Analytical Methods and Procedures Used in The Soil Testing Laboratory

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Introduction

The soil testing laboratory is operated by the Department of Agronomy as a service to farmers and for the purpose of determining the fertility levels and lime status of soils used for crop production. The fertility level and lime status of soils, in conjunction with a knowledge of certain production practices, serve as a guide in making recommendations for plant nutrient additions for correcting deficiencies or maintaining adequate plant nutrient levels in soils for sustained high-level production.

The laboratory is also concerned with making chemical analyses of soils on which experiments are conducted at the Branch Experiment Stations in Louisiana and on out-field research locations where experiments are conducted in cooperation with crop and livestock producers. Soil samples are also processed and analyzed from uniform regional experiments and cooperative soil investigations in the states in the Southern Region of the United States.[2]

During 1967, approximately 15,000 soil samples were processed and analyzed. Over 90,000 individual chemical analyses were made on these samples. This represented an increase of 30 per cent over the number of samples analyzed during the preceding year. Because of the increased emphasis on high yields and improved crop quality, an increase in the number of soil samples submitted for analysis is expected for each year in the future. To facilitate the chemical analyses of a large number of soil samples and to improve the analytical techniques employed in the laboratory, new instrumentation has been introduced. At the same time, a higher degree of accuracy in the chemical determinations has been attained.

The analytical methods and procedures formerly employed in the soil testing laboratory at Louisiana State University were published in Bulletin Number 102, Southern Cooperative Series, June, 1965 (*11*).[3] Because of improvements in instrumentation and modifications in analytical techniques, the methods and procedures in use as recently as one year ago have been revised. The primary purpose of this publication is to briefly describe the analytical methods, procedures, and techniques currently in use in the soil testing laboratory.

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[1] Assistant Professor, Associate, and Professor, respectively.
[2] In cooperation with S-52 Regional Technical Committee.
[3] Italic numbers in parentheses refer to Literature Cited, page 15.

General Laboratory Determinations

Chemical analyses of soil samples include determinations of soil reaction (pH) and lime requirement, extractable calcium, magnesium, potassium, and phosphorus. The contents of extractable sodium and total soluble salts are determined on some soil samples from research and specific problem areas. Textural class is determined by feel.

Soil reaction (pH) is determined by using a pH-meter and a slurry of soil and distilled water at a soil-to-water ratio of approximately 1:1. An estimation of the amount of lime required to adjust the soil reaction to a desired level is made by increment potentiometric titration of a soil with a 0.04 normal solution of calcium hydroxide and distilled water. The hydrogen ion activity of a slurry composed of soil, distilled water, and calcium hydroxide is measured on a pH-meter.

Two different extracting solutions are employed to determine the content of the soil cations calcium, magnesium, potassium, and sodium and the content of soil phosphorus.

The soil cations calcium, magnesium, potassium, and sodium are determined by a method first proposed by Reed and Sturgis (13) and later modified and adapted by Stelly, Brupbacher, and Ricaud (16) for the analysis of a large number of soil samples. A sample of soil is extracted with a 0.10 normal hydrochloric acid solution using a soil-to-extraction solution ratio of 1:20. The contents of calcium, potassium, and sodium in the soil are determined on the extract, without further dilution, on a flame spectrophotometer. The concentration of magnesium in the extract is determined, without further dilution, on the atomic absorption spectrophotometer.

Phosphorus is determined by a method proposed by Bray and Kurtz (3) and modified, correlated, and calibrated with crop yields by Byrnside and Sturgis (4). Dilute acid-extractable and adsorbed phosphorus are determined simultaneously by extracting a sample of soil with a solution of 0.10 normal hydrochloric acid containing 0.03 normal ammonium fluoride. A soil-to-solution ratio of 1:20 and an extracting period of 15 minutes are used to determine extractable phosphorus. The concentration of phosphorus in the soil is determined on an aliquot of the soil extract after a color is developed upon adding a solution containing ammonium molybdate, sulphuric acid, and boric acid and an acidified solution containing stannous chloride. The intensity of the color developed is measured on a spectrophotometer using standard reference solutions containing known amounts of phosphorus for comparison.

