LABORATORY PROCEDURES

Soil Testing Laboratory

Agronomy Department

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INTRODUCTION

The procedures used in the Soil Testing Laboratory were first established in 1955.* Although the basic chemical principles have not changed, procedures have been revised in recent years to utilize advances in instrumentation which allow more accurate and rapid chemical determinations. The revised procedures are reported in this publication.

SAMPLE PREPARATION

Soil samples are received in 1/2-pint paper cartons. Soil sample record sheets are generally packaged with the sample. In the laboratory, the boxes are opened and placed in drying trays. Twenty-nine regular samples plus one control sample are placed in each drying tray. At this time, the sample is assigned a laboratory number which is stamped on the soil record sheet. Samples are numbered consecutively each calendar year, beginning with 1 on January 1.

The trays of samples are placed in a drying cabinet through which filtered air at room temperature is drawn. The air can be heated to 10-15°F. above room temperature for drying extremely wet samples. Samples remain in the drying cabinet overnight.

Dried samples are crushed with a hammermill-type crushing machine** and sieved through a 10-mesh stainless steel sieve. The crushed and sieved samples are placed in the original sample boxes to await measuring of subsamples for the various analyses.

ANALYSES

Routine analyses performed on each sample include pH and available Ca, Mg, P, and K. Special tests performed only by request are percent organic matter, soluble salts, and nitrate nitrogen.

pH

From the prepared sample, one scoop (20-ml capacity) of soil is measured into a 50-ml beaker. Twenty milliliters of distilled water are added; the solution is stirred and allowed to set for 15 minutes, but no longer than 2 hours. Immediately before reading, the solution is stirred again. Readings are taken with either a Beckman Zeromatic or a Fisher Accumet 220 pH meter equipped with glass electrode assemblies. The pH meters are standardized with buffer solutions of pH 4.0 and 7.0 after each 10 determinations.

EXTRACTION OF AVAILABLE Ca, Mg, P, and K

Extraction Solution: 0.05 N HCl and 0.025 N H_2SO_4. Preparation: Measure approximately 15 liters of deionized water into a 20-liter bottle. Add 14.0 ml of concentrated H_2SO_4 and 82.0 ml of concentrated HCl and make to 20-liter volume with deionized water.

Extraction Procedure: One scoop (5-ml capacity) of soil is measured into a 60-ml plastic extraction beaker, and 20 ml of dilute HCl-H_2SO_4 extracting solution

*Rich, C. I. 1955. Rapid soil testing procedures used at Virginia Polytechnic Institute. Virginia Agr. Exp. Sta. Bull. 475, 8 p.

**Custom Laboratory Equipment Co., Raleigh, N. C.

is added with an automatic pipetting machine. The samples are shaken on a mechanical shaker for exactly 5 minutes at 100 reciprocations per minute, with a stroke length of 4.45 cm, and filtered through Whatman No. 1 filter paper. Calcium, Mg, P, and K in solution are determined by the following procedures.

ANALYSIS OF EXTRACT
Calcium and Magnesium

Reagent A: Lanthanum Chloride Diluting Solution: Mix 161.0 g of lanthanum oxide (99.99% La_2O_3) in approximately 125 ml of deionized water in a 1 liter volumetric flask. Mix well. Place the flask in a cold water bath and SLOWLY add 250 ml of concentrated HCl. Swirl constantly as small amounts of acid are added to the flask. Make the solution to volume with deionized water and mix well. Dilute to 20 liters with deionized water for the working solution.

Reagent B: Calcium Standard Stock Solution: Fisher 10,000-ppm calcium solution.

Reagent C: Magnesium Standard Stock Solution: Fisher 10,000-ppm magnesium solution.

Reagent D: Calcium - Magnesium 100% Absorption Standard Solution: Dilute 30 ml of Reagent B and 3 ml of Reagent C to 1 liter with deionized water.

Reagent E: Calcium - Magnesium 50% Absorption Standard Solution: Dilute 15 ml of Reagent B and 1.5 ml of Reagent C to 1 liter with deionized water.

Reagent F: Calcium - Magnesium 0% Absorption Standard Solution: Deionized water.

