

METHODS OF SOIL ANALYSIS

PART 3

Chemical Methods

Methods of Soil Analysis

Part 3 Chemical Methods

Editorial Committee: D. L. Sparks
A. L. Page
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P. N. Soltanpour
M. A. Tabatabai
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Managing Editor: J. M. Bartels

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FOREWORD

Analytical methods are the foundation of a scientific discipline. This was recognized by the Soil Science Society of America when an effort was initiated in 1957 to give recognition to the body of analytical methods developed specifically to characterize soil composition and properties. Publication of the first edition of the “Methods of Soil Analysis” in 1965, under the editorship of Dr. C.A. Black, marked a milestone in the development of the field of soil science. Although there existed several books on soil analysis prior to 1965, this publication was the first authoritative treatise collectively authored by soil scientists under the joint sponsorship of the American Society of Agronomy and American Society of Testing and Materials, and published as volumes in the Agronomy Monograph series. The publication quickly became the primary reference book on methods for analyzing many soil physical, chemical, and microbiological properties.

After the Soil Science Society of America created the Book Series, the Boards of Directors of the American Society of Agronomy and Soil Science Society of America reached an agreement in 1993 “to publish all future reprints, revised editions, and new versions of Methods of Soil Analysis and all subsequent parts as part of the SSSA Book Series.” The third edition of Methods of Soil Analysis will now have three volumes. The volume covering the microbiological and chemical methods was published in 1995. The current volume will cover the chemical methods, and the volume on physical and mineralogical methods is under preparation. This volume includes coverage of newer methods for characterizing soil chemical properties as well as several methods for characterizing soil chemical processes. It should continue to serve as the primary reference on analytical methods and provide soil and environmental scientists additional tools to advance our knowledge of soil properties and soil processes.

H. H. Cheng, *president*
Soil Science Society of America

PREFACE

The second edition of *Methods of Soil Analysis, Part 2, Chemical and Microbiological Properties* was published in 1982. It was edited by A.L. Page, R.H. Miller, and D.R. Keeney. The 2nd edition is recognized as the benchmark reference on chemical and microbiological analyses of soils. It has been used widely by soil scientists and professionals in other fields. More than 11,000 copies have been sold. Due to major advances in analytical equipment and methodology, the desire to include new chapters on analyses of soil chemical processes, and the need to include additional material on microbiological analyses, Part 2 has been divided into two parts and revised. The first book, *Methods of Soil Analysis, Microbiological and Biochemical Properties (Part 2)*, was published in 1995 as SSSA Book Series No. 5. This book, *Methods of Soil Analysis: Chemical Methods*, is Part 3 of SSSA Book Series No. 5.

This book contains 44 chapters, written by 70 authors from throughout the world. A new chapter on quality assurance and quality control is included. Updated chapters are included on the principles of various instrumental methods and their applications to soil analysis. Additionally, new chapters are included on Fourier transform infrared, Raman, electron spin resonance, x-ray photoelectron, and x-ray absorption fine structure spectroscopies. The application of these methods to analyzing soil chemical reactions is currently one of the major research areas in the soil and environmental sciences.

Chapters are included on analyses of soil chemical properties including soil salinity, carbonate and gypsum, soil pH and acidity, lime requirement, cation and anion exchange capacities, and organic matter. Methods for the analyses of soluble, sorbed, and total concentrations of 34 elements are also included. Additionally, these chapters include useful background information on the chemistry of the elements. A new chapter on methods for organic chemical extraction is included.

A new aspect of this book is the addition of procedures for analyzing important soil chemical processes. These include redox and surface charge (points of zero charge) analyses, and kinetic methods and measurements. Chapter 44, the last chapter, discusses equilibrium modeling in soil chemistry.

The editorial committee, that was instrumental in the planning of the book and in the reviewing process, was composed of:

D.L. Sparks, Chairman, University of Delaware, Newark, DE.
 A.L. Page, University of California, Riverside, CA.
 P.A. Helmke, University of Wisconsin, Madison, WI.
 R.H. Loeppert, Texas A&M University, College Station, TX.
 P.N. Soltanpour, Colorado State University, Fort Collins, CO.
 M.A. Tabatabai, Iowa State University, Ames, IA.
 C.T. Johnston, Purdue University, West Lafayette, IN.
 M.E. Sumner, University of Georgia, Athens, GA.