Soil Sample Preparation

Soil samples received in the laboratory are opened daily, assigned a laboratory number, and placed in 12" x 12" stainless steel pans for air drying. Oscillating fans circulating air over the soil samples for a period of 24 hours facilitate drying. The samples are ground mechanically in a Bico Type U A pulverizer so that the particles will pass through a U. S.

Standard Sieve No. 10 with 2-millimeter openings. Samples containing excessive amounts of crop residues and undecomposed organic material are passed through a sieve prior to grinding to remove extraneous material.

The Determination of Soil Reaction (pH)

According to Truog (17), soil reaction, or pH, is the most important single chemical characteristic of a soil that determines its suitability for plant growth. The test for soil reaction is by far the simplest and most commonly made test by state soil testing laboratories (11). It is widely used as a means for estimating the limestone needs of soils.

The pH of a solution is defined as the negative logarithm of the hydrogen-ion concentration. Since hydrogen-ion activity, rather than hydrogen-ion concentration, is actually measured by potentiometric methods, it should be more useful and correct to define pH as the negative logarithm of the hydrogen-ion activity (12). This conventional manner of expressing hydrogen-ion concentration has been widely adopted for convenience, but there is another distinct advantage of using the pH scale for estimating lime requirement of soils from soil reaction. In the pH range from 4.5 to 6.5, there is a linear relationship between the pH value of the soil and its degree of base saturation (14). Thus, within this range, the pH value of any soil should increase linearly with increasing amounts of applied limestone.

Chemical Reagents: *1. Buffer Solution, pH 4.0.*—Exactly 20.44 grams of potassium acid phthalate, dried at 105 degrees centigrade, are dissolved in 1,000 milliliters of distilled water.

2. Buffer Solution, pH 7.0.—Dilute 40 milliliters of Beckman (3581) buffer solution, pH 7.0 concentrate, to 1,000 milliliters with distilled water.

Procedure: Approximately 35 grams of air-dried soil are placed into a 4¼-ounce paper cup and 35 millilters of distilled water are added with a filamatic vial filler. The force of delivery is sufficient to thoroughly mix the soil and water.

After 24 hours the suspension is agitated with an electric stirrer and the soil reaction (pH) is determined by inserting the electrodes into the suspension. A Leeds and Northrup No. 7401 pH-meter has proved to be very satisfactory for soil analysis. The pH-meter is standardized by using buffer solutions adjusted to pH 4.0 and pH 7.0.

Comments: There has been little agreement among different investigators as to the use of the proper soil-water ratio in preparation of the soil suspension for pH measurement. Since the reaction of soils generally increases with increasing dilution of the soil with water, the pH values obtained at such high dilutions as 5 or 10 parts of water to 1 part of soil may be questionable. After a thorough review of the literature, Reed and Cummings (14) concluded that "the pH value is erratic and

unreliable when determinations are made on soils with moisture contents at or below the moisture equivalent." The use of the 1:1 soil-to-water ratio has been adopted by many state soil testing laboratories in the Southern Region of the United States (*11*) and appears to offer a good compromise for routine determination of soil reaction.

The Determination of the Lime Requirement of Soils

Many procedures have been developed for measuring the lime requirement of soils, which is defined as the amount of lime needed to bring the pH value from its present value to any desired pH value.

According to Peech (*12*), the better lime requirement procedures involving the use of hydroxides are based on serial potentiometric titration of the soil with calcium hydroxide, using the glass electrode for measurement of the reaction of the soil. The method used by the Louisiana State University Soil Testing Laboratory to measure the lime requirement of a soil is essentially the method described by Dunn (*6*), and is based on the establishment of a titration curve for a particular soil.

Chemical Reagents: *1. Calcium Hydroxide Solution.*—A solution is prepared containing approximately 1.5 grams of reagent-grade calcium hydroxide per liter of distilled water. The solution is thoroughly mixed and allowed to stand until the excess calcium hydroxide has precipitated. The normality and pH of the saturated solution of calcium hydroxide is determined and recorded. Generally, the normality of the solution of $Ca(OH)_2$ is approximately 0.04 normal. The pH of this solution is approximately 12.0.

