RAPID SOIL TESTING PROCEDURES
AT
VIRGINIA POLYTECHNIC INSTITUTE

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Rapid Soil Testing Procedures Used At Virginia Polytechnic Institute

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Introduction

Procedures in soil testing have changed radically in the past 10 years. These changes generally have made the test more rapid and at the same time more quantitative and accurate. They have been made possible as a result of technological improvements both in an understanding of soil chemistry and in development of new analytical instruments. Alterations in the tests have been made necessary by the interest and requests of those using the soil test and by the rapid increase in the number of samples sent to the laboratories.

The soil testing laboratory at V.P.I. tested 59,681 samples in 1954 as against 4,598 in 1940. At present there are not only more soil samples to be tested but also growing demands on the soil test. In Virginia, not many years ago, nearly all soils were in need of lime, nitrogen, phosphorus, and potassium. These nutrients could be recommended with confidence that crop response would be good regardless of the soil test. Now, an increasing percentage of soils require no lime, and others have accumulated plant nutrients, particularly phosphorus, from fertilizer applications over many years.

Although future changes in the V.P.I. soil tests are expected and desired, the methods and calibrations adopted and adjusted over the past five years are relatively stable. Thus, publication of the methods and calibrations is now justified.

Sources of Methods

Soil testing procedures and designs of special equipment previously employed elsewhere were used freely in establishing the present methods. Some of these procedures have been modified to fit Virginia's types of soil and local laboratory facilities. Ackowledgement is made to the institutions or individuals concerned:

1. Soil Testing Service, North Carolina State Department of Agriculture, Raleigh, North Carolina. (Tests for Ca, Mg, P, K). Procedures, with slight changes were taken from undated Mimeo: *Determination of P, Ca, Mg, K, Na, and* NH_4.
2. *Soil Testing in the United States* prepared by the Soil Test Work Group of the National Soil and Fertilizer Research Committee, December 1951. (Racks, measuring spoons, pH assembly, and dish washing apparatus.)
3. *Testing soils for lime requirement by means of a buffered solution and the glass electrode,* C. M. Woodruff, Soil Science, Vol. 66, 53-63, 1948. (Lime requirement test, modified.)

General Operations

The V.P.I. soil testing service is extended to citizens of Virginia free of charge. This is a function of the Virginia Agricultural Extension Service. The Virginia Agricultural Experiment Station cooperates in developing and in calibrating the soil tests. Approximately 98 percent of the samples are sent to the central laboratory at V.P.I. by county agents and other professional workers. Collapsible half-pint boxes are used to ship the samples. A special form accompanies each sample and contains a record of past lime and fertilizer treatments, previous and future

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crops, the soil type, and other information useful in interpreting the test results. The results of the test are placed on this form which is then returned to the professional worker who makes the fertilizer and lime recommendations.

Sample Preparation

The incoming sample is assigned a number and each day a new number sequence is begun with number one. The sample is air dried at about 30°C which usually requires about 24 hours. The sample is then passed through a soil crushing machine[2] which screens out rocks, sticks, and other large refuse. The soil is crushed between two steel rollers, one of which moves perpendicular to the axis of rotation to allow passage of stones. The soil then falls through a second screen into a pan. The screened sample is then transferred to a half-pint round ice cream container held in a tray containing 24 sample boxes. Aliquots of this screened soil are then taken by means of calibrated spoons (see "Sources of Methods") for the various tests.

Use of Racks and Automatic Pipettes

To speed the testing operations, tubes, vials or bottles are held in racks in a row of 12. A group of 12 can then be moved nearly as easily as a single tube, bottle, etc. Trays hold some multiple of 12. The racks are numbered at one end with the beginning sample number, i.e. 1, 13, 25, etc. Washing of the glassware also is done in multiple units. The rack and tubes are inverted over a washing apparatus consisting of a series of small water pipes spaced the same distance apart as the tubes or bottles. For further details see "Sources of Methods".