The editorial committee expresses its sincerest gratitude to the anonymous reviewers for their careful and thoughtful evaluation of the manuscripts. Many thanks are extended to Jon Bartels of the SSSA Headquarters Staff for his outstanding editorial assistance.

Methods of Soil Analysis: Chemical Properties should be useful not only to students and professionals in soil science, but to those in allied fields such as engineering, chemistry, geosciences, and marine studies, who are increasingly interested in soils.

D.L. Sparks, *Editor-in-Chief*
University of Delaware
Newark, Delaware 19717-1303

A.L. Page, *Associate Editor*
University of California, Riverside
Riverside, California 92521

P.A. Helmke, *Associate Editor*
University of Wisconsin
Madison, Wisconsin 53706-1299

R.H. Loeppert, *Associate Editor*
Texas A&M University
College Station, Texas 77843

CONTRIBUTORS

- Domy C. Adriano** Professor and Head, Biogeochemical Ecology Division, Savannah River Ecology Laboratory, Drawer E, Aiken SC 29802
- Michael C. Amacher** Research Soil Chemist, USDA-FS, Forestry Sciences Laboratory, 850 N. 1200 E., Logan UT 84321
- Y. O. Aochi** Staff Research Associate, Department of Soil and Environmental Sciences, University of California, Riverside CA 92521
- Richmond J. Bartlett** Professor of Soil and Environmental Chemistry, Department of Plant and Science, Hills Building, University of Vermont, Burlington VT 05405
- J. K. Bartz** Chemist, Washington State Department of Ecology, 1315 W. 4th Avenue, Kennewick, WA 99336
- Paul M. Bertsch** Professor, Advanced Analytical Center for Environmental Science, University of Georgia, Savannah River Ecology Laboratory, Drawer E, Aiken SC 29802
- Paul R. Bloom** Professor of Soil Science, Department of Soil, Water, and Climate, University of Minnesota, 1991 Upper Buford Circle, St. Paul MN 55108
- John M. Bremner** Distinguished Professor Emeritus, Department of Agronomy, Iowa State University, Ames IA 50011-1010
- L.D. Calvin** Professor Emeritus of Statistics, Department of Statistics, Oregon State University, Corvallis OR 97331
- Theodore H. Carski** Research Associate/Supervisor, Agricultural Products, Stine-Haskell Research Center, E.I. duPont de Nemours and Company, Building 210N, Room 204, Elkton Road, P.O. Box 30, Newark DE 19714
- D.L. Cocke** J.M. Gill Professor of Chemistry, Department of Chemistry, Lamar University, P.O. Box 10022, Beaumont TX 77710
- James G. Crock** Analytical Geochemist, Branch of Geochemistry, Analytical Chemistry Services Group, U.S. Geological Survey, Denver Federal Center, Mail Stop 973, Building 20, Box 25046, Denver CO 80225-6046
- Harvey E. Doner** Professor of Soil Chemistry, Division of Ecosystem Sciences, ESPM, University of California, 101 Giannini Hall, Berkeley CA 94720-3110
- Gary B. Dreher** Head, Analytical Geochemistry Section, Illinois State Geological Survey, 615 E. Peabody Drive, Champaign IL 61820
- S.P. Faulkner** Assistant Professor, Wetland Biogeochemistry Institute, Louisiana State University, Baton Rouge LA 70803-7511
- Scott Fendorf** Assistant Professor of Soil Science, Soil Science Division, University of Idaho, Moscow ID 83844-2339