2. Buffer Solution, pH 4.0.

3. Buffer Solution, pH 7.0.

Procedure: The method used for estimating the lime requirement of a soil involves increment potentiometric titration of a soil sample with a solution of calcium hydroxide of known normality. Ten-gram samples of soil are placed into 4¼-ounce paper cups. Twenty-five milliliters of distilled water and 2.5-milliliter increments of a 0.04 normal calcium hydroxide solution are added to each sample. The soil sample is thoroughly stirred, allowed to stand approximately 24 hours, and then stirred again. After 48 hours the samples are stirred, and the pH is determined with a glass electrode and a calomel reference electrode.

The calcium hydroxide solution is added with an automatic vial filler which is calibrated to deliver, with each stroke of the filler, a volume of solution that is equivalent to 1,000 pounds of pure calcium carbonate per acre. The number of increments of calcium hydroxide used in the titration will depend upon the initial soil reaction, the soil reaction desired, the crop that is to be produced, and the soil texture.

Increments of calcium hydroxide added to samples of soil, calcium carbonate equivalents, and agricultural limestone conversions used to interpret the results obtained in estimating the lime requirement of a soil are presented in Table 1.

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TABLE 1.—Increments of calcium hydroxide added to samples of soil, calcium carbo-
nate equivalents, and agricultural limestone conversions used to interpret the results
obtained in estimating the lime requirements of a soil

Increments of 0.04 N Ca(OH)$_2$	CaCO$_3$ Equivalent[1]	Agricultural Limestone Conversions[2]
Ml. per 10 g. soil	Pounds per acre	Pounds per acre
2.5	1,000	2,000
5.0	2,000	4,000
7.5	3,000	6,000
10.0	4,000	8,000

[1]Values shown are based on reagent-grade calcium hydroxide and calcium carbo-
nate.

[2]Values are calculated from calcium carbonate equivalents, assuming that agricul-
tural liming materials are 50 per cent as effective as pure calcium carbonate.

Comments: The calcium hydroxide titration method is not considered
to be a rapid method for assessing lime needs of soils, since approxi-
mately three days are required for the reaction of calcium hydroxide with
acid soil to come to equilibrium. Actually, about 97 per cent of the
reaction is complete in this time and the true equilibrium is attained
only after many days (5).

Calcium chloride may be used to hasten the establishment of equilib-
rium of the calcium hydroxide and the soil; however, the pH value at
any point on the titration curve is much lower in the presence of the
calcium chloride, although the equivalence point may not be changed
appreciably by its addition (12).

The Determination of Calcium, Magnesium, Potassium, and Sodium in Soils

Calcium, magnesium, potassium, and sodium exist in soils as cations
that are adsorbed on the collodial complex and are present in the soil
solution. The determination of the cations has been studied rather ex-
tensively during the past 50 years. The classical gravimetric, colorimetric,
and volumetric methods used in former years have largely been replaced
by equally accurate, but more rapid, methods. With the introduction of
new instrumentation, spectrophotometric and flame photometric methods
have been widely used for the determination of the concentration of ca-
tions in soils (11).

Extracting soils with a dilute acid, such as approximately 0.10 normal
hydrochloric acid, for the determination of the soil cation affords a
means of obtaining a reliable measurement of these elements which may
serve as a guide in evaluating the fertility level of soils.

Extracting Solution for Calcium, Magnesium, Potassium, and Sodium

The extracting solution used for calcium, magnesium, potassium, and
sodium is 0.10 normal hydrochloric acid. The extracting solution is
prepared in the following manner.

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A 2.5 normal stock solution of hydrochloric acid is prepared by diluting 207.2 milliliters of concentrated hydrochloric acid, assaying 37 per cent pure acid with a specific gravity of 1.189, to 1,000 milliliters with distilled water. The solution is standardized by titrating the acid solution into a known amount of sodium carbonate, using phenolphthalein and bromcresol green as indicators. The extracting solution is prepared by diluting 1,600 milliliters of the 2.5 normal hydrochloric acid solution to exactly 40 liters. This is a 0.10 normal hydrochloric acid solution.

Procedure for Extracting Calcium, Magnesium, Potassium, and Sodium

A 2.5-gram sample of air-dry soil is placed into a 125-milliliter Erlenmeyer flask. Fifty milliliters of the 0.10 normal hydrochloric acid extracting solution are added to the soil sample and this mixture is agitated for 15 minutes on an automatic shaking machine. The soil suspension is filtered through Whatman No. 12 folded filter paper into a 50-milliliter Erlenmeyer flask. Calcium, magnesium, potassium, and sodium are determined on the extract without further dilution.

