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A Textbook of
SOIL
Chemical Analysis

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SOIL
Chemical Analysis

P. R. HESSE Ph.D., F.R.I.C.

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Preface

The author was trained as a 'pure' chemist and for many years revelled in the niceties of exactitude obtainable in the analysis of true solutions or homogeneous solids. Soil, if it was ever considered at all, was something in which to grow cabbages. Not until force of circumstance precipitated him into basic soil research did he appreciate the enormous challenge of attempting a meaningful analysis of a substance having a constantly changing chemical and biological equilibrium.

Nearly every university now has a Soil Science department and many confer degrees or diplomas specifically in that subject. All such degree courses include, or should include, practical instruction in the chemical analysis of soil. In many cases students merely work through a series of routine analyses using methods established many years ago and, although outmoded, perpetuated in lecture notes. There is no generally accepted textbook on the subject as exists for pure chemical analysis.

Several factors have to be considered when teaching, or learning, soil analysis; the practical manipulation and often the improvisation of apparatus, the chemical theory of the analysis in question, the theory underlying the reason for making the analysis and the interpretation of the result. Most courses rely upon the student attending chemistry lectures to absorb the necessary chemical theory, but although training in chemistry is essential, many theories applying to chemical systems are not applicable to complex biological systems as exist in soil.

One of the prime aims of this textbook is to narrow the gap between the theory of soil chemistry and the manuals of soil analysis. A manual is incorporated, however, thus freeing the student from additional expenditure, and as it is based upon the preceding theoretical discussion it should be more meaningful than the 'cook-book' type of manual.

The subject of soil analysis is relatively young and procedures are constantly being modified, improved or superseded. The only way of competently performing many analyses has been by reference to articles in scientific journals which are not always readily available and not all analysts are up to date in their reading. There is often controversy over the relative merits of different analytical procedures and it is even unknown for one soil laboratory to use method B because another is using method A. At every soils conference there is a plea and even a decision to standardize methods but this, like many conference decisions, has failed to materialize. It must be remembered of course that different soils sometimes need different methods for the same analysis.

The author has attempted to bring together the findings and opinions of soil analysts from all over the world during the last hundred years. The

book may, in a way, be considered as a concise history of soil analysis. The scope of the book is such as to include non-routine methods of analyzing soils and to discuss special techniques and apparatus. For example, the routine procedures for determining nitrate and ammonium in soils are of limited value to a modern student of nitrification; also needed is discussion of techniques such as percolation and respiration.

World food shortage has focused attention on the reclamation and use of previously ignored land such as saline swamps. The cultivation and production of rice has boomed with concomitant increased research into waterlogged soils. Soils which are permanently or periodically wet need special consideration with regard to analytical techniques and interpretation of analytical results. Part of the textbook has been expressly designed to meet the problem of analyzing wet soils—a problem hitherto not considered except in isolated technical papers.

Each chapter commences with a brief résumé of the theoretical background of the particular analysis or group of analyses as related to natural soil conditions. An attempt is made to explain the reasons for making the analysis and although the subject is not exhaustively discussed, sufficient facts are presented to ensure that the procedures are not followed merely to obtain a numerical answer. A list of relevant references from which the analyst can select further reading is given in chapter order at the end of the book.

The chemistry of certain soil constituents such as nitrogen and phosphorus has received limited attention as this forms in its own right the subject matter of whole books. Other substances, such as sulphur, which have been rather neglected by soil scientists, have been discussed more fully. Substances such as yttrium and uranium compounds and other fission products are mentioned briefly but sufficient has been said to make the reader aware of their importance in modern soil chemistry.

Specialized analytical techniques such as spectrography, X-ray diffraction, polarography and chromatography are not detailed although they are commonly used in soil analysis. This is to avoid duplication as experimental details can be found elsewhere; where such techniques can be applied is indicated with relevant references.

Recommended analytical methods have been chosen with the facilities of the average soil laboratory in mind. Other factors being equal, preference has been given to procedures involving simple apparatus and commonly available reagents. More intricate apparatus usually intended for specialized research has not been ignored but a 'do-it-yourself' attitude is encouraged. The author has had long experience of laboratories in out-of-the-way parts of the world where delivery of supplies may take over one year and which have operated under a stringent budget. For those who can afford to contact expensive laboratory furnishers the textbook will give ample scope and at the same time will serve its purpose in student education. When it comes to recommended procedures, however, the student himself has been catered for; not every analyst has a scintillator-spectrometer for measuring

potassium and so, although the method is mentioned, more emphasis is placed upon simple flame photometry.

Nearly every chapter has a section dealing with the determination of plant-available nutrients but a recommended procedure is not necessarily given. The myth of soil analysis being the answer to the farmer's problem is not kept up. The reader will repeatedly be reminded that field experiments are essential.

In order to conform with the present international trend, SI units have been used throughout this book. Thus °C represents degrees Celsius and not degrees Centigrade, although in practice no conversion factor is involved. The Celsius scale is being retained until such time as the Kelvin scale has been more generally accepted.

No doubt many readers (like the author) will, for a while, be mildly exasperated at having to translate mentally 'cm³' into the more familiar 'ml' and in making standard solutions with $x \text{ g dm}^{-3}$ instead of with $x \text{ g/l}$. More drastic changes, though, exist for the soil analyst; for example the replacement of mmhos/cm by mS cm^{-1} (millisiemens per cm) and the use of the pascal (N m^{-2} or newton per square metre) instead of atmospheres. In such cases therefore, the more commonly understood unit is given in parentheses after the SI unit. Non-SI units have been retained when quoting the results of previously published work and the corresponding SI units put in parentheses. The unit used in the special field of ion exchange has been retained as the milliequivalent. Some of the more frequently used symbols are explained in Appendix III.

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The author has drawn not only upon his own experience but upon that of many colleagues and he wishes to express his gratitude for their permission to do so. Numerous published works, books and articles in periodicals have been consulted and these are acknowledged in the text. Certain diagrams and tables have been reproduced with the kind permission of their authors and publishers and these too are acknowledged in the text. Certain of the photographs have been provided by institutions and commercial firms as acknowledged in the captions. Special thanks are due to colleagues, students and assistants who have helped in the practical investigations necessary and to those who have criticized the text, in particular Dr P. B. Tinker of Oxford University, and Drs S. J. Tinsley and J. W. Parsons of Aberdeen University.

Contents

PREFACE	v
1 INTRODUCTION	
1 Origin and nature of soils	1
2 Purpose of soil analysis	2
3 Precision and accuracy	3
4 Recording and presentation of results	6
Immediate recording	6
Permanent recording	6
Reporting	6
Checking	7
Expression	7
5 Interpretation of results	8
2 PREPARATION OF SOIL SAMPLES FOR ANALYSIS	
1 Reception at the laboratory	10
2 Drying	10
Methods	10
Effects	12
<i>Chemical 12 Physical 14</i>	
3 Grinding and sieving	14
4 Storage	16
5 Determination of moisture content	17
3 SOIL REACTION (pH VALUE) AND LIME POTENTIAL	
1 Introduction	19
2 pH values	20
Background and theory	20
Electrometric measurements	22
<i>Methods 22 Sources of error 24 Limits of accuracy 26</i>	
Colorimetric measurement	26
<i>Methods 26 Sources of error 27</i>	
<i>Limits of accuracy 28 Field measurement 28</i>	

3	Lime potentials	28
4	Recommended methods: pH	30
	Electrometric measurement in the laboratory	30
	<i>Preparation 30 Measurement 30</i>	
	Electrometric measurement in the field	31
	<i>Soil sampling 31 Measurement 32</i>	
	Colorimetric measurement in the laboratory	32
	Colorimetric measurement in the field	33
5	Recommended method: Lime potential	34
4	TITRATEABLE ACIDITY, EXCHANGEABLE HYDROGEN, AND LIME REQUIREMENT	
1	Introduction	35
2	Titrateable acidity	35
3	Exchangeable hydrogen	36
4	Lime requirement	38
5	Recommended methods	40
	Titrateable acidity	40
	Exchangeable hydrogen	41
	Lime requirement	43
5	INSOLUBLE CARBONATES AND SULPHUR REQUIREMENT	
1	Introduction	45
2	Insoluble carbonates	46
	Total insoluble carbonates	46
	Magnesium carbonate	48
3	Sulphur requirement	51
4	Recommended methods	52
	Carbonates	52
	<i>Total insoluble carbonates 52 Calcium and magnesium carbonates 57 Field test 58</i>	
	Sulphur requirement	59
	<i>From cation exchange data 59 Buffer method 60</i>	
6	SOLUBLE SALTS	
1	Introduction	61
2	Determination: Background and theory	63
	Total salinity	63
	Individual soluble ions	72
	<i>Chloride 72 Carbonate and hydrogen carbonate 73</i>	
	Gypsum	74