- W.T. Frankenberger, Jr.** Professor of Soil Microbiology and Biochemistry, Department of Soil and Environmental Sciences, University of California, Riverside CA 92521-0424
- Roger Fujii** Research Chemist, Water Resources Division-California District, U.S. Geological Survey, 2800 Cottage Way, Sacramento CA 95825
- R.P. Gambrell** Professor, Wetland Biogeochemistry Institute, Louisiana State University, Baton Rouge LA 70803-7511
- Ben F. Hajek** Professor, Department of Agronomy and Soils, 201 Funchess Hall, Auburn University, Auburn AL 36849
- Liming He** Research Assistant, Scripps Institute of Oceanography, University of California, La Jolla CA 92093-0202
- Philip A. Helmke** Professor of Soil Science, Department of Soil Science, University of Wisconsin, 1525 Observatory Drive, Madison WI 53706-1299
- T. R. Hess** Gill Chair, Department of Chemistry, Lamar University, P.O. Box 10022, Beaumont TX 77710
- L. R. Hossner** Professor of Soil Chemistry, Department of Soil and Crop Sciences, Texas A&M University, College Station TX 77843-2474
- P. M. Huang** Professor of Soil Science, Department of Soil Science, University of Saskatchewan, 51 Campus Drive, Saskatoon, SK S7N 5A8 Canada
- William P. Inskeep** Professor, Department of Plant, Soil and Environmental Sciences, Montana State University, Bozeman MT 59717
- Bruce R. James** Associate Professor of Soil Chemistry, Department of Agronomy, University of Maryland, College Park MD 20742
- Greg W. Johnson** Spectroscopist, Matheson Gas Products, 1861 Lefthand Circle Drive, Longmont CO 80501
- C.T. Johnston** Associate Professor of Soil Chemistry, Department of Agronomy, Purdue University, 1150 Lilly Hall, West Lafayette IN 47907-1150
- J. Benton Jones, Jr.** Vice President, Micro-Macro International, Inc., 183 Paradise Boulevard, Athens GA 30607
- R. Lewis Jones** Professor of Soil Mineralogy and Ecology, Department of Natural Resources and Environmental Sciences, University of Illinois, 1102 S. Goodwin Avenue, Urbana IL 61801
- A.D. Karathanasis** Professor of Soil Mineralogy and Pedology, Department of Agronomy, University of Kentucky, N-122 Agricultural Sciences Building North, Lexington KY 40546-0091
- Rami Keren** Director, Institute of Soils and Water, The Volcani Center, ARO, P.O. Box 6, Bet Dagan 50250, Israel
- E. J. Klesta, Jr.** Director of Technology, Cozz Iron & Metal, Inc., 605 Alexandria Drive, Naperville IL 60565
- Shiou Kuo** Soil Scientist, Department of Crop and Soil Sciences, Washington State University, 7612 Pioneer Way E., Puyallup WA 98371-4998
- Richard H. Loeppert** Professor, Department of Soil and Crop Sciences, Texas A&M University, College Station TX 77843-2474

CONTRIBUTORS

- D. C. Martens** W.G. Wysor Professor of Agriculture and Life Sciences, Department of Soil and Environmental Sciences, Virginia Polytechnic Institute and State University, Blacksburg VA 24061-0404
- S. V. Mattigod** Battelle Pacific Northwest Laboratory, P.O. Box 999, Richland WA 99352
- Robert O. Miller** Soil Scientist/Extension Soil Specialist, Department of Land, Air and Water Resources, Hoagland Hall, University of California, Davis CA 95616-8627
- William P. Miller** Professor of Soil Science, Department of Crop and Soil Sciences, 3111 Miller Plant Sciences Building, University of Georgia, Athens GA 30602-7503
- Richard L. Mulvaney** Professor of Soil Science, Department of Natural Resources and Environmental Sciences, University of Illinois, 1102 S. Goodwin Avenue, Urbana IL 61801
- Darrell W. Nelson** Dean and Director, Agricultural Research Division, 207 Agricultural Hall, University of Nebraska, Lincoln NE 68583-0704
- W. H. Patrick, Jr.** Boyd Professor and Director, Wetland Biogeochemistry Institute Louisiana State University, Baton Rouge LA 70803-7511
- Roger G. Petersen** Professor Emeritus of Statistics, Department of Statistics, Oregon State University, Corvallis OR 97331
- Stewart T. Reed** Assistant Professor of Soil Science, CESTA, 306E South Perry Paige Building, Florida A&M University, Tallahassee FL 32307
- J. D. Rhoades** Laboratory Director, USDA-ARS, U.S. Salinity Laboratory, 450 W. Big Springs Road, Riverside CA 92507
- Brij L. Sawhney** Soil Chemist, The Connecticut Agricultural Experiment Station, 123 Huntington Street, P.O. Box 1106, New Haven CT 06504
- Nicola Senesi** Professor of Soil Chemistry and Director, Istituto di Chimica Agraria, Università di Bari, Via Amendola 165/A, Bari-70126, Italy
- Larry M. Shuman** Professor of Soil Chemistry, Department of Crop and Soil Sciences, Georgia Agricultural Experiment Station, University of Georgia, Griffin GA 30223-1797
- John L. Sims** Professor Emeritus of Agronomy, Department of Agronomy, N-122 Agricultural Science Building North, University of Kentucky, Lexington KY 40546-0091
- J. Thomas Sims** Professor of Soil Science, Department of Plant and Soil Science, 149 Townsend Hall, University of Delaware, Newark DE 19717-1303
- Parviz N. Soltanpour** Professor of Soil Science, Department of Soil and Crop Sciences, C117 Plant Sciences Building, Colorado State University, Fort Collins CO 80523
- Lee E. Sommers** Professor of Soil Science and Department Head, Department of Soil and Crop Sciences, Colorado State University, Fort Collins CO 80523
- Donald L. Sparks** Distinguished Professor of Soil Science, Department of Plant and Soil Sciences, University of Delaware, Newark DE 19717-1303