The Determination of Calcium in Soils

Chemical Reagents: A standard calcium solution is prepared by dissolving 2.4972 grams of oven-dried reagent-grade calcium carbonate in a solution containing approximately 500 milliliters of distilled water and 8 milliliters of concentrated hydrochloric acid. This solution is diluted to 1,000 milliliters with 0.10 normal hydrochloric acid and contains 1,000 parts per million of calcium. The 1,000-parts-per-million calcium solution serves as a stock solution from which other standard calcium reference solutions are prepared.

Five standard reference solutions containing 0, 50, 100, 150, and 200 parts per million of calcium are prepared by measuring 0, 25, 50, 75, and 100 milliliters of the solution containing 1,000 parts per million of calcium and transferring these to 500-milliliter volumetric flasks. The calcium standards are diluted to a volume of 500 milliliters with 0.10 normal hydrochloric acid.

Procedure: The concentration of calcium in the soil solution extract is determined by means of a Beckman Model DU Flame Spectrophotometer. The instrument settings are listed in Table 2.

The per cent transmittance of each of the calcium standard reference solutions and the soil solution extract is measured according to the operating procedure described in Section II of the Instruction Manual for the Beckman Model DU Flame Spectrophotometer (7). The amount of calcium in the soil solution extract is determined by referring to a calibration curve obtained by plotting the per cent transmittance readings against the calcium concentration of the five reference solutions.

TABLE 2.—Operating conditions for the Beckman Model DU Flame Spectrophotometer and the Beckman Model B Flame Spectrophotometer employed to determine the concentrations of calcium, sodium, and potassium in soil extracts

Operating Conditions	Beckman Model DU		Beckman Model B
	Calcium	Sodium	Potassium
Oxygen, psi.	20.0	20.0	20.0
Acetylene, psi.	4.0	4.0	3.5
Wave length, millimicrons	422.7	590.0	765.0
Sensitivity	2.0	2.0	4.0
Zero suppression	1.0	1.0	——
Photo tube	Blue	Blue	Red
Slit width, mm.	0.03	0.01	0.35
Load resistor	2.0	2.0	——

The Determination of Magnesium in Soils

Chemical Reagents: A standard magnesium solution is prepared by dissolving one gram of reagent-grade magnesium metal in dilute hydrochloric acid solution containing 400 milliliters of distilled water and 20 milliliters of concentrated hydrochloric acid. This solution, diluted to 1,000 milliliters with distilled water, contains 1,000 parts per million of magnesium. A solution containing 100 parts per million of magnesium is prepared by diluting exactly 100 milliliters of the 1,000-parts-per-million magnesium solution to 1,000 milliliters with distilled water. The solution containing 100 parts per million of magnesium serves as a stock solution from which other standard magnesium reference solutions are prepared.

Five standard reference solutions containing 0, 5, 10, 15, and 20 parts per million are prepared by measuring 0, 25, 50, 75, and 100 milliliters of a solution containing 100 parts per million of magnesium and transferring these to 500-milliliter volumetric flasks. The magnesium standards are diluted to a volume of 500 milliliters with a 0.10 normal hydrochloric acid solution.

Procedure: The concentration of magnesium in the soil solution extract is determined with the use of a Perkin-Elmer Model 303 Atomic Absorption Spectrophotometer. The instrument settings are shown in Table 3.

TABLE 3.—Operating conditions and instrument settings for the Perkin-Elmer Model 303 Atomic Absorption Spectrophotometer used to determine the concentration of magnesium in soil extracts

Operating Conditions	Instrument Settings
Wave length, 2852 Angstroms	Range, ultraviolet
Slit	Slit, 5
Source, Hollow cathode	Source, 6 milliamps.
Fuel, Acetylene	Flow, 5 psi.
(Reducing flame)	
Oxidizer, Air	Flow, 4 psi.

The per cent absorption of each of the magnesium standard reference solutions and the soil solution extract is measured according to the operating procedures described in Section II, B, page 12 of the Instruction Manual for the operation of the Perkin-Elmer Model 303 Atomic Absorption Spectrophotometer (8). The per cent absorption of each of the magnesium standard solutions and the soil solution extract is converted to absorbance by using Table III on page 7 of the instruction manual. The amount of magnesium present in the soil solution extract is determined by referring to a calibration curve obtained by plotting the absorbance readings against the magnesium concentration of the five reference solutions.