3	Recommended methods	75
	Total salinity	75
	<i>By paste resistance</i> 75	
	<i>By electrical conductivity of extracts</i> 81	
	Soluble cations (<i>see under appropriate element</i>)	
	Soluble anions	83
	<i>Carbonate and hydrogen carbonate</i> 83	
	<i>Chloride</i> 85	
	Gypsum	85
	<i>Conductance</i> 85	
	<i>Deb's method</i> 86	
7	CATION AND ANION EXCHANGE PROPERTIES	
1	Introduction	88
	Cation exchange in soils	88
	Anion exchange in soils	90
2	Determination: Background and theory	91
	Cation exchange capacity	91
	Total exchangeable cations	98
3	Recommended methods	101
	Cation exchange capacity	101
	<i>Using sodium as index ion</i> 101	
	<i>Using barium as index ion</i> 102	
	Total exchangeable bases	103
	Individual exchangeable bases	104
	<i>Preparation of leachate</i> 104	
	Anion exchange capacity	105
8	CALCIUM AND MAGNESIUM	
1	Introduction	106
2	Calcium	106
	Background and theory	106
	Total calcium	107
	Exchangeable calcium	107
	Calcium in solution	108
3	Magnesium	113
	Background and theory	113
	Total magnesium	115
	Exchangeable magnesium	115
	Available magnesium	116
	Magnesium in solution	117

4	Recommended methods	120
	Total calcium	120
	Exchangeable calcium	120
	Calcium in solution	120
	Total magnesium	124
	Exchangeable magnesium	124
	Available magnesium	124
	Magnesium in solution	125
9	POTASSIUM AND SODIUM	
1	Potassium	127
	Background and theory	127
	<i>Forms of potassium in soil</i> 127 <i>Fixation of soil potassium</i> 127 <i>Role of potassium in plant nutrition</i> 129	
	Determination	130
	<i>Total potassium</i> 130 <i>Exchangeable potassium</i> 131 <i>Available potassium</i> 131 <i>Potassium in solution</i> 136	
2	Sodium	138
	Background and theory	138
	Determination	140
	<i>Total sodium</i> 140 <i>Exchangeable sodium</i> 140 <i>Sodium in solution</i> 141	
3	Recommended methods	143
	Total potassium	143
	Exchangeable potassium	143
	Available potassium	143
	<i>Woodruff's method</i> 143 <i>Activity ratios and quantity-intensity</i> 144	
	Total sodium	146
	Exchangeable sodium	146
	Exchangeable sodium percentage	147
	Sodium in solution	147
	<i>Potentiometric</i> 147	
10	NITROGEN	
1	Introduction	149
	<i>Forms in soils</i> 149	

2 Determination: Background and theory	150
Total nitrogen	150
<i>The Kjeldahl process</i> 150	
<i>Other methods</i> 165	
<i>Conclusions</i> 165	
Ammonium-nitrogen	166
<i>Extraction</i> 166	
<i>Determination</i> 168	
<i>Non-exchangeable</i> 173	
<i>Capacity for ammonia retention</i> 176	
Nitrite-nitrogen	178
<i>Extraction</i> 178	
<i>Determination</i> 178	
Nitrate-nitrogen	180
<i>Extraction</i> 180	
<i>Determination</i> 180	
Available nitrogen	183
<i>Mineralization of nitrogen</i> 185	
<i>Techniques for measurement</i> 188	
Denitrification	195
3 Recommended methods	197
Standard solutions	197
Total nitrogen: Kjeldahl	197
Exchangeable ammonium-nitrogen	200
<i>Extraction</i> 200	
<i>Measurement</i> 200	
Non-exchangeable ammonium-nitrogen	201
Nitrite-nitrogen	201
<i>Extraction</i> 201	
<i>Measurement</i> 202	
Nitrate-nitrogen	202
<i>Extraction</i> 202	
<i>Measurement</i> 202	
Available nitrogen: <i>Anaerobic incubation</i>	203
Amino nitrogen (<i>see Chapter 11, §11:3:6</i>)	203
11 CARBON AND ORGANIC MATTER	
1 Introduction	204
2 Background and theory	204
Total carbon	204
Total organic matter	205
<i>Total organic carbon</i> 205	
<i>Expression of results</i> 209	
<i>By weight loss</i> 209	
Oxidizable organic matter	211
<i>Expression of results</i> 215	
Fractionation of organic matter	216
<i>Fractionation of humic acid</i> 219	

<i>Fractionation of fulvic acid</i>	220	<i>Carbohydrates</i>	221
<i>Lignin</i>	223	<i>Amino compounds</i>	223
Acidity in organic matter			225
Carbonyl groups in organic matter			225
Organic matter decomposition			226
<i>Factors affecting measurement</i>	238		
Carbon–nitrogen ratios			242
3 Recommended methods			242
Total carbon			242
Total organic carbon			245
Oxidizable organic carbon			245
Fractionation of organic matter			246
Carbohydrates			247
Amino-compounds			249
Organic matter decomposition			250
<i>Apparatus and method</i>	250	<i>Calculations</i>	253
12 PHOSPHORUS			
1 Introduction			255
2 Background and theory			255
Total phosphorus			255
Inorganic phosphorus compounds			256
<i>Total inorganic phosphorus</i>	256		
<i>Fractionation of inorganic phosphorus compounds</i>	257		
Organic phosphorus compounds			261
<i>Total organic phosphorus</i>	261	<i>Specific organic compounds</i>	267
Mineralization of organic phosphorus			269
Available phosphorus			271
<i>Nature, source and determination</i>	271	<i>Chemical methods of determination</i>	272
<i>Phosphate potentials</i>	277	<i>Biological methods of determination</i>	282
Phosphorus fixation			283
<i>Determination</i>	286		
Phosphorus in solution			288
<i>Volumetric methods</i>	288	<i>Gravimetric method</i>	288
<i>Colorimetric methods</i>	289		
3 Recommended methods			294
Phosphorus in solution			294

Total phosphorus	295
Total inorganic phosphorus	296
Total organic phosphorus	296
Fractionation of inorganic phosphorus	296
Phospho-lipids	297
Inositol hexaphosphate	298
Available phosphorus	299
Phosphorus fixation capacity	300

13 SULPHUR

1 Introduction	301
<i>Available sulphur</i> 302	
<i>Effects on soil properties</i> 304	
<i>Acid sulphate soils</i> 304	
2 Determination: Background and theory	305
Total sulphur	305
Sulphate-sulphur	307
<i>Extraction and determination</i> 308	
<i>Gravimetric method</i> 309	
<i>Volumetric methods</i> 310	
<i>Colorimetric method</i> 311	
<i>Conductometric method</i> 311	
<i>Turbidimetric method</i> 311	
<i>Reduction method</i> 314	
Sulphide-sulphur	315
Elemental sulphur	316
Other inorganic sulphur	317
Organic sulphur	317
3 Recommended methods	319
Total sulphur	319
Sulphate-sulphur	321
<i>Extraction</i> 321	
<i>Determination</i> 322	
Available sulphur	324
Sulphide-sulphur	324
Elemental sulphur	326
Organic sulphur	326
<i>Total organic sulphur</i> 326	
<i>Methionine</i> 327	
<i>Cystine and cysteine</i> 329	

14 IRON, ALUMINIUM AND MANGANESE

1 Iron	332
Introduction	332
<i>Occurrence of iron</i> 332	
<i>Movement of iron</i> 334	
<i>Available iron</i> 335	

Determination: Background and theory	335
<i>Total iron</i> 335 <i>Total iron(II)</i> 336 <i>Free oxides</i> 336	
<i>Available iron</i> 337 <i>Iron in solution</i> 338	
2 Aluminium	340
Introduction	340
<i>Occurrence of aluminium</i> 340 <i>Exchangeable aluminium</i> 341	
<i>Aluminium and plant growth</i> 341	
Determination: Background and theory	343
<i>Total aluminium</i> 343 <i>Available aluminium</i> 343	
<i>Exchangeable aluminium</i> 343 <i>Aluminium in solution</i> 345	
3 Manganese	348
Introduction	348
<i>Occurrence of manganese</i> 348 <i>Manganese and plant growth</i> 349	
Determination: Background and theory	350
<i>Total manganese</i> 350 <i>Available and exchangeable manganese</i> 350	
<i>Active or free manganese</i> 351	
<i>Manganese in solution</i> 351	
4 Recommended methods	353
Total iron, aluminium and manganese in the same sample	353
Total iron	353
Total iron(II)	354
Available iron(II)	354
Free iron oxides	355
Removal of free iron oxides	355
Iron in solution	355
Field test for iron(III) and (II)	357
Total aluminium	357
Available, extractable and exchangeable aluminium	357
Aluminium in solution	358
Total manganese	358
Exchangeable manganese	359
Water-soluble manganese	360
Easily reducible manganese	360
Field test for manganese	361