- Frank J. Stevenson** Professor Emeritus, Department of Agronomy, University of Illinois, 1102 S. Goodwin Avenue, Urbana IL 61801-4798
- Tomasz I. Stuczynski** Soil Scientist, Institute of Soil Science and Plant Cultivation, Osada Palacowa, 24-100 Pulawy, Poland
- Donald L. Suarez** Research Leader, USDA-ARS, U.S. Salinity Laboratory, 450 W. Big Springs Road, Riverside CA 92507
- Malcolm E. Sumner** Regents' Professor of Soil Science, Department of Crop and Soil Sciences, 3111 Miller Plant Sciences Building, University of Georgia, Athens GA 30602
- Roger S. Swift** Chief, CSIRO, Division of Soils, PMB 2, Glen Osmond 5064, Australia
- M. A. Tabatabai** Professor of Soil Chemistry, Department of Agronomy, Iowa State University, Ames IA 50011-1010
- Grant W. Thomas** Professor of Agronomy, Department of Agronomy, University of Kentucky, Lexington KY 40546-0091
- Charles V. Toner, IV** Graduate Research Fellow, Department of Plant and Soil Sciences, University of Delaware, Newark DE 19717-1303
- An M. Vanwormhoudt** 49 Lillibrooke Crescent, Maidenhead, Berkshire SL6 3XJ U.K.
- R. K. Vempati** Department of Chemistry, Lamar University, P.O. Box 10022, Beaumont TX 77710
- Stephen M. Workman** Technical Manager, Analytical Technologies, Inc., 225 Commerce Drive, Fort Collins CO 80524
- Robert J. Wright** Research Soil Scientist, USDA-ARS, Environmental Chemistry Laboratory, Building 007, Room 224, BARC-West, Beltsville MD 20705
- J. M. Zachara** Chief Scientist, Pacific Northwest Laboratory, P.O. Box 999, Mail Stop K3-61, Richland WA 99352
- Lucian W. Zelazny** Thomas B. Hutchison, Jr., Professor of Soil Chemistry/Mineralogy, Department of Crop and Soil Environmental Science, Virginia Polytechnic Institute and State University, Blacksburg VA 24061-0404

Conversion Factors for SI and non-SI Units

Conversion Factors for SI and non-SI Units

xviii

To convert Column 1 into Column 2, multiply by	Column 1 SI Unit	Column 2 non-SI Units	To convert Column 2 into Column 1, multiply by
Length			
0.621	kilometer, km (10^3 m)	mile, mi	1.609
1.094	meter, m	yard, yd	0.914
3.28	meter, m	foot, ft	0.304
1.0	micrometer, μm (10^{-6} m)	micron, μ	1.0
3.94×10^{-2}	millimeter, mm (10^{-3} m)	inch, in	25.4
10	nanometer, nm (10^{-9} m)	Angstrom, \AA	0.1
Area			
2.47	hectare, ha	acre	0.405
247	square kilometer, km^2 (10^3 m) ²	acre	4.05×10^{-3}
0.386	square kilometer, km^2 (10^3 m) ²	square mile, mi^2	2.590
2.47×10^{-4}	square meter, m^2	acre	4.05×10^3
10.76	square meter, m^2	square foot, ft^2	9.29×10^{-2}
1.55×10^{-3}	square millimeter, mm^2 (10^{-3} m) ²	square inch, in^2	645
Volume			
9.73×10^{-3}	cubic meter, m^3	acre-inch	102.8
35.3	cubic meter, m^3	cubic foot, ft^3	2.83×10^{-2}
6.10×10^4	cubic meter, m^3	cubic inch, in^3	1.64×10^{-5}
2.84×10^{-2}	liter, L (10^{-3} m ³)	bushel, bu	35.24
1.057	liter, L (10^{-3} m ³)	quart (liquid), qt	0.946
3.53×10^{-2}	liter, L (10^{-3} m ³)	cubic foot, ft^3	28.3
0.265	liter, L (10^{-3} m ³)	gallon	3.78
33.78	liter, L (10^{-3} m ³)	ounce (fluid), oz	2.96×10^{-2}
2.11	liter, L (10^{-3} m ³)	pint (fluid), pt	0.473