The Determination of Potassium in Soils

Chemical Reagents: A standard potassium solution is prepared by dissolving exactly 0.9530 gram of oven-dried reagent-grade potassium chloride in 100 milliliters of distilled water. Dilute to 1,000 milliliters with a 0.10 normal hydrochloric acid solution. This solution contains 500 parts per million of potassium and serves as a stock solution from which other standard potassium reference solutions are prepared.

Five standard reference solutions containing 0, 5, 10, 20, and 25 parts per million are prepared by measuring 0, 5, 10, 20, and 25 milliliters of the solution containing 500 parts per million of potassium and transferring them to 500-milliliter volumetric flasks. The potassium standards are diluted to a volume of 500 milliliters with a 0.10 normal hydrochloric acid solution.

Procedure: The concentration of potassium in the soil solution extract is determined by means of a Beckman Model B Flame Spectrophotometer. The instrument settings are listed in Table 2. The per cent transmittance of each of the potassium standard reference solutions and the soil solution extract is measured according to the operating procedure described in Section D of the Instruction Manual for the Beckman Model B Flame Spectrophotometer (7). The amount of potassium in the soil solution extract is determined by referring to a calibration curve obtained by plotting the per cent transmittance readings against the potassium concentration of the five reference solutions.

The Determination of Sodium in Soils

Chemical Reagents: A standard sodium solution is prepared by dissolving 1.2710 grams of oven-dried reagent-grade sodium chloride in 100 milliliters of distilled water. Dilute to 1,000 milliliters with a 0.10 normal hydrochloric acid solution. This solution contains 500 parts per million of sodium and serves as a stock solution from which other standard sodium reference solutions are prepared.

Five standard reference solutions containing 0, 5, 10, 20, and 25 parts per million are prepared by measuring 0, 5, 10, 20, and 25 milliliters of the solution containing 500 parts per million of sodium and transferring

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them to 500-milliliter volumetric flasks. The sodium standards are diluted to a volume of 500 milliliters with a 0.10 normal hydrochloric acid solution.

Procedure: The concentration of sodium in the soil solution extract is determined by means of a Beckman Model DU Flame Spectrophotometer. The instrument settings are listed in Table 2. The per cent transmittance of each of the sodium standard solutions and the soil solution extract is measured according to the operating procedure described in Section II of the Instruction Manual for the Beckman DU Flame Spectrophotometer (7). The amount of sodium present in the soil solution extract is determined by referring to a calibration curve obtained by plotting the per cent transmittance readings against the sodium concentration of the five reference solutions.

Comments

The use of 0.10 normal hydrochloric acid for determining extractable calcium, magnesium, potassium, and sodium affords a rapid and reliable index of the status of these elements in the soils of Louisiana. Unpublished data on file in the Department of Agronomy indicate that the dilute acid extractant currently in use in the Louisiana State University Soil Testing Laboratory compares favorably with ammonium acetate used to determine exchangeable cations.

The Determination of Phosphorus in Soils

The dilute acid-soluble and ammonium flouride-extractable phosphorus method has been used extensively as an index of the availability of phosphorus in soils. The combination of hydrochloric acid and ammonium fluoride is designed to extract the easily acid-soluble fraction of phosphorus, principally calcium phosphates, and a part of the aluminum and iron phosphates. The ammonium fluoride dissolves only aluminum and iron phosphates by its complex ion formation with these metal ions in acid solution (10).

Aluminum and iron phosphates are relatively more abundant in soils having pH values below 6.0 than they are in soils with higher pH values (3). Iron and aluminum forms of phosphorus are particularly important in evaluating extractable phosphorus in soils in Louisiana that are acid in reaction. These forms of native soil phosphorus are of much less importance in estimating the soil phosphorus that is available to crops grown on recent alluvial soils having a soil reaction approaching neutrality (4).