15 SILICON, TITANIUM AND SESQUIOXIDES	
1 Introduction	362
2 Silicon	362
Background and theory	362
Determination	364
<i>Total silicon 364 Water-soluble silicon 364</i>	
<i>Silicon in solution 364</i>	
3 Titanium	365
Background and theory	365
Determination	365
4 Sesquioxides	366
Background and theory	366
<i>Calculations 367</i>	
5 Recommended methods	367
Total silicon	367
Water-soluble silicon	368
Titanium	369
Silica–sesquioxide ratios	369
16 TOTAL (ELEMENTAL) ANALYSIS AND SOME TRACE ELEMENTS	
1 Introduction	371
Total analysis	373
Trace nutrient elements	374
Elements in solution	374
Calculations	374
2 Fusion and digestion of soils	375
Fusion	375
<i>Sources of error in sodium carbonate fusion 375</i>	
<i>Fusion with other materials 375</i>	
Acid digestion	376
<i>Perchloric–nitric acids 376 Perchloric–hydrofluoric acids 376</i>	
Precautions	376
Recommended methods	377
3 Antimony	378
Background and theory	378
<i>Determination</i>	378
Recommended method	378

4 Arsenic	379
Background and theory	379
<i>Determination</i>	379
Recommended method	380
5 Barium	381
Background and theory	381
<i>Determination</i>	382
Recommended method	382
6 Beryllium	382
Background and theory	382
<i>Determination</i>	382
Recommended method	383
7 Boron	384
Background and theory	384
<i>Determination</i>	384
Recommended methods	386
<i>Total boron</i>	386
<i>Water-soluble boron</i>	387
8 Bromine	387
Background and theory	387
<i>Determination</i>	387
Recommended method	388
9 Cadmium	388
<i>Determination</i>	389
10 Caesium	389
<i>Determination</i>	390
11 Chlorine	390
<i>Determination</i>	390
12 Chromium	391
<i>Determination</i>	391
13 Cobalt	392
Background and theory	392
<i>Determination</i>	392
Recommended methods	393
14 Copper	395
Background and theory	395
<i>Occurrence</i>	395
<i>Total copper</i>	397
<i>Available copper</i>	397
<i>Copper in solution</i>	397
Recommended methods	398

	<i>Total copper</i> 398	<i>Available copper</i> 398	<i>Copper in solution</i> 399	<i>Organically complexed copper</i> 400
15	Fluorine			401
	Background and theory			401
	<i>Occurrence</i> 401	<i>Total fluorine</i> 401	<i>Available fluorine</i> 401	<i>Fluorine in solution</i> 401
	Recommended methods			403
	<i>Total fluorine</i> 403	<i>Fluorine in solution</i> 403		
16	Germanium			404
17	Gold			405
	Background and theory			405
	<i>Determination</i> 405			
	Recommended method			405
18	Iodine			406
	<i>Determination</i> 406			
19	Lead			407
	Background and theory			407
	<i>Determination</i> 407			
	Recommended method			407
20	Lithium			408
	Background and theory			408
	<i>Determination</i> 408			
	Recommended method			409
21	Mercury			409
	Background and theory			409
	<i>Determination</i> 410			
	Recommended method			410
22	Molybdenum			413
	Background and theory			413
	<i>Determination</i> 414			
	Recommended methods			415
	<i>Total molybdenum</i> 415	<i>Available molybdenum</i> 416	<i>Molybdenum in solution</i> 416	
23	Nickel			417
	<i>Determination</i> 417			
24	Rubidium			418
25	Selenium			418
	Background and theory			418
	<i>Determination</i> 419			

	Recommended methods	420
	<i>Total selenium</i> 420 <i>Extraction of water-soluble selenium</i> 421 <i>Selenium in solution</i> 421	
26	Silver	423
27	Strontium	423
	<i>Determination</i> 424	
28	Thallium	424
	<i>Determination</i> 425	
29	Tin	425
30	Tungsten	425
	Background and theory	425
	<i>Determination</i> 425	
	Recommended method	426
31	Uranium	427
32	Vanadium	427
	<i>Occurrence in soils</i> 427 <i>Determination</i> 427	
33	Yttrium	428
34	Zinc	428
	Background and theory	428
	<i>Total zinc</i> 429 <i>Available zinc</i> 429 <i>Zinc in solution</i> 430	
	Recommended method	431
35	Zirconium	433
	Background and theory	433
	<i>Determination</i> 433	
	Recommended method	434
17	OXIDATION–REDUCTION POTENTIALS	
1	Background and theory	436
	Introduction	436
	Oxidation–reduction potentials	438
	<i>Measurement</i> 440	
	<i>rH</i> values	441
	Relationship between <i>Eh</i> and pH	441
	Reducing power and redox levels	447
2	Potentiometric measurement	448
	Apparatus	448
	Preparation of soil samples	452
3	Colorimetric measurement	455

4	Recommended methods	458
	Potentiometric: line-operated pH meter method	458
	<i>Preparation of soil sample</i> 458 <i>Apparatus</i> 458	
	<i>Procedure</i> 459	
	Potentiometric: independent salt bridge method	459
	<i>Preparation of soil sample</i> 459 <i>Apparatus</i> 459	
	<i>Procedure</i> 460	
18	WATERLOGGED SOILS	
1	Background and theory	461
	Introduction	461
	Chemistry	462
	<i>Anaerobic decomposition and nitrogen changes</i> 463	
	<i>Iron(III)–iron(II) systems</i> 464 <i>Changes in reaction</i> 465	
	Classification	466
2	Field sampling, storage and preparation	467
	Introduction	467
	Field sampling	467
	Storage and preparation	469
3	Laboratory sampling	470
4	Special analytical methods	471
	Procedures	471
	Calculation and interpretation	472
5	Special laboratory techniques for study	473
	APPENDICES	
I	Selected international atomic weights and preparation of standard solutions	477
II	Strength of common acids and alkalis and preparation of standard solutions	480
III	Conversion factors, definitions and interrelationships	481
IV	Bibliographical note	483
	REFERENCES	485
	INDEX	513

Plates

between pages 6 and 7

- 1 Riffle sampler for reducing the bulk of soil samples
- 2 Battery-operated conductivity bridge for field work
- 3 Detail of battery-operated bridge showing conductivity cells
- 4 Bureau of Soils cup and holder for measuring the resistance of saturated soil paste
- 5 Apparatus for the preparation of soil saturation extracts
- 6 Portable vacuum filtration apparatus for saturation extracts
- 7 Pressure membrane apparatus for extracting soil solutions
- 8 Pipette-type conductivity cell and Solu bridge
- 9 Detail of pipette-type conductivity cell
- 10 Semi-micro Kjeldahl flasks on electric digestion stand with glass fume exhaust
- 11 Jenkin mud sampler

Introduction

1:1 ORIGIN AND NATURE OF SOILS

Of the great many different kinds of soil on earth, each has its own special collective characteristics upon which its behaviour will depend; consequently, any definition of the term 'soil' must be comprehensive in context. It is necessary to distinguish between soil as a substance and soil as a natural body. In the laboratory there is an unfortunate, yet persistent, tendency to regard soil as a material in a bottle, but in the field due notice must be taken of the fact that soils have shape, volume, boundaries and so on. A general definition of soil is given in the *Soil Survey Manual* of the United States Department of Agriculture (1951):

'Soil is the collection of natural bodies occupying portions of the earth's surface that support plants and that have properties due to the integrated effects of climate and living matter acting upon parent material, as conditioned by relief, over periods of time.'

This definition takes into account the landscape nature of soils and the many varied combinations of genetic factors.

The origin of soil is the earth's crust, the primary rocks of which have weathered to produce what is known as 'soil parent material'. The rocks from which parent material is formed are known as 'parent rocks' and these may in some cases be the primary rocks themselves, or secondary rocks formed by weathering.

Four main classes of soil parent material are now recognized:

- i. Those formed *in situ* from soft rocks such as chalk and volcanic ash.
- ii. Those formed *in situ* from hard rocks which may be igneous (e.g. granite and basalt), sedimentary (e.g. limestone and sandstone) or metamorphic (e.g. gneiss and marble).
- iii. Those transported from their place of origin before weathering to form soils. This is the most important of the parent material classes and includes all material moved by water (such as alluvium, colluvium, lacustrine deposits and marine and beach sediments), by wind (such as loess and eolian sands), and by glacial phenomena such as glacial drift and till.
- iv. Those formed from organic material such as peat.

Rocks are weathered mechanically by the action of frost, water and wind, and chemically by such processes as oxidation, hydration, hydrolysis

2 | Introduction

and solution in carbonic acid. Similar or even identical rocks can produce quite different materials according to the kind of weathering.

The products of the weathered rock, that is, the soil parent material, then undergo further change to produce soil as we know it. This process does not necessarily occur at the same time or place as production of parent material, nor on the other hand is a parent material necessarily found between a soil and its parent rock; for example, the soil-forming processes may have proceeded concomitantly with rock weathering. In many soils as now found, more than one parent material may have been involved in their formation.