Motor-operated automatic pipettes are used to dispense small volumes of reagents used in the tests. The Brewer pipette is used to dispense the extracting solution and the sodium dichromate and calcium acetate-triethanolamine solutions. The volume added is constant for all three solutions. The other motor operated pipettes are used for smaller volumes of reagents.

Chemical Principles of the Tests

EXTRACTION: In order to remove "available" nutrients for analysis, the soil chemist attempts to extract in a few minutes an amount equal to, or proportional to, the quantity of nutrients a plant would be able to take up over its entire growing season. If the quantity removed approximates the uptake of plants it is obvious that some stronger process of nutrient removal is required of the soil test than that possessed by the plant. Thus, in most soil tests mineral acids replace the weak carbonic acid excreted by plant roots. In some tests, salt solutions furnish ions to exchange with ions held by the soil. In this test activated carbon is added to adsorb dissolved organic matter which would interfere with the results.

CALCIUM: Calcium is precipitated as calcium oxalate and measured photometrically by the degree of turbidity produced by the precipitate.

MAGNESIUM: Magnesium hydroxide forms a lake with thiazol yellow. The intensity of the color produced is measured photometrically.

POTASSIUM: The intensity of light emitted at 7665 angstroms by potassium ions excited in a flame is measured by a flame photometer.

PHOSPHORUS: The yellow complex of vanadium phospho-molybdates is measured photometrically.

ORGANIC MATTER: Sodium dichromate partially oxidizes organic

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[2] May be obtained from Soil Testing Division, Agronomy Dept., Iowa State College, Ames, Iowa.

matter in soils under the conditions of the test. The residual organic matter in solution is measured photometrically. The amount of residual organic matter is related to the amount originally present.

LIME REQUIREMENT: A buffered solution made up of calcium acetate and triethanolamine as indicated in the procedure has nearly a linear relationship between amount of acid added and pH of the system for the pH range 8 to 6, (3.75 me. per 200 ml per pH unit). In order to determine the potential acidity of the solution it is necessary to measure the exchangeable hydrogen and aluminum present. The calcium ions displace the exchangeable hydrogen and aluminum which then lower the pH of the suspension. The depression in pH of the buffer solution when soil is added is a measure of the potential acidity. This change in pH together with the soil pH, crop to be grown, and other factors are used to determine the lime requirements.

THE pH: The pH of a soil-water suspension is measured by means of the glass electrode, the emf of which is altered by the hydrogen ion concentration.

DETERMINATION OF P, CA, MG, AND K

Extraction

EXTRACTING SOLUTION: Appoximately 0.05*N* HCL and 0.025*N* H_2SO_4 mixture. Measure about 15 liters of H_2O into a 20 liter bottle. Add 14.0 ml. concentrated H_2SO_4 and 83 ml. concentrated HCl, make up to 20 liters with H_2O and mix.

PROCEDURE: Measure 1 scoop (4 ml. capacity corresponding to about 5 gm.) of dried and sieved soil into an extraction bottle. Add 1 scoop (about 250 mgm.) of activated carbon[3] and then add 20 ml. of extracting solution by means of the Brewer automatic pipette. Shake in the mechanical shaker (see Table 7) for 5 minutes and filter through 12.5 cm. Watman No. 1 filter paper held in a one-piece funnel-tube. The filtrate is used to determine P, Ca, Mg, and K. Aliquots are obtained with a hand semi-automatic pipettor.

Calcium and Phosphorus

REAGENTS: 1. Oxalic acid — triethanolamine mixture. Dissolve 6 gm. oxalic acid in about 700 ml. of H_2O, add 50 ml. of commercial triethanolamine and make up to 1 liter with H_2O. (During summer months store in refrigerator and use while cold). 2. Ammonium molybdate — ammonium vanadate mixture. Dissolve 25 gm. ammonium molybdate in 500 ml. of H_2O. Dissolve 1.25 gm. ammonium vanadate in 500 ml. of 1:1 HNO_3 and mix equal volumes of these solutions. Prepare mixture fresh each week. 3. Calcium standard — 0.02*N* Ca. Dissolve 1.0009 gm. $CaCO_3$ in 1 liter of extracting solution. 4. Phosphorus standard — 25 ppm.P. Dissolve 0.1098 gm. KH_2PO_4 in 1 liter of extracting solution.