CONVERSION FACTORS FOR SI AND NON-SI UNITS

Mass

2.20 × 10 ⁻³	gram, g (10 ⁻³ kg)	pound, lb	454
3.52 × 10 ⁻²	gram, g (10 ⁻³ kg)	ounce (avdp), oz	28.4
2.205	kilogram, kg	pound, lb	0.454
0.01	kilogram, kg	quintal (metric), q	100
1.10 × 10 ⁻³	kilogram, kg	ton (2000 lb), ton	907
1.102	megagram, Mg (tonne)	ton (U.S.), ton	0.907
1.102	tonne, t	ton (U.S.), ton	0.907

Yield and Rate

0.893	kilogram per hectare, kg ha ⁻¹	pound per acre, lb acre ⁻¹	1.12
7.77 × 10 ⁻²	kilogram per cubic meter, kg m ⁻³	pound per bushel, lb bu ⁻¹	12.87
1.49 × 10 ⁻²	kilogram per hectare, kg ha ⁻¹	bushel per acre, 60 lb	67.19
1.59 × 10 ⁻²	kilogram per hectare, kg ha ⁻¹	bushel per acre, 56 lb	62.71
1.86 × 10 ⁻²	kilogram per hectare, kg ha ⁻¹	bushel per acre, 48 lb	53.75
0.107	liter per hectare, L ha ⁻¹	gallon per acre	9.35
893	tonnes per hectare, t ha ⁻¹	pound per acre, lb acre ⁻¹	1.12 × 10 ⁻³
893	megagram per hectare, Mg ha ⁻¹	pound per acre, lb acre ⁻¹	1.12 × 10 ⁻³
0.446	megagram per hectare, Mg ha ⁻¹	ton (2000 lb) per acre, ton acre ⁻¹	2.24
2.24	meter per second, m s ⁻¹	mile per hour	0.447

Specific Surface

10	square meter per kilogram, m ² kg ⁻¹	square centimeter per gram, cm ² g ⁻¹	0.1
1000	square meter per kilogram, m ² kg ⁻¹	square millimeter per gram, mm ² g ⁻¹	0.001

Pressure

9.90	megapascal, MPa (10 ⁶ Pa)	atmosphere	0.101
10	megapascal, MPa (10 ⁶ Pa)	bar	0.1
1.00	megagram, per cubic meter, Mg m ⁻³	gram per cubic centimeter, g cm ⁻³	1.00
2.09 × 10 ⁻²	pascal, Pa	pound per square foot, lb ft ⁻²	47.9
1.45 × 10 ⁻⁴	pascal, Pa	pound per square inch, lb in ⁻²	6.90 × 10 ³

(continued on next page)

Conversion Factors for SI and non-SI Units

xx

To convert Column 1
into Column 2,
multiply by

Column 1 SI Unit

Column 2 non-SI Units

To convert Column 2
into Column 1,
multiply by

Temperature

1.00 (K – 273) (9/5 °C) + 32	Kelvin, K Celsius, °C	Celsius, °C Fahrenheit, °F	1.00 (°C + 273) 5/9 (°F – 32)
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Energy, Work, Quantity of Heat

9.52 × 10 ⁻⁴	joule, J	British thermal unit, Btu	1.05 × 10 ³
0.239	joule, J	calorie, cal	4.19
10 ⁷	joule, J	erg	10 ⁻⁷
0.735	joule, J	foot-pound	1.36
2.387 × 10 ⁻⁵	joule per square meter, J m ⁻²	calorie per square centimeter (langley)	4.19 × 10 ⁴
10 ⁵	newton, N	dyne	10 ⁻⁵
1.43 × 10 ⁻³	watt per square meter, W m ⁻²	calorie per square centimeter minute (irradiance), cal cm ⁻² min ⁻¹	698