The process of soil formation from parent material can be divided into two main parts. One involves addition and admixture of organic matter and the other involves solution and translocation of certain constituents and is known as 'eluviation'. Eluviation itself can be further divided into mechanical eluviation (movement of finer particles, usually downwards by washing) and chemical eluviation, which involves the partial decomposition of colloidal matter with subsequent movement of the decomposition products.

Either after or during the formation of a soil, its nature can be modified by otherwise unrelated effects such as volcanic action, floods, erosion and human activities.

The eventual soil is a chemically, physically and biologically complex, dynamic system, the constituents of which are constantly undergoing change. Hall (1949)* likens soil to a 'three-phase system of solid, liquid and gaseous components with constantly shifting equilibria'.

The reader is referred to standard works on soil genesis and constitution (e.g. Hall, 1949; Russell, 1961) for a full discussion of the origin of soils, but from the analyst's point of view it is apparent that soil, even as a material in a bottle, provides a stimulating challenge to his skill and capabilities.

1:2 PURPOSE OF SOIL ANALYSIS

Any particular soil may be analyzed for such varied reasons as to predict its behaviour if planted with wheat, if used to construct a road, if drained, if fertilized and so on; or it may be analyzed to discover its origin or to see if a certain constituent is present or to investigate the effects of a changing environment.

In the study of soil as a nutrition factor, for example, the analyses may be of a routine nature for advisory purposes, or highly specific as in basic research. For the classification of soils as in a soil survey, again the analyses may be general in nature, but if a soil map is required illustrating a particular soil property such as salinity, then specific analyses are called

* All references are listed in chapter order on pp. 485-512.

for. In this book emphasis is upon the analysis of soil in its capacity as a medium for plant growth but the reader should realize that factors other than the chemistry of a soil influence plant growth. Such factors include climate, microbiological conditions, structure and other physical aspects. These factors are but briefly touched upon here but their importance in limiting the usefulness of soil chemical analysis must be emphasized.

For whatever purpose a soil is to be analyzed, plans should be made in advance as to what analyses are required, why they are required and what level of accuracy is needed. This is particularly important when an investigator has the analyses done for him rather than performing them himself, as all too often a field or research worker will demand an excessive number of analyses chosen from the book rather than from the requirements of the investigation.

1:3 PRECISION AND ACCURACY

The degree of accuracy with which a determination is carried out is very often a neglected factor in soil analysis and a particular procedure is merely followed from written instructions without reference to the purpose of analysis. As a consequence some measurements are made with unnecessary accuracy, often at the expense of considerable time. For example, there is no point in using a lengthy gasometric method to measure the carbonate content of a soil to three decimal places when all that is called for is a statement as to whether or not the soil is calcareous. The farcicality of such a measurement is more apparent if one considers that perhaps 1 g of a 2-mm sample will have been used in the experiment, this 1 g having been taken from about a kilogramme sample, itself selected from a much larger bulk of soil which may or may not have been representatively sampled in the field. The variability of calcium carbonate distribution in a soil could be such that the sampling error is enormous.

Another and oft-quoted example is that of pH determination. It is well known that the pH of a soil may vary by as much as a whole unit over a relatively small area, and thus it is pointless (as a routine) to measure pH values with greater accuracy than 0.2 or even 0.5 of a unit.

Conversely, some analyses are made with insufficient attention to accuracy. For example in a specific experiment concerning changes in pH of a particular soil sample with time or with certain treatments such as dilution of suspension, it may be necessary to obtain results much closer than to within 0.2 of a unit. This is the case for example when measuring exchange acidity by Brown's method (section 4:3). Similarly great accuracy would be demanded in ammonium or nitrate determination when investigating the effect of certain treatments upon nitrification in a soil, whereas it would be unnecessary to achieve such accuracy when analyzing as a routine for the inorganic nitrogen content of a soil.

Another common malpractice is to achieve an apparent accuracy by

4 | Introduction

applying mathematical techniques to an experimental result out of proportion to the accuracy with which that result was obtained. An extreme example of this was once encountered by the author who found a soil chemist reporting fertilizer responses to seven decimal places using yield figures obtained in lb/acre to the nearest 5 lb. His explanation was that the calculating machine gave the answer to seven decimal places. While such examples are fortunately rare, less obvious cases are extremely common and it should be borne in mind that in order to be significant, only the last reported digit may be uncertain.

The chief errors involved in a chemical analysis have been adequately discussed by, *inter al.*, Vogel (1962). Additional remarks of particular relevance to soil analysis can be made regarding personal errors, sampling errors and errors of method.

Personal errors, apart from those due to bad technique and carelessness, are due to personal characteristics which influence results in a standard manner, resulting in bias. These errors can be revealed by having the same analysis performed by more than one person but in practice such a procedure is not justified as a routine. It is, however, an advisable precaution when testing new or modified procedures.

Errors of method, although common, are difficult to detect. Good agreement of replicate analyses is meaningless if the method of analysis itself does not yield the correct result. As an example we can consider the Kjeldahl procedure for measuring total nitrogen in a material which may contain much nitrate; unless the necessary modifications to the method are introduced the nitrate-nitrogen will remain undetected. Whenever possible, but always when introducing a new method, independent methods of measuring the same quantity should be used. A control analysis using material of known composition should always be carried out and helps detect errors of method. Standardization of method permits the comparison of analyses of different soils made in different places. It is far more important to use a standard method of analysis than to modify it for what may be a slight increase in accuracy. Not that such modifications are to be discouraged, but their adoption should be made by all if results are to be comparable. Regression analysis can be used to compare statistically a new method of analysis with a standard method and an example of this is given by Pantony (1961). All proposed new methods of soil analysis should have a statement of the standard deviation and of the number of degrees of freedom involved in its calculation.

Sampling errors are perhaps the most common errors encountered in soil analysis owing to the extreme heterogeneity of material. In the laboratory such errors can be minimized by grinding and mixing, but the real sampling error occurs in the field. Again, procedures exist for reducing these, but interpretation of soil analyses must always be made keeping in mind the probabilities of field sampling errors.

As a general means of reducing errors all experiments should include a blank determination which will reveal errors of reagent, a control which

Index

Page references in bold type are to the principal treatment of the subject.

- 'A' values: **281, 303**
absolute error: 5
accuracy: of determinations, 3-6, 24, 26, 28, 46, 50, 52, 64, 72, 73, 85, 102, 112, 120, 121, 142, 143, 158, 159, 162, 171, 178, 182, 210, 211, 243, 310, 340, 345, 347, 353, 395, 419, 429, 433, 434, 454, 457, 460
acidity: of humus, 225, 238; titrateable, 35, 40, 333, 341
acid stability plateau: 290
acid sulphate soils: 63, 304-5, 332, 462, 469
actinomycetes: 17
activity: coefficients, 24, 26, 27, 142, 145; errors, 25; of ions, 19, 24, 25, 29, 35, 142, 257, 423, 439; ratios, 117, 132-6, 141, 143, 144-6
agar-salt bridge: 22, 73, 449, 459
air-dry soil: *see* Drying
alkali soils: 51, 61, 94, 106, 129, **138, 139, 149, 384**
alkaline pyrogallol: 469
alluvium: 1, 274, 433
aluminium: accumulators, 342; determination - available, 343, 357, exchangeable, 343, **357**, in solution, 345-8, **358**, recommended methods, 357-8, total, 343, 357; exchangeable, 35, 39, 89, 341, 343, 357; occurrence, 340, 469; oxides, 340, 344, 363, 391, 414; phosphate, 257-61, 273, 276, 281, 283-6; soil acidity and, 20, 35, 36, 37-8, 39, 41, 52, 341; toxicity of, 20, **341-3, 395, 414**
aluminon: reagent for aluminium, 345
amino-acids: 129, 216, 220, 223-4, 235, 239, 249-50, 303, 318, 395, 428; determination, 223, 249-50, 318
amino-compounds: 216, 223, 249
1,2,4-Aminonaphtholsulphonic acid: as reductant, 289, 292
ammonia: absorption of - by soil, 176-8, in boric acid, 162; distillation of, 169, 198-9; forms of in soil, 166, 173
ammonification: **185, 304, 463-4**
ammonium acetate: as soil extractant, 36, 91, 98, 101, 103, 107, 115, 131, 140, 314, 343, 359, 471
ammonium fluoride: as soil extractant, 259-61, 263, 274
ammonium-nitrogen: determination, 158-164, 200, 201, by distillation, 169, 200, **201**, by microdiffusion, 170-2, by Nesslerization, 170; extraction, 166, 200, 471; non-exchangeable, 173, 199, 201
ammonium purpurate: 109
anaerobic: conditions in soil, 129, 186, 195, 391, 413, 442; decomposition in soil, 337, 436, 438, 463, 473, 476
analysis: purpose of, 2, 21, 287, 371, 410, 433, 467
Angstrom: 481
anhydron: 46, 55, 205, 206, 243
anion exchange: capacity, 90, 105
antagonism: ionic, 9, 114, 115, 129, 130
antimony: determination, 378; electrode, 38; occurrence, 378
antipyrone: 365
arc spectrography: *see* Spectrography
arcometer: 71
arid soils: 14, 19, 45, 61, 139, 301, 362, 384, 418
arsenic: determination, 379; occurrence, 379
ascarite: 243
ascorbic acid: as reductant, 293, 415
Aspergillus niger: 116, 132, 184, 283, 302, 350, 430
asymmetric potential: 23
atomic absorption: 117, 120, 408, 424
atomic weights: 477-479
aurin tricarboxylic acid: *see* Aluminon
autoanalysis: 120, 137, 166, 294, 455
available ions: 9, 88, 106, 116, 124, 127, 128, 131, 143, 149, 183-5, 203, 204, 271-83, 302, 337, 343, 350, 471
azotobacter: 283