STANDARD CURVE: Prepare solutions in colorimeter vials as indicated in Table 1. Add 1 ml. of Reagent 1 and continue as given under "Photelometer Adjustment" and "Procedure". Prepare curves for colorimeter readings versus concentrations of phosphorus or calcium in soil.

PHOTELOMETER ADJUSTMENT: Adjust to zero by means of the adjuster. Switch to "on" position and allow to "warm up" for 15 minutes. Insert vial containing 4 ml. of extracting solution, 1 ml. of each of reagents 1 and 2, and adjust meter

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[3] Darco G-60 Reagent Grade, Atlas Powder Co. 60 East 42nd Street, New York 17, N. Y.

Table 1.—Solutions for calcium and phosphorus standardization.

Reagent 3, ml. Ca	Reagent 4, ml. P	Ca in soil		P in soil	
		me. /100 gm.	lbs. CaO /Acre	ppm. P	lbs. P_2O_5/Acre
0.0	4	0.0	0	100	458
0.5	3.5	1.0	560	87.5	401
1.0	3	2.0	1120	75	344
2.0	2	4.0	2240	50	229
3.0	1	6.0	3360	25	114
3.5	0.5	7.0	3930	12.5	57
4.0	0	8.0	4490	0	0

to 100. Use a purple (420 mu.) filter.

Procedure: Measure into glass vials 4 ml. of the soil extract, add 1 ml. of reagent 1 (with Sterling automatic pipette) and mix. After 10 minutes and no longer than 2 hours mix (by inverting the vial) and measure the transmission of light of the turbid solution. See Ca standard curve for conversion of readings to concentration of Ca in the soil.

Add 1 ml. (with Volustat automatic pipette) of reagent 2 and read after 20 minutes or longer. See phosphorus standard curve for conversion of readings to concentration of P in the soil.

If in the test for Ca the photelometer reading is less than 10, repeat by using a smaller aliquot diluted with extracting solution to 4 ml. Proceed as above and multiply the results by the appropriate dilution factor.

Magnesium

Reagents: 1. Calcium chloride 2%. Dissolve 20 gm. $CaCl_2 \cdot 2H_2O$ in 1 liter of H_2O.

2. Hydroxylamine hydrochloride, 5%. Dissolve 50 gm. of the salt in 1 liter of H_2O.

3. Aluminum chloride, 1%. Dissolve 10 gm. $AlCl_3 \cdot 6H_2O$ in H_2O and make up to 1 liter and mix.

4. Thiazol yellow, 0.3%. Dissolve 3.0 gm. Thiazol yellow in H_2O and make up to 1 liter and mix.

5. Sodium hydroxide, 30%. Dissolve 300 gm. NaOH in H_2O, cool, and make up to 1 liter and mix.

6. Measure about 800 ml. H_2O into a liter flask and add in the following order: 20 ml. each of stock solutions 1, 2, and 3, and 50 ml. of triethanolamine, make up to volume with H_2O and mix.

7. Measure about 900 ml. H_2O into a liter flask, add 20 ml. of stock solution 4 and 50 ml. of stock solution 5, make up to volume and mix.

8. Standard Mg solution (0.01-*N*): Dissolve 1.2325 gm. $MgSO_4 \cdot 7H_2O$ in 1 liter of extracting solution.

Standard curve: Prepare a range of standards as indicated in Table 2 (use 50 ml. volumetric flasks). Transfer 1 ml. of each solution to vials and continue as indicated under "Procedure". Prepare a curve of colorimeter readings versus concentration of Mg in the soil.

Table 2.—Solutions for magnesium standardization.