Transpiration and Photosynthesis

3.60 × 10 ⁻²	milligram per square meter second, mg m ⁻² s ⁻¹	gram per square decimeter hour, g dm ⁻² h ⁻¹	27.8
5.56 × 10 ⁻³	milligram (H ₂ O) per square meter second, mg m ⁻² s ⁻¹	micromole (H ₂ O) per square centi- meter second, μmol cm ⁻² s ⁻¹	180
10 ⁻⁴	milligram per square meter second, mg m ⁻² s ⁻¹	milligram per square centimeter second, mg cm ⁻² s ⁻¹	10 ⁴
35.97	milligram per square meter second, mg m ⁻² s ⁻¹	milligram per square decimeter hour, mg dm ⁻² h ⁻¹	2.78 × 10 ⁻²

Plane Angle

57.3	radian, rad	degrees (angle), °	1.75 × 10 ⁻²
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CONVERSION FACTORS FOR SI AND NON-SI UNITS

Electrical Conductivity, Electricity, and Magnetism

10	siemen per meter, S m ⁻¹	millimho per centimeter, mmho cm ⁻¹	0.1
10 ⁴	tesla, T	gauss, G	10 ⁻⁴

Water Measurement

9.73 × 10 ⁻³	cubic meter, m ³	acre-inches, acre-in	102.8
9.81 × 10 ⁻³	cubic meter per hour, m ³ h ⁻¹	cubic feet per second, ft ³ s ⁻¹	101.9
4.40	cubic meter per hour, m ³ h ⁻¹	U.S. gallons per minute, gal min ⁻¹	0.227
8.11	hectare-meters, ha-m	acre-feet, acre-ft	0.123
97.28	hectare-meters, ha-m	acre-inches, acre-in	1.03 × 10 ⁻²
8.1 × 10 ⁻²	hectare-centimeters, ha-cm	acre-feet, acre-ft	12.33

Concentrations

1	centimole per kilogram, cmol kg ⁻¹	milliequivalents per 100 grams, meq 100 g ⁻¹	1
0.1	gram per kilogram, g kg ⁻¹	percent, %	10
1	milligram per kilogram, mg kg ⁻¹	parts per million, ppm	1

Radioactivity

2.7 × 10 ⁻¹¹	becquerel, Bq	curie, Ci	3.7 × 10 ¹⁰
2.7 × 10 ⁻²	becquerel per kilogram, Bq kg ⁻¹	picocurie per gram, pCi g ⁻¹	37
100	gray, Gy (absorbed dose)	rad, rd	0.01
100	sievert, Sv (equivalent dose)	rem (roentgen equivalent man)	0.01

Plant Nutrient Conversion

	<i>Elemental</i>	<i>Oxide</i>	
2.29	P	P ₂ O ₅	0.437
1.20	K	K ₂ O	0.830
1.39	Ca	CaO	0.715
1.66	Mg	MgO	0.602

SSSA BOOK SERIES: 5



Methods of Soil Analysis
Part 3—Chemical Methods

concentrations of individual inorganic solutes (i.e., Na^+ , Ca^{2+} , Mg^{2+} , K^+ , Cl^- , SO_4^{2-} , HCO_3^- , NO_3^- , CO_3^{2-} , H_3BO_3 , etc.) in waters and soil extracts in common use in laboratories having modern instrumentation are described elsewhere in this publication. Analogous methodology suited to laboratories without such conveniences are given in an earlier publication (see Bower & Wilcox, 1965).

SATURATION AND OTHER AQUEOUS EXTRACTS

Principles

Because present methods of obtaining soil water samples at typical field water contents are not very practical, aqueous extracts of the soil samples are usually made at higher than normal water contents for routine soil salinity diagnosis and characterization purposes. Because the absolute and relative amounts of the various solutes are influenced by the soil/water ratio at which the extract is made (Reitemeier, 1946), the soil/water ratio used to obtain the extract should be standardized to obtain results that can be applied and reasonably interpreted generally. Soil salinity is most generally defined and measured on aqueous extracts of so-called, saturated soil-pastes (U.S. Salinity Laboratory Staff, 1954). This water content (and water/soil ratio) varies with soil texture but is used because it is the lowest one for most soils for which sufficient extract can be practically removed from a soil sample for the compositional analysis of major constituents and because it is better related to soil-water contents under field conditions. For these same reasons, crop-tolerance to salinity also is expressed in terms of the EC of the saturation extract (EC_e , Mass & Hoffman, 1977; Maas, 1986, 1990).