bacteria: 17, 195, 240, 283, 302, 305, 335, 348, 350, 351, 390
bacterial potential: 446
barium: chloride, reagent for sulphate, 309, 310, 312, 314; determination, 382; diphenylamine sulphonate indicator, 213; occurrence, 381
basalt: 1, 336
base exchange: *see* Cation exchange
base saturation: 29, 38, 43, 98, 101, 481
basic ferric sulphate: 305, 332
bauxite: 19, 340, 364
Beer-Lambert Law: 118, 291, 340, 353, 365, 398, 423, 481
benzidine: reagent for sulphate, 310, 311

- beryllium: determination, 382; occurrence, 382
- bicarbonate: *see* Hydrogen carbonate
- 2,2'-Biquinoline: 398, 400
- black alkali soils: 139
- boric acid: 162
- boron: determination – total, 384, 386, water-soluble, 385–7; occurrence, 384; toxicity of, 384
- Brilliant Green: reagent for gold, 405
- Brilliant Yellow: reagent for magnesium, 119
- bromocresol green: 162, 383
- bromine: determination, 387; occurrence, 387
- Brönsted: theory of indicators, 26
- buffer: action of soils, 20, 39; power, 146; solutions, 22, 24, 36, 37, 39, 43, 92, 120, 250, 291, 311, 337, 339, 343, 355, 356, 358, 360, 393, 404, 411
- cadmium: in soils, 388
- caesium: in soils, 389
- calcareous soils: 21, 29, 45, 52, 107, 115, 308, 332, 363, 437, 464, 465
- calcimeter: 47, 49
- calcite: 45, 50, 51, 332
- calcium: carbonate – determination, 46–8, 52–7, occurrence, 45, 106; determination – exchangeable, 107, 471, in solution, 108–113, recommended methods, 120–4, total, 107; function of, 106–7; occurrence, 45, 106; quick test for, 112; sulphate – as soil amendment, 51–2, 59, 107, determinatin, 74, 85
- Calcon: indicator, 110
- calomel electrode: *see* Electrodes, calomel
- carbamate: 109, 112, 118, 397, 418, 431
- carbohydrates: 129, 216, 219, 221–3, determination, 247–9
- carbon: determination – total, 13, 204, 242–50, total organic, 205–211, 245, total oxidizable, 211–16, 245, 471; forms of in soil, 204
- carbonate: determination – gravimetric, 46, 54, manometric, 47–50, 55, 57, rapid, 46, 52, volumetric, 46, 54; occurrence, 45, 61, 72, 204; soluble, 73, 83
- carbon dioxide: determination, 253; effect on pH of, 21–2, 32, 350, 465; effect on phosphate potential, 280–1; evolution of, 226–42, 436; partial pressure of, 22, 37, 280, 465
- carbon-nitrogen ratio: 186, 209, 242, 463
- carbonyl groups: in organic matter, 225; determination, 226
- carmine: reagent for boron, 385
- catalysts: for Kjeldahl digestion, 150, 151–2
- cat-clays: *see* Acid sulphate soils
- cation exchange: 16, 65, 88–90, 91–98, 204, 225, 332, 343, 464; capacity – definition, 88, 481, determination, 91–8, 101–3
- cell: conductivity, 68, 82; constant, 82
- cellulose: 222, 248
- chalk: 1
- checking: results, 7
- chelates: 51, 217, 334, 335, 363, 389, 428
- chernozems: 45, 269, 334
- chestnut soils: 45
- chloride: determination, 72, 85; occurrence, 61, 62, 390
- chlorine: determination, 390; occurrence, 390
- chloroplatinic acid: 450
- chlorophyll: 114, 304, 392, 395
- chlorosis: 107, 114, 129, 335, 349, 429
- chlorostannous acid: reductant, 289–90, 294
- choline: 269
- chromatographic analysis: 118, 216, 220, 224, 268, 269, 299, 319, 331, 417, 427
- chromium: in soil, 391
- clay minerals: 89, 113, 128, 210, 217, 239, 308, 362, 389
- clay-pan: 139, 338
- cobalt: determination, 392–5; occurrence, 392
- coefficient: of variation, 5, 110, 306
- colloids: 2, 45, 118, 308, 309, 312, 334, 346, 349, 362, 381, 391, 405, 408, 418, 423, 437
- colluvium: 1
- colour change interval: 26
- combustion: dry, 205, 243; wet, 206, 243
- complexometric titration: 108–11, 288
- complementary ion effect: 106
- compulsive exchange: 94
- conductance: 65, 481
- conductivity: 61, 62, 65, 68–70, 74, 82–3, 97
- continuous flow percolation: 193, 237
- conversion factors: 481
- Conway units: 170–2, 388
- copper: determination – available, 397, 398, in solution, 397, 399, 400, total, 397, 398; fixation, 396; function in plant nutrition, 395; occurrence: 395, 465
- crumb-size: effect on mineralization, 471
- cupferron: 258, 345, 366
- curcurmin: 383, 385
- cutans: 130
- cysteine: 301, 318, 329, 437
- cystine: 301, 303, 318, 329, 437, 463
- Debye–Huckel equation: 465
- denitrification: 150, 168, 181, 195–7, 438
- Devarda's alloy: 163, 180
- dichlorfluorescein: 72
- diffusion: ionic, *see* Ionic diffusion; micro, *see* Microdiffusion
- digestion: Kjeldahl, 150–8, 470; for total analysis, 110, 255, 295, 306, 335, 343, 350, 353, 369, 373, 376, 377–8, 397, 423, 429
- dimethylglyoxime: 418
- diphenylamine: 213, 455
- discontinuous titration: 225
- dithiol: 415, 425, 426
- dithionite: 51, 217, 259, 261, 336, 351
- dithizone: 389, 393, 407, 410, 423, 430, 431
- dolomite: 39, 45, 50, 116, 401
- double layer: electric, 21, 90, 92
- drift: glacial, 1; pH, 31

