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SOIL CHEMICAL ANALYSIS

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SOIL CHEMICAL ANALYSIS,

by M.L. Jackson

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Preface

As Sir Francis Bacon wrote in his *Of Studies*, "Books must follow sciences, not science books." The maturity of any science may be measured in the growth of integrative publications—reviews, monographs, and specialized textbooks. Soil Science is an infant compared to the older disciplines of mathematics, astronomy, physics, chemistry, and medicine, but the trend toward integrative writing in the field has been firmly established. The annual review, *Advances in Agronomy*, and the monograph series, *Agronomy*, first appeared in 1949 and textbooks both general and specialized are available. The author hopes that the comprehensive treatment of procedures and fundamental principles presented in *Soil Chemical Analysis* will foster progress in the science of soil chemistry.

Methods applicable to the chemical analysis of soils are as numerous and varied as the field of chemistry itself. The *extraction* of a chemical constituent from soil is purely a procedure of soil chemistry, while the *determination* of the extracted constituent is an analytical process, limited in range of methods only by consideration of soil characteristics. Although the powerful techniques of fritted glass filtration, centrifugation, absorption and emission spectrophotometry are emphasized, some procedures are included because they can be executed with simple equipment.

The original publications of procedures and critical studies are cited throughout *Soil Chemical Analysis* and the many modifications and improvements developed by the author and his students are included. The free availability of information during the 20 years in which the materials for this book were accumulated made many improvements possible. Great pains have been taken to eliminate flaws in procedure and the author will be grateful to readers who may call his attention to those inadvertently missed.

The author wishes to extend grateful acknowledgments to Dr. M. D. Weldon of the University of Nebraska, who first introduced him to soil science; to Professor E. Truog of the University of Wisconsin, who encouraged the writing of soil chemical methods; to Dr. N. J. Volk of Purdue University, who fostered a broadened outlook of the field of soils and soil chemistry; and to Dr. I. L. Baldwin of the University of Wisconsin for his warmth and encouragement. Special thanks are extended to Dr. B. R. Bertramson of Washington State College, to Dr. J. L. White of Purdue University, and to the many other former associates who encouraged com-

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M. L. JACKSON
Madison, Wisconsin

To the Reader

This book gives the most frequently used soil chemical analysis procedures, useful in instruction and research in soil chemistry, soil fertility, and soil genesis. Because plant growth is essentially related to these fields, procedures are given for plant inorganic constituents. More specialized procedures of these fields have had to be excluded in the interest of space economy. The student in a soil chemical analysis course will, later in research, find a continuing need of the information given. The teacher will find time-saving discussions of principles. The following are suggested weekly assignments for an undergraduate-graduate course:

Determinations

Exercise number

1. Check desk, apparatus and reagents; take soils samples in field (§ 2-19) or if weather does not permit, prepare samples issued by instructor; in which case field sampling to be introduced as soon as weather permits; exercise on colorimetric pH indicators (§ 3-45, 3-46).
2. Determine soil pH with glass electrode (§ 3-22); colorimetric measurement of soil pH (§ 13-91); thiocyanate test for acidity (§ 13-93).
3. Exchangeable hydrogen of soils (§ 4-50); lime requirement of soils (§ 4-64).
4. Neutralizing equivalence of limestone (§ 4-66).
5. Exchangeable metallic cations of soils (§ 5-1 or 18-24).
6. Available phosphorus of soils (§ 7-69, 7-86, 7-99).
7. Organic matter of soils (§ 9-57).
8. Total nitrogen of soils (§ 8-13).
9. Ammonia of soils (§ 8-33); nitrate of soils (§ 8-48).
10. Plant tissue analysis (§ 12-19).
11. Soluble salts of soils and waters (§ 10-25).
12. Rapid soil tests (§ 13-75).
13. Plant tissue test (§ 13-3).
14. Total phosphorus of soils (§ 7-128).
15. Total potassium and sodium of soils (§ 11-37, 11-177); silicate analysis of soils (§ 11-100).
16. Check in apparatus and reagents; turn in reports; check out.

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Introduction

The soil is a medium of great complexity, and soil science has not progressed very far.

—RICHARDS¹

1-1. Soil Presents a Complex Analytical System. The analysis of soils and plants presents an interesting challenge to a prospective soil scientist. In his Fisher award address, H. H. Willard² states,

The number of chemical reactions, the types of apparatus used, and the varied techniques utilized by the analytical chemist today makes one who is broadly trained in this field valuable in solving problems quite outside of analytical chemistry.

This challenge is acute because a soil consists of an extraordinarily complex chemical mixture of different mineral and organic substances. The plant presents a special type of soil extraction. Soil chemical analysis deals (as determinations of soil elements or indirectly as reagents and equipment) with over 60 of the naturally occurring chemical elements (Fig. 1-1). Additional soil elements continually become of interest in plant nutrition, in relation to essentiality or toxicity, or for physiological substitution. As many as half of the remaining 32 naturally occurring elements may be involved ultimately one way or another in soil and plant analysis. Appropriate chemical analysis systems have been developed for quantitative analysis of the mineral elements present, but the determination of mineral structure requires an advanced system of mineralochemical analysis. Likewise, the organic matter in soil or plants growing on or applied as residues to soil is an analytical field in itself. But the total organic matter of soil can

¹ *Diagnosis and Improvement of Saline and Alkali Soils* (Riverside, Calif.: U.S. Salinity Lab., 1947), p. iii.