Apparatus

1. Plastic containers with snaptight lids of 250-mL capacity or greater.
2. Vacuum line, suction apparatus (Richards filter funnel², Buchner funnel, or commercial vacuum manifold) filter paper (medium grade, such as Whatman no. 50) and sample bottles [28.4 g (1 oz.) or larger, to collect and store extracts] with sealable caps.
3. Balance or scale accurate to at least 1 g.
4. Extraction bottles for soil suspensions.
5. Mechanical shaker.

Graveel (1986); a simple field-pressure filtration method by Ross and Bartlett (1990); adsorption techniques by Shimshi (1966) and by Tadros and McGarity (1976) and centrifugation techniques by Davies and Davies (1963), Yamasaki and Kishita (1972), Gilman (1976), Dao and Lavy (1978), Kinniburgh and Miles (1983) and Elkhatib et al. (1987). Comparisons of the various methods have been made by Adams et al. (1980); Kittrick (1983); Wolt and Graveel (1986); Menzies and Bell (1988) and Ross and Bartlett (1990). The different suction-type samplers and other methods for sampling soil solution and various errors associated with them have been critically reviewed by Rhoades (1978), Rhoades and Oster (1986), Litaor (1988) and Grossman and Udluft (1991).

² Richards, L.A. (1949).

Reagent

1. Sodium hexametaphosphate $[(\text{NaPO}_3)_6]$ solution, 0.1%. Dissolve 0.1 g of $(\text{NaPO}_3)_6$ in water, and dilute the solution to 100 mL.

Procedure

Saturation Extract

Weigh 200 to 400 g of air-dry soil of known water content into a tared plastic container having a snaptight lid. Weigh the container plus contents. Add distilled water to the soil with stirring until it is nearly saturated. Allow the mixture to stand covered for several hours to permit the soil to imbibe the water and the readily soluble salts to dissolve, and then add more water with stirring to achieve a uniformly saturated soil-water paste. At this point, which is generally reproducible to within $\pm 5\%$, the soil paste glistens as it reflects light, flows slightly when the container is tipped, slides freely and cleanly off a smooth spatula, and consolidates easily by tapping or jarring the container after a trench is formed in the paste with the side of the spatula. After mixing, allow the sample to stand (preferably for another 2 h), and then recheck the criteria for saturation. Free water should not collect on the soil surface, nor should the paste stiffen markedly or lose its glisten. If the paste is too wet, add additional dry soil of known amount (weight) to the paste mixture. Upon attainment of saturation, weigh the container plus contents. Record the increase in weight due to the amount of water added. Transfer the saturated soil paste to a filter funnel fitted with highly retentive filter paper. Apply vacuum, and collect the filtrate in a test tube or bottle. If the initial filtrate is turbid, refilter or discard it. Terminate the filtration when air begins to pass through the filter. Add 1 drop of 0.1% $(\text{NaPO}_3)_6$ solution for each 25 mL of extract.

Extracts of Soil/Water Ratios of One:One and One:Five

Weigh a sample of air-dry soil of appropriate size, and transfer it to a flask or bottle. Add the required amount of distilled water (an equal weight for a 1:1 extract, 5 times the weight for a 1:5 extract), stopper the container, and shake it in a mechanical shaker for 1 h. If a mechanical shaker is not available, shake the container vigorously by hand for 1 min at least four times at 30-min intervals. Filter the suspension using highly retentive filter paper. (Discard or refilter the initial filtrate if it is turbid.) Add 0.1% $(\text{NaPO}_3)_6$ solution at the rate of 1 drop/25 mL of extract.

Calculations

Calculate the saturation water percentage (SP) of the saturated paste from the weight of oven-dry soil (W_s) and the weight of water (W) added (W_w), including that initially present in the soil sample as,

$$SP = 100 \cdot W_w / W_s \quad [1]$$

Comments

The extraction ratios (1:1, 1:5, etc.) are easier to make than that of saturation, but they are less well related to field soil water composition and content. More importantly, salinity and compositional errors from dispersion, hydrolysis, cation exchange, and mineral dissolution increase as the water/soil ratio increases (Reitemeier, 1946). As a compromise, Sonneveld and van den Ende (1971) recommended a 1:2 (1 part soil = 2 parts water) *volume* extract, since it is closer to the saturation extract ratio but quicker to make. When relative changes rather than absolute solute concentrations are needed, these wider extraction ratios have the advantages of speed and greater volume.

The EC of extracts of gypsiferous soils decreases less, as the water/soil ratio increases, due to the dissolution of gypsum. This gypsum dissolution exaggerates the concentration of soluble salts present in the soil at lower water contents, especially those existing under field conditions.