- drying: of soils, 10; effect on soil properties, 8, 12-14, 89, 128-9, 166, 186-7, 239-242, 269, 271, 279, 284, 304, 344, 351, 364, 467, 469-70
- Dumas: method for nitrogen, 150, 158, 166
- 'E' value: 282
- Egnér-Riehm's reagent: 273
- electric conductivity: *see* Conductivity
- electric double layer: *see* Double layer
- electrodes: antimony, 38; calomel, 23, 24, 25, 30, 148, 448, 458; carbon, 373; compound, 32; glass, 22-3, 30, 73, 138, 147; gold, 438, 448; hydrogen, 22, 439, 440, 448; platinum, 22, 32, 68, 231, 388, 392, 417, 438, 448, 450, 458, 476; polarization of, 26, 315; potentials, 438-40; quinhydrone, 22, 37, 73, 456; reference, 25, 30-2, 447, 448; silver, 73; sodium, 142, 147-8
- electrodialysis: 108, 132, 219
- electrolytic respirometers: 231-7, 250-4
- electrophoresis: 220, 299
- elements: table of, 477-9
- elemental analysis: 337, 371
- eluviation: 2
- emission spectrophotometry: 108, 117, 136, 141, 373
- Eriochrome Black T: indicator, 110, 112, 117
- Eriochrome Blue Black: indicator, *see* Calcon
- Eriochrome Cyanine R: reagent, 347
- errors: in soil analysis, 3-6, 24-6, 27-8, 50, 64, 91-5, 101, 136, 141, 158, 159, 190, 210, 212, 262, 310, 312, 375, 471
- exchangeable bases: *see* Exchangeable cations
- exchangeable cations: determination, 98-101, 103-5, 115, 120, 124, 131, 140, 143, 146, 166-8, 200, 471; *see* Cations, exchangeable
- exchange capacity: *see* Cation exchange capacity
- exchangeable hydrogen: 35, 36-8, 41, 98, 100
- exchangeable sodium percentage: 51, 59, 139, 147, 481
- expression: of results, 17, 100, 209, 215
- extinction coefficient: 482
- extraction: non-equilibrium, 261
- factor: of organic matter, 209, 215, 246
- Faraday: 438; laws of electrolysis, 438
- ferric iron: *see* Iron (III)
- ferron: 347
- ferrous iron: *see* Iron (II)
- field tests: 31, 33, 58, 357, 361
- fission: products, 389, 424, 428
- fixation: of phosphorus, 19, 270, 283, 342; of potassium, 127-9; of sulphur, 308
- flame photometry: 51, 94, 105, 108, 117, 136-7, 141, 143, 146, 147, 353, 374, 382, 385, 390, 408, 418, 424
- flooding: 413, 437, 438, 461, 464, 473
- fluorides: extraction of phosphorus with, 259-61; *see* Fluorine
- fluorine: determination - available, 401, in solution, 401, 403, total, 401, 403; occurrence, 401
- fluorometric analysis: 120, 220, 383, 433
- fractionation: of organic matter, 216-25, 246; of phosphorus, 257-61; of sulphur, 330
- free energy: concept, 132
- free oxides: determination, 336, 355; occurrence, 333, 340; removal, 337, 355, 391
- freezing: of soil samples, 187, 221, 231, 240
- fulvic acid: analysis, 220, 246, 268; separation, 216-19, 246
- fungi: 17
- furnace: induction, 205, 206
- fusion: of soil samples, 255, 256, 305-6, 335, 343, 353, 364, 373, 375, 414, 425, 426, 429, 433; recommended methods, 377
- gas chromatography: 158
- germanium: in soils, 404
- glass electrodes: *see* Electrodes, glass
- gley soils: 332, 438, 448
- glyoxal *bis* (2-hydroxyanil): reagent for calcium, 112
- goethite: 333, 334, 340
- gold: determination, 405; electrode, *see* Electrodes, gold; occurrence, 405
- gneiss: 1, 365, 433
- gradient elution: 261
- granite: 1, 336, 342, 348, 365, 382, 392, 413, 425, 473
- gravimetric analysis: 46, 54, 71, 72, 111, 137, 141, 256, 286, 288, 309, 338, 364, 365, 366, 374, 391, 397, 433
- Griess-Ilosvay: reaction, 178-9
- grinding: soil samples, 14, 46, 47, 157, 204, 374, 470
- groundwater: 139, 334
- Gutzzeit: test for arsenic, 380
- gypsum: determination, 52, 59-60, 68, 74, 85, 108; occurrence, 45, 61, 71, 95, 301, 401; soil amendment, 52
- Hellige-Truog: pH test, 28, 33
- hematite: 333
- hexosamines: 224, 250
- hexoses: 221, 247
- Hoskins still: 160
- humic acid: analysis, 219; separation, 216, 218-19, 246
- humic: 217
- humus: fractionation, 217; isolation, 216, 246
- hydrogen: electrode, *see* Electrodes, hydrogen; exchangeable, 35, 36-8, 41, 104; ion, 19, 21, 23, 26, 29, 35, 71, 114, 332, 344, 456
- hydrogen carbonate: 437; determination, 73, 83; reagent for phosphorus extraction, 273, 472
- hydrogen sulphide: 302, 304, 315, 316, 319, 336
- hydrolysis: of organic matter, 96, 167, 168, 184, 216-19, 220, 221, 223-4, 249, 263, 264, 266, 269; of salts, 1, 35, 37, 89, 92-4, 332, 335, 362
- 8-hydroxyquinoline: 117, 258, 425, 430

- igneous rocks: *see* Rocks, igneous
 ignition: of soil, 89, 107, 209–211, 243, 256, 263, 266, 296, 367, 375, 376, 397
 incubation: techniques, 39, 188–95, 226–237, 302, 470, 473
 indicator solutions: for ammonia distillation, 162; for carbon determination, 213; for Eh determination, 455–8; for pH determination, 26–8, 32, 33; universal, 27, 33
 indigenous fixed ammonia: *see* Ammonium, non-Exchangeable
 inositol hexaphosphate: 267, 298
 interpretation: of results, in relation to method, 8, 90, 187, 240, 327, 472; in relation to purpose, 8, 187, 472
 interrelationships: of analyses, 5, 45, 70, 73, 255, 301, 472
 iodimetry: 164, 307, 315, 338, 391, 420, 425
 iodine: in soils, 406
 ion exchange resins: *see* Resins
 ionic activity: 24, 26, 27, 28–30, 35, 278
 ionic antagonism: *see* Antagonism
 ionic diffusion: 9, 24, 130
 iron (II): determination – exchangeable, 354, total, 336, 354; extraction, 337–8; occurrence, 332–4; waterlogged soils and, 332, 334, 442–4, 464
 iron (III): determination – available, 337, 354, exchangeable, 89, 337, 354, in solution, 338–40, 355–7, total, 335, 353; occurrence, 332–4; oxides, *see* Free oxides
 irrigation: 62, 461
 isohydic indicator method: for pH, 27
 isotopic dilution: 277, 281
 Jenkin mud sampler: 468
 Jones reductor: 256, 290, 366
 junction: liquid, 24; potentials, 24–5
 K-values: 131
 kaolinitic soils: 128, 334, 340, 363, 396, 473
 kilogramme: per hectare, as analytical unit, 481
 kinetics: of chemical equilibria, 462–6
 Kirk's still: 160
 Kjeldahl: method for nitrogen – apparatus, 157, 159–63, 197, catalysts, 151–2, procedure, 471
 'L' values: 281–2
 lacustrine deposits: 1, 61, 437
 lakes: bottom deposits, 305, 406, 437, 438, 442, 447, 453, 461, 462, 463, 468, 473
 La Motte–Hester: pH test, 28, 33
 lateritic soils: 34, 151, 274, 333, 334, 340, 348, 362
 lattice structure: clay mineral, 113, 127, 129, 136, 153, 154, 333, 341, 389
 leaching: apparatus, 99, 100; effects, 21, 35, 46, 61, 62, 88, 92–3, 113, 334, 348, 364, 387, 390, 424, 463
 lead: in soils, 407
 lepidocrocite: 333
 lignin: 219, 223
 lignoin: 393
 lime potential: determination, 34; theory, 28–30, 132, 277, 281
 lime requirement: 38–40; determination, 43
 limestone: 1, 39, 365, 381, 401, 406, 433
 limonite: 333
 liquid junction potential: *see* Junction potential
 lithium: glass electrode, *see* Electrodes, sodium; in soils, 408
 litter: plant, 209, 216, 334, 389
 loess: 1, 139, 433
 loss on ignition: 209–10, 342, 471
 macro-respirometer: *see* Respirometers
 maghemite: 333
 magnesite: 45
 magnetite: 333
 magnesium: carbonate – determination, 48–51, occurrence, 45; determination – available, 116, 124, exchangeable, 115, 124, 471, in solution, 117, 121–3, 125, recommended methods, 121–3, 124–6, total, 115, 124; occurrence, 89, 113; perchlorate, 46, 55, 243, 244; role in plant nutrition, 114; uranyl acetate, 141
 manganese: determination – active, 351, 360, available, 350, exchangeable, 14, 104, 350, 359, 471, in solution, 351, 360, total, 350, 353, 358; occurrence, 348, 437, 464; role in plant nutrition, 349
 mangrove swamp: 270, 284, 341, 343, 344, 396, 437, 463
 mannitol: 283, 385
 marble: 1
 Markham still: 160
 mass flow: 9, 115
 mean deviation: 5
 mercaptans: 302, 304, 463
 mercury: determination, 410–12; occurrence, 409
 methane: 158, 236, 463
 methane base: 353, 360
 methionine: 301, 304, 318, 327, 463
 methylene blue: 40, 96, 162, 315, 318, 385, 456
 mhos, 65, 481
 mica: 113, 127, 131, 332, 340, 408
 microbiological changes: 12, 17, 52, 62, 149, 150, 166, 180, 184, 185, 226, 238, 270, 280, 350, 469
 microburettes: 172
 microdiffusion: 170–2, 181, 388, 391, 407
 microgramme: 481
 micro-organisms: 12, 13, 14, 17, 20, 115, 149, 226, 231, 238, 270, 285, 379, 419, 463, 469
 micro-pipettes: 171
 milliequivalents: as analytical unit, 100, 481
 mineralization: of nitrogen, 12, 63, 185–195; of organic matter, 9, 13, 63, 226–242; of phosphorus, 269–71; of sulphur, 302
 minor elements: *see* Trace elements
 Mohr's titration: method for chloride, 72, 85
 moisture: contents for incubation, 188–9, 238; determination, 17; expression of, 7