² *Anal. Chem.*, 23:1726 (1951).

field of soil or plant analysis. However, some procedures can effectively be executed with simple equipment, in the absence of more specialized equipment. For example, potassium may be determined by cobaltinitrite when a flame emission spectrophotometer is not available. Powerful techniques include absorption and emission spectrophotometry, fritted glass filtration, centrifugation, and systematic schemes of analysis of several constituents. These avoid the time-consuming steps of evaporation, paper filtration, and heating to constant weight. The analyst seeking a suitable procedure for a given determination should consult first textbooks on analysis to learn the range of choice and some of the advantages and disadvantages of different procedures. Sometimes he will need to consult abstract journals and journal articles for any recent advances on a particular determination.

ANALYTICAL REAGENTS

1-4. Reagents are supplied commercially in different grades, the purest being "analytical reagent" or "reagent grade," the second, "C.P." and the third, "technical" or "U.S.P." Each of the various grades has a distinct purpose and range of uses for which it is satisfactory. The alert chemist will find that there seems to be a suitable reagent for each specialized analytical function.

1-5. Concentrated Acids and Bases of Commerce. During manufacture, it is customary to express the strength of concentrated acids and bases of liquid form in terms of specific gravity because it is used for controlling their strength and because specified weights can be approximated volumetrically. However, in the laboratory, their strength is best expressed on the basis of chemical equivalence or normality (Table 1-1).

TABLE 1-1
Strength of "concentrated" acids and bases

Reagent	Concentration		Approximate specific gravity
	Normality	Per cent by weight	
HCl	11.6	37 to 38	1.19
H ₂ SO ₄	35 to 36	97 to 100	1.84
HOAc	17.5	99.5	1.13
HNO ₃	16	70 to 71	1.42
HClO ₄	9 to 11.6	60 to 70	1.51 to 1.67
H ₃ PO ₄	45	85	1.71
NH ₄ OH	15	28 to 29	0.90
HOH	55	100	1.00

1-6. Distilled Water. Water freed to varying extent of dissolved substances is essential for all chemical analysis. The quality varies from simple distilled water condensed in a copper or tin still to double or triple

distilled water. A number of treatments are sometimes applied to the water between distillations to effect oxidation of organic substances and to suppress the passage of ions into the distillate. The substances employed for oxidation include KMnO_4 or Br_2 . Sometimes even for minor element work, the redistillation of ordinary distilled water in a Pyrex glass still makes it sufficiently free from metallic ions for analytical purposes.

1-7. Sometimes condensation of high pressure steam coupled with some filtration of the gas produces condensate of a quality suitable to be used as distilled water. Steam condensate usually contains volatile oils and to some extent suspension of solid particles carried from the steam lines. Successful steam condensation as a substitute for distilled water was reported by Margolis.⁵ A commercial high pressure steam condenser designed by Truog is described by Stark.⁶

1-8. Commercially available ionic columns are able to produce water of sufficiently low ionic content for analytical work. Commercial sources include LaMotte Chemical Products Co. (Towson, Baltimore, Md.), Wilken-Anderson Co. (Chicago, Ill.), Sargent Co. (Chicago, Ill.), and Enley Products, Inc. (254 Pearl St., New York 38, N.Y.).

1-9. Filter Paper. The several types of filter paper vary greatly in their content of total ash and in their content of major and minor elements. There is no substitute for experimental check on contamination; the question of paper purity parallels that of reagent purity. Filter paper pulp often is a useful expediter for filtrations through paper. Several types of filter paper in a range of suitable porosities have been prepared by commercial manufacturers. The leading brand names of filter paper, available from the usual chemical supply houses, are Whatman, Munktells, Schleicher and Schuell, and "E and D" (Lapine Co., Chicago, Ill.).

1-10. Chromic Acid Cleaning Solution. Chromic-sulfuric acid cleaning solution is valuable for the final cleaning of glassware. Visible materials and organic solvents should be rinsed out with water before using the cleaning solution.

1-11. The solution is made up by dissolution of 80 gm of $\text{K}_2\text{Cr}_2\text{O}_7$ or $\text{Na}_2\text{Cr}_2\text{O}_7$ in 300 ml of water (with heating). The aqueous solution is placed in a Pyrex container, and one liter of technical grade H_2SO_4 is added cautiously with stirring. Considerable red chromic oxide (Cr_2O_3) precipitates.

1-12. A cleaning solution which does not involve Cr_2O_3 crystallization is made by dissolution of 5 gm of $\text{K}_2\text{Cr}_2\text{O}_7$ in a minimum of water and addition of this solution to one liter of technical grade H_2SO_4 .

1-13. Aqua Regia. The acid oxidant aqua regia is prepared by mixing

⁵ *Sci.*, 115:552 (1952).

⁶ *Modern Hosp.*, 43(3) (Sept. 1934).