Mg solution (0.01*N*), ml.	Extracting solution, ml.	Mg in soil	
		me./100 gms	lbs. MgO /acre
0	50.0	0	0
2.5	47.5	0.2	80
5	45.0	0.4	160
10	40.0	0.8	320
15	35.0	1.2	480
20	30.0	1.6	640

Procedure: Measure 1 ml. portions of the Mg standard solutions or the soil extract into vials, add 2 ml. of reagent 6, mix and then add 2 ml. of reagent 7. Mix and take colorimeter readings within 5 minutes. Use a green filter. For the 100 setting use 1 ml. of extracting solution containing 2 ml. each of reagents 6 and 7. Use a smaller aliquot of the soil extract if the readings are less than 40. In this case, add sufficient extracting solution for a final volume of 1 ml. Obtain Mg concentration from the standard curve and multiply the results by the appropriate dilution factor.

Potassium

STANDARD POTASSIUM SOLUTION: KCl—0.015*N*). Dissolve 0.3728 gm. of dried KCl in 1 liter of extracting solution.

Table 3.—Standard Potassium solutions.

Use the following amounts of KCl solution and make up to 100 ml. with extracting solution	K in soil me./100 gms	lbs. K_2O /acre
ml.		
0	0.00	0
2.5	0.05	47
5.0	0.10	94
7.5	0.15	141
10.0	0.20	188
12.5	0.25	235
15.0	0.30	282
20.0	0.40	376

PROCEDURE: Put the instrument into operation based on the instructions of the manual supplied by the manufacturer. Prepare standard curve for the above solutions giving flame photometer readings versus K concentration. When measuring the unknown, check performance with standard solution after every 4 to 6 samples. To do this, use the standard nearest the last measured unknown. If the concentration of K is too high, dilute an aliquot with extracting solution. Obtain K concentration from standard curve as with Ca, P, and Mg.

pH

PROCEDURE: Measure approximately 37 gm. (30 ml. scoop) into a 4 ounce jar and add 37 ml. of distilled water. Mix occasionally over a one-hour period. Measure pH with glass electrode assembly. Stir the sample immediately before reading but not during the measurement. Washing of the electrodes between samples is accomplished by means of perforated copper tube shaped in the form of a ring so that distilled water flows out the small holes and washes the electrodes. The flow of water is controlled by a valve operated by foot. The suspension is stirred by motor. For details see reference under "Source of Methods".

Organic Matter

REAGENTS:

(1) Sodium dichromate solution —Dissolve 4000 gm. of technical grade sodium dichromate and make up to 20 liters with water.

(2) Technical sulfuric acid.

(3) Blank solution — To 20 ml. sodium dichromate solution, add 20 ml. H_2SO_4, 100 ml. H_2O, and mix.

PROCEDURE: Measure one scoop (approximately 1.5 gm.) of soil into the organic matter test tube and add by means of the Brewer pipette 20 ml. of the sodium dichromate solutions. Add, under a hood, 20 ml. of H_2SO_4. Allow to cool 40 minutes, add 100 ml. H_2O, and mix. After the suspension has remained undisturbed at least 8 hours transfer approximately 5 ml. with hand-operated pipette to a colorimeter vial and read on the colorimeter, using a red filter. The instrument is set on 100 using the blank solution. The percentage of organic matter is determined by reference to Table 4.

Lime Requirement

REAGENT: Calcium acetate — triethanolamine — dissolve 1600 gm. of calcium acetate in about 10 liters of H_2O contained in a 20 liter bottle. One-hundred ml. of triethanolamine (Practical) is added and the solution is made up to 20 liters and mixed thoroughly. The bottle is fit-

Table 4.—Colorimeter readings versus percentage organic matter[1].