Chemical Reagents: *1. Ammonium Molybdate-Sulphuric Acid-Boric Acid Solution*—Dissolve 5 grams of ammonium molybdate, $(NH_4)_6 Mo_7O_{24} \cdot 4H_2O$, in approximately 100 milliliters of distilled water in a 250-milliliter Erlenmeyer flask. The distilled water may be heated to approximately 60 degrees centigrade to facilitate dissolution of the ammonium molybdate. Add 55 milliliters of reagent-grade concentrated sulphuric acid to 800 milliliters of distilled water in a 1,000-milliliter volu-

metric flask. Cool both solutions. Slowly add the ammonium molybdate solution to the sulphuric acid solution and gently shake the solutions. Dissolve 40 grams of reagent-grade boric acid, H_3BO_3, in the solution containing ammonium molybdate and sulphuric acid. This solution, diluted to a final volume of 1,000 milliliters, contains 0.005 gram of ammonium molybdate and 0.04 gram of boric acid per milliliter of solution and is approximately 2 normal with respect to the sulphuric acid.

2. *Standard Phosphorus Solution*—Dissolve 0.2195 gram of recrystallized potassium dihydrogen phosphate, KH_2PO_4, (dried for one hour at 105 degrees centigrade) in distilled water and dilute to 1,000 milliliters (5). This solution of potassium dihydrogen phosphate contains 50 parts per million of phosphorus. A solution containing 5 parts per million of phosphorus is prepared by diluting exactly 100 milliliters of the 50-parts-per-million phosphorus solution to 1,000 milliliters with distilled water. The solution containing 5 parts per million of phosphorus serves as a stock solution from which other standard phosphorus solutions are prepared.

3. *Stannous Chloride Solution*—Dissolve 25 grams of stannous chloride, $SnCl_2 \cdot 2H_2O$, in 1,000 milliliters of dilute hydrochloric acid, 10 per cent by volume. The acidified solution of stannous chloride should be filtered if a precipitate develops. A thin layer of mineral oil should be added to the stannous chloride to prevent deterioration.

4. *Extracting Solution for Phosphorus*—The extracting solution used for the phosphorus is composed of a mixture of 0.10 normal hydrochloric acid and 0.03 normal ammonium fluoride. The extracting solution is prepared as shown in steps *A, B,* and *C* below.

(*A*) A 2.5 normal solution of hydrochloric acid is prepared by diluting 207.2 milliliters of concentrated hydrochloric acid, assaying 37 per cent pure acid with a specific gravity of 1.189, to 1,000 milliliters with distilled water. The solution is standardized by titrating the acid solution into a known amount of sodium carbonate using phenolphthalein and bromcresol green as indicators.

(*B*) A 1.5 normal solution of ammonium fluoride is prepared by dissolving 55.56 grams of reagent-grade ammonium fluoride in distilled water and diluting the solution to a final volume of 1,000 milliliters. The ammonium fluoride solution is stored in an opaque polyethylene bottle.

(*C*) The extracting solution is prepared by mixing 1,000 milliliters of the 2.5 normal hydrochloric acid solution with 500 milliliters of the 1.5 normal ammonium fluoride solution and diluting these two solutions to exactly 25 liters with distilled water. The extracting solution is 0.10 normal in hydrochloric acid and 0.03 normal in ammonium fluoride.

Procedure: A 2.5-gram sample of air-dried soil is placed in a 125-milliliter Erlenmeyer flask. Fifty milliliters of the phosphorus extracting solution are added to the soil sample. The soil sample and extracting solution are agitated for 15 minutes on an automatic shaking machine. The soil suspension is filtered through Whatman No. 12 folded paper, and

the extract is collected in a 50-milliliter Erlenmeyer flask. A 3-milliliter aliquot of the solution extract is transferred to a 125-milliliter Erlenmeyer flask. Thirty-seven milliliters of distilled water and 10 milliliters of the ammonium molybdate-sulphuric acid-boric acid solution are added to the 3-milliliter aliquot.

Seven phosphorus standard reference solutions containing 0.0, 0.1, 0.2, 0.4, 0.6, 0.8, and 1.0 part per million are prepared by measuring 0, 1, 2, 4, 6, 8, and 10 milliliters of a solution containing 5 parts per million of phosphorus and transferring these to 50-milliliter volumetric flasks. Twenty milliliters of distilled water, 3 milliliters of the phosphorus extracting solution, and 10 milliliters of the ammonium molybdate-sulphuric acid-boric acid solution are added to each of the seven phosphorus standard solutions. The standards are diluted to a volume of 50 milliliters with distilled water.