Soil samples should not be oven-dried before extracting for determination of soluble salts, because heating to 105°C converts at least a part of the gypsum ($\text{CaSO}_4 \cdot 2 \text{H}_2\text{O}$) to plaster of paris ($\text{CaSO}_4 \cdot 1/2 \text{H}_2\text{O}$). The latter hydrate has a higher solubility in water than does the former. The solubilities of other salts and minerals also may be affected.

The weight of soil required will depend on the number and kind of determinations to be made on the extract, the analytical methods employed, and the salt content of the soil. In general, from one-fourth to one-third of the water in saturated soil pastes can be practically removed by vacuum filtration.

In determinations of extract water contents, especially when a high ratio of soil to water is used, it is desirable to correct for the water content of the soil sample. For example, an air-dry sample containing 2% water on an oven-dry basis can be adjusted to a soil/water ratio of 1:1 by adding 98 mL of water to 102 g of air-dry soil. At soil/water ratios of 1:5 or greater, no correction is ordinarily made for water in the air-dry sample.

Special precautions should be taken in preparing a saturated soil paste with peat and muck soils or very fine or very coarse-textured soils (Prichard et al., 1983). If possible, peat and muck soils should not be allowed to dry appreciably following collection because their saturation water content changes with dehydration. Peat and muck, especially if coarse or woody, require an overnight imbibition period to obtain a definite endpoint for the saturation point. After the first wetting, pastes of these soils usually stiffen upon standing. Adding water and remixing then yields a mixture that usually retains the characteristics of a saturated paste. With fine-textured soils, enough water should be added immediately, with a minimum of mixing, to bring the sample nearly to saturation. This minimizes the formation of clumps of soil during stirring, speeds the mixing process, and helps attain a more definite endpoint. Care also should be taken not to unduly overwet coarse-textured soils. The presence of free water on the surface of the paste after standing is an indication of oversaturation in the case of coarse-textured soils. Even small amounts of free water can lead to appreciable errors in saturation paste water contents for these materials. However, the effect on the value

of the EC of the extract (EC_e) is small and usually does not significantly affect salinity diagnoses.

Sodium hexametaphosphate is added to the extract to prevent the precipitation of $CaCO_3$ from the extract upon standing. The quantity of $(NaPO_3)_6$ solution added increases the Na concentration about 0.5 ppm, or 0.02 mmol/L, which is inconsequential compared with the possible loss of $CaCO_3$. Alternatively, a subsample of the extract should be analyzed immediately or immediately diluted twofold and used for the Ca and alkalinity determinations.

Alternative methods of preparing the saturated soil paste have been described by Longenecker and Lyerly (1964), who proposed wetting the soil sample on a capillary saturation table, by Beatty and Loveday (1974) and Loveday (1972), who recommended predetermining the amount of water at saturation on a separate soil sample using a capillary wetting technique, and by Allison (1973), who recommended slowly adding soil to water (oversaturation method). Similar results are obtained with these methods. The choice of method is primarily one of personal preference.

Thymol can be added to the paste to minimize the effect of microbial activity on saturation extract composition during equilibration (Carlson et al., 1971).

The extracts should be stored at about 4°C until analyzed.

ELECTRICAL CONDUCTIVITY OF WATERS AND AQUEOUS EXTRACTS

Principles

Electrical conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. It is generally related to the total solute concentration and can be used as a quantitative expression of dissolved salt concentration, even though it is also affected by the mobilities, valences and relative concentrations of the individual ions present in the solution.

The determination of EC generally involves the physical measurement of resistance (R) in ohms. The R of a conducting material (such as a saline solution) is inversely proportional to its cross-sectional area (A) and directly proportional to its length (L). The magnitude of the R measured therefore depends on the characteristics (dimensions and spacings) of the conductivity cell and electrodes. Specific resistance (R_s) is the R of a cube of the sample 1 cm on edge. Practical conductivity electrodes are not of this dimension and measure only a given fraction of the specific R ; this fraction (R/R_s) is commonly referred to as the cell constant (K).

The reciprocal of R is conductance (C). It is expressed in reciprocal ohms, or mhos. When the cell constant is applied, the measured conductance at a specified temperature is converted to specific C , the reciprocal of the specific R (herein called EC)

$$EC = \frac{1}{R_s} = K/R. \quad [2]$$