Dial Reading	Percent O. M.	Dial Reading	Percent O. M.	Dial Reading	Percent O. M.	Dial Reading	Percent O. M.
93	0.1	57	1.7	39	3.1	25	5.6
90	0.2	55	1.8	38	3.3	24	5.8
85	0.3	54	1.9	37	3.4	23	6.1
82	0.4	53	2.0	36	3.6	22	6.3
79	0.5	51	2.1	35	3.7	21	6.6
77	0.6	50	2.2	34	3.8	20	6.8
72	0.8	49	2.3	33	4.0	19	7.6
70	0.9	48	2.4	32	4.1	18	8.4
68	1.0	46	2.5	31	4.3	17	9.2
65	1.2	45	2.6	30	4.4	16	10.0
63	1.3	44	2.7	29	4.6	15	10.8
61	1.4	42	2.8	28	4.9	14	11.6
60	1.5	41	2.9	27	5.1	13	12.4
58	1.6	40	3.0	26	5.4	12	13.2
						11	14.

[1] Prepared from a curve found between colorimeter readings and organic matter determined by titration (U.S.D.A. Circ. 757) in many Virginia soils.

ted with a delivery tube and protected from atmospheric CO_2. The pH of this solution should be 9.6.

PROCEDURE: Transfer one 8 ml. scoop (about 10 grams) of soil to a 50 ml. beaker and add 20 ml. of the calcium acetate — triethanolamine solution by means of the Brewer automatic pipette. Stir occasionally and have beakers exposed to the air until the blank reaches a pH of 9.3. This should require about four hours. Measure the pH of this suspension according to the pH procedure. Soils having a pH of 5.0 have varied from 6.6 to 8.3 for the "lime requirement pH" value. At present it is used along with pH and other factors as a guide as to the amount of lime that is recommended. If the procedure is used elsewhere, calibration for the soils in the area would be necessary.

Present Calibration of Soil Tests

The calibration of the soil tests is based on field fertility experiments, plant tissue analyses, and field checking of test results and crop responses on farms. This is a continuing process and the calibrations given below may be changed in the future. They may be made more specific as regards kind of crop because the ability of various plants to use nutrients varies. In Table 5 the symbols have the following significance:

L—Low—Good response to application of element very probable.

M—Medium—Some response to application of element probable.

H—High—Little or no response to application of element probable.

VH—Very High—Response to application of element not probable.

These calibrations apply to field crops such as corn and small grain under average conditions. If crops are grown more intensively with exceptionally good management or if irrigation is practiced, then responses to fertilizer when the soil test is "High" is more probable. Also in the case of crops which require large

Table 5.—Present Calibration of soil tests.

Element (Expressed as oxide)	POUNDS PER ACRE FOR			
	L	M	H	VH
K_2O	0-90	90-212	212-376	376+
MgO	0-40	40-200	200-475	475+
CaO	0-200	200-775	775-3000	3000+
P_2O_5	0-25	25-85	85-250	250+

amounts of fertilizer such as tobacco, alfalfa, or truck crops responses are more likely when the soil test is "High" than with general field crops. Another factor to be considered is maintaining the fertility at a high level. Although response to fertilizer may not be obtained for some crops when the soil test is "High", the fertility as well as yields will decline with time without fertilizer application.

Table 6.—Glassware used for soil tests.

Name	Determination	Outside dimensions, mm. Height	Bottom diam.	Top diam.	Volume ml.
Bottle	Extraction	65	35	33	40
Funnel—tube	Extraction	120	19	50	20
Colorimeter Vial	Ca, Mg, P, O.M.	60	17	17	10
Bottle	pH	60	56	54	110
Beaker (Pyrex)	Lime requirement	50	40	47	50
Tube (Pyrex)	Organic matter	200	38	38	195

Table 7.—Equipment used for soil tests.

Name	Make	Model	Capacity, ml.
Automatic Pipette	Brewer	40	50
Automatic Pipette	Fisher	Volustat	5
Automatic Pipette	Servall	Sterling	3
Hand Pipettor	Bicknell	——	5
Hand Pipettor	Bicknell	——	2
Shaking Apparatus	Eberbach	**	—
Flame Photometer	Perkin Elmer	52-C	—
pH Meter (2)	Beckman	M and N	—
Soil Crushing Machine	Special	——	—
Colorimeter	Cenco Photometer	Ind. B2	—

*Approximately 210 oscillations per minute.