- molybdenum: determination, 414–17; occurrence, 413; role in plant nutrition, 414, 425
 molybdenum blue: 112, 265, 289, 290–3, 365, 368, 425
 monosulphide: determination, 315, 324; occurrence, 301, 305
 montmorillonite: 106, 113, 128, 332, 341, 363, 389, 429
 Morgan's reagent: 113, 273, 308, 321, 397
 mud: 305, 308, 316, 437, 438, 442, 447, 453, 456, 463, 468, 469
 murexide: *see* Ammonium purpurate

 naphthol green: indicator, 109
 native fixed ammonium: *see* Ammonium, non-exchangeable
 negative absorption: 90
 nephelometry: 314, 387, 423
 Nernst equation: 439
 Nessler solution: 168, 170
 Nesslerization: 170, 183
 Neubauer technique: 132, 282
 nickel: for determination of exchange capacity, 96; in soils, 417
 ninhydrin: 223
 nitrate: dissimilation, *see* Denitrification; extraction, 180, 202, 471; measurement, 180–3, 199, 202; occurrence, 61, 62, 63, 149
 nitrification: in soil, 12, 62, 63, 185–8, 304, 379, 437; measurement, 197
 nitrite: destruction of, 168, 180; determination, 153, 178–80, 199, 201, 471; extraction, 178, 201, 471; occurrence, 149, 437
 nitrogen: determination – ammonium, 166–76, 200, 201, available, 183–95, 203, gaseous, 150, nitrate, 180, 202, nitrite, 178, 201, organic, 150, recommended methods, 197–203, total, 150–166, 197–200; mineralization, 185–195
 Nitroso-R-salt: 339, 392
 nucleic acids: 267

 occluded phosphate: *see* Phosphate, occluded
 ohms (mhos): 65, 481
 Olsen's method: for nitrite and nitrate, 153
 Olsen's reagent: for phosphorus, *see* Hydrogen carbonate
 optical density: 482
 organic matter: acidity of, 225, 238; amino-compounds in, 216, 233; carbohydates in, 216, 221; carbonyl groups in, 225; cation exchange of, 89, 225; decomposition – 89, 226–42, 436, 463, factors affecting, 63, 238, 463–5, measurement, 226–38, 250; determination – by dry combustion, 205, 243, by wet combustion, 206, 243, by weight loss, 204, 209, oxidisable, 211, 245; fractionation, 216–224, 246; lignin in, 216, 223
 osmotic pressure: 62, 70, 129, 140, 390, 439

 oxidation-reduction: definition, 436; discussion, 436, 462; potentials, measurement – colorimetric, 455, potentiometric, 438, 448, 458–60; relation to pH, 441
 oxine: *see* 8-Hydroxyquinoline
 oxygen: determination in soil solution, 448, 474; potential, 334, 445; uptake, measurement, 230–8, 253

 paddy soils: *see* Rice soils
 parent material: 1, 2, 127, 371
 partial pressure: of CO₂, *see* Carbon dioxide
 parts per million: as analytical unit, 481
 Parnas-Wagner still: 160, 164
 Parr bomb: 306
 Patton & Reeder's indicator: 110
 pC values: 70, 309
 peat: sampler, 468; soils, 1, 19, 20, 21, 34, 75, 89, 113, 151, 209, 217, 269, 392, 396, 417, 428, 463
 pentosans: 216
 pentoses: 216, 247
 percent base saturation: *see* Base saturation
 perchloric acid: dehydration of silica with, 364, 367, 369; digestion with, 110, 120, 364, 367, 369, 373, 376, 377, 378
 percolation: 191–195, 235
 permeability: 60, 62, 95, 138, 333
 pF values: 277, 278
 pH values: determination – colorimetric, 26, 32, electrometric, 22, 30, field, 23, 26, 28, 31, 33; discussion, 19, 35, 48, 98, 465–466; effect of CO₂ on, 21, 22, 465; effect of salts on, 21, 25, 28; meters 20, 23, 32, 458; relation to Eh, 441
 o-Phenanthroline: 213, 339, 347, 357
 phenoldisulphonic acid: 165, 167, 181
 phosphate: aluminium forms, 257, 258, 259–61, 273, 275, 276, 281, 284, 286, 297, calcium forms, 257, 258, 259–61, 281, 283, 286, 297; iron forms, 257, 258, 259–61, 281, 283, 286, 297; occluded forms, 259, 261, 276, 297; potentials, 277–82; reductant forms, 259, 261, 297, saloid-bound, 259, 274, 297
 phosphorus: available, 19, 269, 271–283, 299, 363, 437, 470; determination – available, biological, 282, chemical, 272, 472; in solution, colorimetric, 289–95, gravimetric, 288, volumetric, 288; inorganic, 13, 256; organic, 261, 267; recommended methods, 294–300; total, 255, 295; fixation, 20, 283–8, 342, 364; fixation capacity, 286–8, 300; fractionation, 257, 276, 296; mineralization, 269; transfer of from Al to Fe, 284, 342
 phospholipids: 267, 268–9, 297
 photophosphorylation: 390
 phytin: 268, 271, 298
 picrolinic acid: 112
 plant nutrients: 89, 106, 114, 129, 140, 149, 183, 204, 255, 271–83, 302, 335, 337, 341, 343, 349, 350, 363, 392, 395, 406, 414, 418, 419, 428, 429

- platinum electrodes: *see* Electrodes, platinum
- podsoils: 40, 45, 139, 219, 269, 332, 334, 340, 428
- polarization: of electrodes, 26, 315, 450
- polarographic analysis: 130, 140, 182, 226, 340, 353, 374, 388, 392, 402, 406, 415, 417, 430, 474
- polysaccharides: 220, 221
- polysulphides: 301, 305, 316
- polyuronides: 222
- polyvinyl alcohol: 110, 118, 125
- potassium: as amendment of saline-alkali soil, 130; determination – available, 131–6, 143, exchangeable, 13, 131, 143, 471, in solution, 136–8, recommended methods, 143–6, total, 130, 143; electrode, 142; fixation, 13, 127–9; occurrence, 127; potentials, 132–6, 144; role in plant nutrition, 129
- potential: asymmetric, 23; bacterial, 446; chemical, 277–8; electrode, 439; equilibrium, 279; half-wave, 130; junction, 24; lime, 28, 34, 38, 132, 279, 281; mediator, 456; oxidation-reduction, 225, 438, 439; oxygen, 445; phosphate, 277–81; potassium, 132–6, 144, 277–81; standard, 439, 448; water, 277
- potentiometric titration: 34, 40, 214, 225, 226, 310, 391, 439, 448
- pounds per acre: as analytical unit, 481
- precision: of analysis, 3–6, 63
- preparation: of soil samples, 10–12, 14–18, 452–454
- pressure membrane: 67–8, 81
- protective colloid: 312
- protein: 129, 149, 216, 223, 301, 302, 303, 304, 349, 392
- pS value: 70
- puddling: 188
- pyrites: 316, 418, 428
- pyrocatechol violet: 102
- pyrogallol: 469
- pyrosulphate: fusion, 375, 429
- Q/I relationship: 133–6, 144–6
- quartering: of soil samples, 14
- quartz: 189, 334, 343, 362
- quinalizarin: 383, 385
- quinhydrone electrode: *see* Electrodes, quinhydrone
- quinolinium phosphate: 288
- radioactive elements: 90, 96, 97, 108, 150, 174, 185, 197, 237, 256, 274, 277, 281, 286, 287, 303, 308, 342, 350, 351, 372, 389, 418, 424, 429
- Raney nickel: 317
- ratio: C/N, 186, 209, 242, 463; law, 278; silica-alumina, 362, 367; silica-sesquioxide, 362, 363, 367, 369
- reception: of soil samples, 10
- recorders: 31
- recording: of results, 6
- redox indicators: 455–8
- redox levels: 447
- redox potentials: *see* Oxidation-reduction potentials
- reductant-soluble phosphorus: 259, 261, 297
- reduction: in analysis, 153, 181, 214, 289–293, 307, 314, 323, 336–7, 365, 405, 415, 425, 427; processes, 28, 308, 334, 436, 438, 442, 462–6
- reference electrodes: *see* Electrodes
- regression analysis: 4, 210, 277
- relative mean deviation: 5
- reporting: of results, 6
- resin: exchange, 71, 98, 109, 218, 219, 223, 224, 265, 276, 281, 299, 311, 318, 327–8, 345, 408
- resistance: electrical, 63, 64, 65, 76, 78
- respiration: in soils, 226–42, 436
- respiratory quotient: 230, 237, 250, 253
- respirometers: 195, 230–7, 250, 270, 476
- rH values: 441
- rhizosphere: 444
- rhodizonate titration: 310, 378
- rice soils: 284, 338, 342, 364, 396, 444, 461, 466, 470
- rifle sampler: 15
- rocks: igneous, 1, 139, 332, 348, 362, 365; metamorphic, 1; primary, 1, 348; secondary, 1; weathering of, 1, 139, 348, 371
- RQ-values: *see* Respiratory quotient
- rubidium: in soils, 389, 418
- S-values: 100, 481
- salicylic acid: in Kjeldahl digestion, 153, 199
- saline-alkali soils: 61, 74, 95, 138, 462
- saline soils: characteristics of, 61, 94, 129, 384; occurrence, 61, 138, 462
- salinity classes: 62, 70, 83
- saloid-bound phosphorus: 259, 274, 297
- salt-bridge: 24, 25, 73, 138, 449, 459
- salt retention: 92–4
- salts: soluble, determination – by conductivity, 65–70, 81–3, by paste resistance, 63, 76–80, gravimetrically, 71, individual, 72–4, 85–7, in soil solution, 67, 81, recommended methods, 75–87, 471, total, 63–71, 75–84; effect of upon organic matter decomposition, 63, 239; effect of upon pH, 21, 25, 28; effect of upon plant growth, 62
- sampling: soils, 467–9
- sand: 1
- sandstone: 1
- saturated paste: 21, 30, 63, 64, 75
- saturation extract: 65, 81, 95
- saturation percentage: 67, 75
- Schollenberger's method: for organic carbon, 211, 212, 213
- Schweitzer's solution: 222, 248
- sea-water: 138, 305, 461, 462
- selenium: as catalyst, 151; determination, 419–23; in phosphorus extraction, 258; occurrence, 418; role in plant nutrition, 418–19
- self-diffusion coefficients: 418, 424
- sesquioxides: 111, 139, 151, 217, 268, 284, 285, 362, 366, 369
- siderite: 332, 464
- siemens: as analytical unit, 65, 481

