an aliquot of the soil extract and record the reading or soil-test rating. Recalibrate the instrument with one of the Zn standards after every tenth soil sample.