Procedure: One ml aliquots of Reagents D, E, and F and of the soil extracts are diluted with 9 ml of Reagent A. The diluted standards and extracts are read on a Perkin-Elmer Model 290 Atomic Absorption Spectrophotometer. The instrument is adjusted to zero with the diluted Reagent F and to 100 with the diluted Reagent D. The diluted Reagent E should read approximately 50. The scale is approximately, but not exactly, linear. Standard curves are then prepared for scale readings versus pounds per acre of Ca and Mg. Calcium and Mg in the soil extracts are determined using these standard curves.

Phosphorus

Reagent A: Dissolve completely 100 g of ammonium molybdate $[(NH_4)_2\ MoO]$ in approximately 500 ml of distilled water in a 2-liter volumetric flask. Dissolve 2.425 g of antimony potassium tartrate $[K\ (SbO)\ C_4H_4O_4\ .1/2\ H_2O]$ in the molybdate solution. Place the flask in a cold water bath and slowly add 1400-ml concentrated H_2SO_4. Mix well, cool, and make to volume with deionized water. Store in a polyethylene bottle in a dark, cool compartment.

Reagent B: Dissolve 176.0 g of L-Ascorbic acid powder in approximately 500 ml of distilled water in a 2-liter volumetric flask. Dilute to volume with deionized water. Mix well and store in a dark bottle in a cool compartment.

Reagent C: Working Solution: Dilute 20 ml of Reagent A and 10 ml of Reagent B to 1 liter with the extracting solution. Prepare fresh daily. Allow to stand at least 2 hours before adding to soil extracts. For various quantities, mix the following proportions.

No. of Determinations	Reagent B (ml)	Reagent A (ml)	Final Volume (ml)
50	20	40	2,000
100	40	80	4,000
200	60	120	6,000
300	80	160	8,000
400	100	200	10,000
500	130	260	13,000
600	150	300	15,000
700	180	360	18,000
800	200	400	20,000
1,000	240	480	24,000

Reagent D: Phosphorus Standard Solution: Dissolve 4.394 g of potassium phosphate (KH_2PO_4) in 1 liter of extracting solution. This solution contains 1,000-ppm phosphorus.

Phosphorus Standardization

In 1-liter volumetric flasks, place the following amounts of Reagent D and dilute to volume with extracting solution.

Std. No.	Reagent D (ml)	P in solution (ppm)	P in soil lbs./acre
1	0	0	0
2	2.5	2.5	20
3	5.0	5.0	40
4	7.5	7.5	60
5	10.0	10.0	80
6	12.5	12.5	100
7	15.0	15.0	120

Dilute 1 ml of each standard with 24 ml of Reagent C. Allow 20 minutes for color development. Read % transmittance of solutions with the Spectronic 20 set at 882 mμ. Adjust dark current to zero and set 100% transmittance using standard no. 1. Prepare standard curve of % transmittance versus lbs./acre P. Procedure: Dilute 1-ml aliquots of the soil extracts with 24 ml of Reagent C. Allow color to develop for 20 minutes and read on the Spectronic 20 colorimeter, which has been adjusted as indicated in the standardization.

Potassium

Reagent A: Potassium standard stock solution, 1,000 ppm K: Dissolve 1.912 g of oven-dried KCl in 1 liter of distilled water.

Reagent B: Lithium stock solution, 1500 meq/L: Instrumentation Laboratories Standard.

Reagent C: Lithium working solution, 18.75 meq/L: Dilute 12.5 ml of Reagent B to 1 liter with deionized water. For a larger quantity, dilute 250 ml of Reagent B to 20 liters with deionized water.

Reagent D: Potassium 0 standard, 0 ppm K: Deionized water.

Reagent E: Potassium 50 standard, 200 ppm K: Dilute 20 ml of Reagent A to 1 liter with deionized water.

Reagent F: Potassium 100 standard, 40 ppm K: Dilute 40 ml of Reagent B to 1 liter with deionized water.

Potassium Standardization: Working standards are prepared by diluting 2 ml each of Reagent D, E, and F with 8 ml of Reagent C. Adjust the Instrumentation Laboratories Model 143 flame photometer to 0 and 100 with the appropriate standard. Prepare a standard curve of scale readings versus K lbs./acre. Procedure: Dilute 2 ml of soil extract with 8 ml of Reagent C. Read on the Instrumentation Laboratories Model 143 flame photometer which has been standardized according to the previous procedure. Determine K lbs./acre from standard curve.

SPECIAL ANALYSES

Organic Matter

Reagent A: 0.67 M sodium dichromate solution: Dissolve 4,000 g of technical grade sodium dichromate in distilled water and make to a volume of 20 liters.