- sieving: 14, 47, 204, 205, 218, 374, 470, 482
- silica: amorphous, 363; cation exchange and, 363; dehydration, 364, 367, 369; determination – total, 364, 367, water-soluble, 276, 364, 368; occurrence, 362, 465
- silica-alumina ratios: *see* Ratios
- silica-sesquioxide ratios: *see* Ratios
- silicon: determination, 364, 367, 368; occurrence, 61, 362; role in plant nutrition, 363
- silver: in soils, 423
- sodium: determination – exchangeable, 140, 146, in solution, 141, 147, recommended methods, 146–8, total, 140, 146; exchangeable, 21, 45, 51, 89, 139, 140, 146; exchangeable percentage, 51, 139, 147, 481; effect upon plant growth, 140; electrode, *see* Electrodes; fluoride, 37, 41; nitroresol, reagent for cobalt, 392, 393; occurrence, 138
- soil: definition of, 1; formation of, 1–2; parent material, 1; solution, 26, 65, 67, 81, 95, 258, 279, 446, 455, 458, 470
- Solochrome Dark Blue: *see* Calcon
- solonetz soils: 113, 362
- Solu bridge: 82
- soluble salts: *see* Salts
- solubility product: 257, 285, 342, 443
- spark emission spectrophotometry: 130, 373
- spectrographic analysis: for exchangeable cations, 98, 105, 108, 117, 120, 125, 136, 141, 143, 146, 428; for total analysis, 130, 140, 373, 374, 380, 385, 389, 392, 407, 415, 417, 419, 424, 425, 428, 429
- γ -spectrophotometric analysis: 131, 390
- standard deviation: 5, 391
- standard solutions: 477–9, 480
- standard redox potentials: 439
- stannous chloride: *see* Tin (II)chloride
- starch: 114, 199
- statistical techniques: 5–6
- steam distillation: 169, 199, 200, 201, 202, 316
- storage: of soil samples, effect upon soil properties, 16–17, 116, 166, 186–8, 468, 469; methods, 16
- strontium: in soils, 107, 423
- sugars: 114
- sulphamic acid: 168, 293
- sulphate: in acid soils, 62, 302, 304, 437; in arid soils, 60, 301; adsorption, 302, 308; determination – colorimetric, 311, conductimetric, 311, gravimetric, 309, 322, nephelometric, 314, turbidimetric, 311–314, 322, volumetric, 310; extraction, 13, 308, 321; reduction, 314, 323, 440; water-soluble, 303, 309
- sulphide: accumulation, 301, 305, 462; determination, 315, 320, 324–6; occurrence, 301, 437; oxidation, 305; toxicity, 304, 462
- sulphur: active-oxidizable, 303; available, 301, 302, 324; cycle, 302; deficiency, 301, 302, 303, 305; determination – elemental, 316, 326, mono-sulphidic, 315, 324, organic, 317, 326, polysulphidic, 316, polythionate, 317, recommended methods, 319–31, sulphate, 13, 307, 321, thiosulphate, 317, total, 305, 319; easily-oxidizable, 303; effect on alkali soils, 304; effect on nitrification, 304; elemental, 52, 301, 316, 326; mineralizable, 302; occurrence, 62, 301; requirement, 51, 59, 304; role in plant nutrition, 303
- Superchrome Black TS: 110
- swamp soils: analysis, 471–2; chemistry, 270, 335, 341, 343, 462–6; sampling, 467–9, 470–1
- T-values: 101, 481
- tannin: 219, 365;
- temperature: effects
upon Kjeldahl digestions, 151
- tetraphenylboron: 136, 137
- thallium: in soils, 424
- thermal decomposition: of dichromate, 211–12
- thermobalance: 51, 210
- Thiazole Yellow: 119
- Thiobacillus thiooxidans*: 20
- thioglycollic acid: 338, 345, 415
- thiosulphate: 301, 304, 317
- thiourea: 304, 415
- thorium nitrate: 401, 403
- time: effect of upon Kjeldahl digestion, 151
- tin: in soils, 425
- tin(II)chloride: reductant, 289–92, 294, 415
- Tiron: 339, 356, 366
- Titan Yellow: 118–19
- titanium: determination, 365, 369; occurrence, 362, 365
- titanium (III) sulphate: reductant, 181
- titrateable acidity: *see* Acidity, titrateable
- Tiurin: method for total carbon, 213; method for total nitrogen, 165
- toluene: as bacterial inhibitor, 166, 180, 453, 468
- toluene-3,4-dithiol: *see* Dithizone
- total analysis: acid digestion, 373, 376, 377, 378; for nutrients – available, 372, trace, 372, 374; fusion, 373, 375, 377; methods, 373, 377; purpose, 372; reporting results, 374; spectrography and, 373
- total exchangeable bases: 481
- total salts: *see* Salts
- toxicity: in soils, 20, 129, 140, 285, 304, 335, 341–2, 372, 378, 379, 381, 384, 387, 389, 391, 395–6, 407, 408, 410, 414, 417, 418, 423, 424, 425, 427, 462
- trace elements: 16, 19, 46, 304, 335, 371, 414
- trace nutrient elements: definition, 372; determination, 374
- triethanolamine: buffer solution, 37, 43, 92, 102, 105
- tungsten: in soils, 425
- turbidimetric analysis: 136, 311, 317, 382, 402
- ultra-violet: irradiation, 337, 355, 371, 383; spectrum range, 311, 392, 428, 481
- units: for analytical results, 481; SI, xxv, 481

- uranium: in soils, 427
 uranyl magnesium acetate: 141–142
 uranyl zinc acetate: 141–2
 uronic acids: 221, 222, 248
- vanadium: in soil, 427
 vanadomolybdate: reagent for phosphorus, 293, 295
 vermiculite: 91, 113, 128, 141, 173, 190, 332, 389
 violuric acid: 142
 volcanic: ash, 1; gas, 387; soils, 1, 14, 275, 301, 334, 401
 volume basis: for soil analysis, 470, 472
 Volhard method: for chloride, 72
- Walkley–Black method: for organic carbon, 213, 245, 471
 Warburg apparatus: 197, 230, 231
 washing: for cation exchange, 92–4
 water: in soil – available, 129, 140, determination, 17, extracts, 68
 waterlogged soil: analysis, 28, 154, 212, 469–73; chemistry of, 149, 178, 186, 188, 238, 272, 301, 304, 333, 348, 462–6; classification, 466; interpretation of analyses, 472; investigation, 461, 473; oxygen in, 463; reduction in, 28, 436, 444, 462–6; sampling, 467–9; types of, 461
 water-soluble salts: *see* Salts
 wavelength: 481
 weathering: of rocks, 1, 45, 113, 127, 371; of soils, 89, 127, 332, 362, 363, 418, 433
 wet oxidation: of organic matter, 206–9, 211, 243
 Wheatstone bridge: 64, 65, 76, 97
 white alkali soil: 139
- X-ray: analysis, 16, 48, 50, 88, 226, 287, 316, 337, 340, 342, 433
- yttrium: in soils, 428
- zinc: determination – available, 429, in solution, 430, total, 429; occurrence, 428; role in plant nutrition, 428, 429
 zincon: 430
 zirconium: determination, 433–435; occurrence, 433