Three drops of the stannous chloride solution are added to the phosphorus standards and to the aliquot of the soil solution extract. Ten minutes are allowed after the addition of the stannous chloride for complete chemical reaction which results in maximum color development. The optical density of the phosphorus solution is recorded by means of a Bausch and Lomb Spectronic 20 Spectrophotometer. A red filter and a wave length of 650 millimicrons are used to measure the phosphorus concentration in the phosphorus standard reference solutions and in the soil solution extract. The amount of phosphorus present in the soil solution extract is determined by reference to a calibration curve obtained by plotting the optical density readings against the phosphorus concentration in the seven standard reference solutions.

Comments: Boric acid eliminates possible interference of fluoride in the ammonium molybdate color development. However, the necessity for using boric acid for the analysis of most soils has not been established. Interference from fluorides may be encountered on some acid sandy soils *(10)*.

The Determination of Water Soluble Salts in Soils

The term "soluble salts" as applied to soils refers to the inorganic soil constituents that are appreciably soluble in water *(2)*. The determination of soluble salts consists essentially of two steps: (1) the preparation of a soil-water extract, and (2) the measurement of the salt concentration of the extract.

Soluble salts produce harmful effects to plants by increasing the salt content of the soil, thus affecting the osmotic pressure of the soil solution, and by increasing the degree of saturation of the exchange complex with exchangeable sodium *(2, 15)*. With a given quantity of dissolved salt present in the soil, the salt concentration of the soil solution varies inversely with the water content of the soil. The water content of soils in which plants are growing generally fluctuates between a lower limit which may approach the wilting point and an upper limit which is

near the field capacity. The upper and lower limits of field-water contents vary directly with the degree of fineness of the soil. For example, at both the upper and lower limits, a clay soil may contain five times as much water as a sandy soil. If the clay and sandy soils have equal contents of soluble salts expressed on a dry-soil basis, it is evident that, because of the inverse relation between salt concentration and water content, the salt concentration of the soil solution at both limits will be approximately five times as great for the sandy soil as for the clay soil.

The amounts of total salts extracted from soils are influenced to some extent by the water content at which the extract is made. Usually, the total quantity of dissolved salts increases with increasing water content. This phenomenon is probably due to exchange reactions in which dissolved divalent cations replace adsorbed univalent cations, decreased negative adsorption of ions, and the increased solution of gypsum and alkaline-earth carbonates (2).

The water retention characteristic of a soil must be considered in appraising the soluble salt content. An ideal method would consist of measuring the salt concentration of an extract obtained at field-water content, but the difficulty of obtaining such an extract prohibits its routine use. An extract made at a higher water content has been proved to be satisfactory in following changes in the salt content with time or treatment (2).

Procedure: The method used for the determination of water-soluble salts in soils is a modification of the procedure described in the Official Methods of Analysis of the Association of Official Agricultural Chemists (1). Soluble salts in a sample of soil are extracted with distilled water and the total dissolved solids are determined gravimetrically.

A 50-gram sample of soil is placed in a 250-milliliter Erlenmeyer flask. One hundred and twenty-five milliliters of distilled water are added, and the soil and water suspension is agitated for 30 minutes on a mechanical shaking machine. The suspension is allowed to stand overnight and filtered through a Pasteur-Chamberlain filter. A 50-milliliter aliquot of the filtrate is placed in a porcelain or platinum evaporating dish that has been previously heated, cooled in a desiccator, and weighed. The evaporating dish containing the filtrate is placed in an oven and allowed to dry. The residue is then ignited with a Fisher Burner at a very low heat to remove the organic matter. The evaporating dish containing the soluble salts is cooled and placed in a desiccator and weighed. The difference between the weight of the dish and the weight of the dish plus the ignited residue is the weight of the salts that have been extracted from 20 grams of soil.

Comments: According to Jackson (9), the gravimetric method for the determination of dissolved solids affords a direct measure of total salinity and serves as a check on the results by the more rapid electrical conductance methods.

Prolonged heating to remove organic matter should be avoided because of losses of salts, particularly magnesium chloride.

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