Reagent B: Technical grade sulfuric acid.

Procedure: One scoop, 1.5-ml capacity, of soil is measured into a 200-ml test tube. Under a hood, 20 ml of Reagent A is added to the soil followed by 20 ml of Reagent B. The solution is allowed to cool at least 40 minutes. After cooling, 100 ml of water is added and the solution is mixed and allowed to stand at least 8 hours. After incubation, 5 ml of the solution are withdrawn using a syringe-type pipette and placed in a colorimeter vial. Readings are taken using a Cenco colorimeter equipped with a red filter. The percentage of organic matter is determined by reference to Table 1.

*Table 1. Colorimeter Readings and Percent Organic Matter for Virginia Soils.

Colorimeter Reading	Organic Matter %	Colorimeter Reading	Organic Matter %
100	0.0	65	1.8
99-95	0.1	64	1.9
94-90	0.2	63	2.0
89-87	0.3	62	2.1
86	0.4	61	2.2
85-84	0.5	60	2.3
83	0.6	59	2.4
82	0.7	58	2.5
81	0.8	57	2.6
80-79	0.9	56	2.7
78	1.0	55	2.9
77-76	1.1	54	3.0
75	1.2	53	3.1
74-73	1.3	52	3.2
72-71	1.4	51	3.3
70-68	1.5	50	3.4
67	1.6	49	3.5
66	1.7	48	3.6
47	3.8	35	6.5
46	4.0	34	6.8
45	4.2	33	7.1
44	4.4	32	7.4
43	4.6	31	7.8
42	4.8	30	8.5
41	4.9	29	9.3
40	5.0	28	10.1
39	5.3	27	10.9
38	5.6	26	11.7
37	5.9	25	12.5
36	6.2	24-23	13.3
		22	14.1
		21	14.5
		20	15.0

*Prepared from a curve found between colorimeter readings and organic matter determined by titration (USDA Circ. 757) in many Virginia soils.

Soluble Salts

Reagent A: 0.01 N KCl Standard Solution: Dissolve 0.7456 g of KCl in 1 liter of deionized water.

Procedure: Twenty milliliters of distilled water are added to the 1:1 soil to water solution used for pH determination. The solution is stirred and allowed to stand for at least 1 hour. The solu bridge apparatus is standardized with Reagent A. At 25°C., Reagent A has an electrical conductivity of 0.0014118 mho per cm. The electrical conductivity (EC) of the supernatant liquid of the soil-water solutions is determined and the ppm soluble salts in soil are calculated from the following equation.

$$*\text{ppm soluble salts in soil} = (EC \times 10^{-5})\ 10^{6} \times .64 \times 2$$
$$= EC \times 12.8$$

*Bower, C. A. and L. V. Wilcox. Soluble Salts. [In] C. A. Black Ed., Methods of Soil Analysis. American Society of Agronomy, Madison, Wisconsin, p. 939.

Nitrate Nitrogen

Reagent A: NO_3-N Stock Solution, 1,000 ppm N: Dissolve 6.061 g of $NaNO_3$ in 1 liter of deionized water.

Reagent B: 0.02 N Copper Sulfate Solution: Dissolve 1.6 g of $CuSO_4$ anhydrous in deionized water in a one liter flask. Dilute to volume and mix well.

Standardization: Prepare standard solutions according to the following table.

Std. No.	Reagent A diluted to 1 liter ml	NO_3-N in solution ppm
1	5	5
2	10	10
3	25	25
4	50	50
5	75	75
6	100	100

Readings in millivolts are taken with an Orion Model 801 pH meter equipped with an Orion Nitrate Specific Ion electrode assembly. The solutions are stirred at a constant rate while readings are taken. A standard curve is prepared by plotting millivolt readings versus ppm NO_3-N in solution.

Procedure: One scoop (20 ml capacity) of soil is measured into a 125 ml Erlymer flask. Fifty ml of Reagent B are added and the solution is shaken for 10 minutes on a wristaction shaker. The solutions are filtered through Whatman No. 1 filter paper and readings are taken in the same manner as described in the Standardization section. Nitrate nitrogen in solution is read from the standard curve and ppm NO_3-N in soil is calculated with the following equation.

ppm NO_3-N in solution x 2.5 = ppm NO_3-N in soil.