Unit d Stat s
Department of
Agriculture

Soil
Cons rvation
Service

Soil Survey
Investigations
Report No. 1

Procedures for Collecting Soil Samples and Methods of Analysis for Soil Survey

Trade names are used in this publication solely for the purpose of providing specific information. The mention of a trade name does not constitute a guarantee of the product by the U.S. Department of Agriculture nor does it imply an endorsement by the Department over other products not mentioned.

PREFACE

This is the second revision of the first volume of a report series to make available technical information from cooperative field and laboratory investigations of soils of the United States, Puerto Rico, and the Virgin Islands. Thirty-six volumes of soil data and descriptions have been published in this continuing series. Soil sampling

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INTRODUCTION

The methods and procedures described in this publication are those in current use in the National Soil Survey Laboratory (NSSL) of the Soil Conservation Service. Also included are methods established subsequent to the 1972 revision but later dropped

Suggestions for Using SSIR No. 1

SSIR No. 1 has eight sections identified according to

Most of the methods are described in enough detail that they can be reproduced in many laboratories without reference to other sources. For some methods, however, an investigator may need or want to read more about the theory or technique of a method. Therefore, references to the literature are included.

Headings for columns of laboratory data include method codes of up to four characters (e.g., 6A1c) that refer to a method described in this volume. Method codes and the text are organized into a standard four-level outline. Methods are identified on a code sheet (fig. 1) included in each volume of the Soil Survey Investigation Report series. The code sheet will

characterization of soils.

1. Sample collection and preparation.
2. Conventions (for presenting data).
3. Particle-size analyses.
4. Fabric-related analyses (bulk density, water retention, plasticity, micromorphology, and coefficient of linear extensibility [COLE]).
5. Ion-exchange analyses (cation exchange capacity, extractable bases, base saturation, exchangeable sodium percentage [ESP], sodium adsorption ratio [SAR]).
6. Chemical analyses.

SI Units—Possible Changes in NSSL Data Sheet

The scientific community has urged that the International System of Units (SI units) be adopted for all scientific notation. For the NSSL data sheet to conform to SI notation, the following are examples of changes that are probable and are included here for information:

Soil physics	Present notation	× Factor	SI units	Comment
Water suction	bars	× 100 =	kPa	kilopascals—potential energy of soil water
Bulk density	g/cm^3	× 1 =	Mg/m^3	megagrams per cubic meter
Soil chemistry				
Extractable bases	meq/100 g	× 1 =	cmol (p+)/kg	centimoles of positive charge per kilogram
Cation exchange capacity	meq/100 g	× 1 =	cmol (NH_4+)/kg	centimoles of ammonium ion charge per kilogram (specifies ammonium ion as the replacing cation)
Soluble cations	meq/liter	× 1 =	mmol (p+)/L	millimoles of positive charge per liter
Soluble anions	meq/liter	× 1 =	mmol (e–)/L	millimoles of negative charge per liter
Electrical conductivity	mmhos/cm	× 1 =	dS/m	decisiemens per meter

Figure 1. Code Sheet for Laboratory Methods

1. SAMPLE COLLECTION AND PREPARATION
   - A. Field sampling
     1. Site selection
     2. Pedon sampling
        - a. Soils with rock fragments
        - b. Marsh and swamp soils
        - c. Hydraulic probe
   - B. Laboratory preparation
     1. Standard (air-dry)
        - a. Square-hole 2-mm sieve
        - b. Round-hole 2-mm sieve*
     2. Field moist
     3. Carbonate-containing material
     4. Carbonate-indurated material*
     5. Rock fragments (>2 mm)
        - a. 2-20 mm
        - b. <20 mm
     6. Whole soil
2. CONVENTIONS
   - A. Size-fraction base for reporting
     1. <2 mm
     2. >2 mm, specified size
   - B. Data sheet symbols
3. PARTICLE-SIZE ANALYSES
   - A. Particles <2 mm (pipet method)
     1. Air-dry samples
        - a. Carbonate and noncarbonate clay I
        - b. Fine clay (<0.2μ)
        - c. Water-dispersible clay
        - d. Carbonate and noncarbonate clay II
     2. Moist samples
        - a. Carbonate and noncarbonate clay I
        - b. Fine clay (<0.2μ)
        - c. Water-dispersible clay
        - d. Carbonate and noncarbonate clay II
   - B. Particles >2 mm
     1. Weight estimates
        - a. By field and laboratory weighing
        - b. From volume and weight estimates
     2. Volume estimates
4. FABRIC-RELATED ANALYSES
   - A. Bulk density
     1. Saran-coated clods
        - a. Field state (Db_f)
        - b. Air-dry*
        - c. 30-cm absorption*
        - d. 1/3-bar desorption I ($Db_{1/3}$)
        - e. 1/3-bar desorption II*
        - f. 1/3-bar desorption III*
        - g. 1/10-bar desorption*

** Denotes methods not currently in use by NSSL, and not included in this publication.*

h. Ovendry (Db_d)
i. Rewet (Db_r)

2. Paraffin-coated clods*
   a. Ovendry*
3. Cores
   a. Field moist
4. Nonpolar-liquid-saturated clods*

B. Water retention

1. Pressure-plate extraction
   a. Air-dry <2 mm (0.06, 1/10, or 2 bars)
   b. Soil pieces*
   c. Natural clods (0.06, 1/10, 1/3, or 1 bar)
   d. Cores
   e. Rewet
2. Pressure-membrane extraction
   a. Air-dry <2 mm (15 bars)
   b. Field-moist <2 mm (15 bars)
3. Sand-table absorption*
4. Field state
5. Air-dry

C. Water-retention difference (WRD)

1. Between 1/3 and 15 bars
2. Between 1/10 and 15 bars
3. Between 1/3 bar rewet and 15 bars (air-dry)

b. Effective cation exchange capacity (ECEC)
4. KOAc, pH 7.0*
5. $BaCl_2$, pH 8.2*
   a. Barium by flame photometry*
6. NH_4OAc, pH 7.0, leaching tube* (CEC-7)
   a. Direct distillation*
7. NH_4Cl
   a. Direct distillation
8. NH_4OAc, pH 7.0, automatic extractor (CEC-7)
   a. Direct distillation
   b. Steam distillation
9. NH_4Cl, automatic extractor
   a. Direct distillation
   b. Steam distillation

B. Extractable bases

1. NH_4OAc, pH 7.0, Büchner funnel*
   a. Uncorrected*
   b. Corrected (exchangeable)*
2. KCl-TEA extraction, pH 8.2*
3. KCl-TEA, pH 8.2 (revised)*
   a. Uncorrected*
   b. Corrected (exchangeable)*
4. NH_4OAc, pH 7.0, leaching tube*
   a. Uncorrected*
   b. Corrected (exchangeable)*

D. Coefficient of linear extensibility (COLE)

1. Air-dry or ovendry to 1/3 bar

E. Micromorphology

1. Thin sections
   a. Preparation
   b. Interpretation
   c. Moved-clay percentage*
2. Scanning electron microscopy*

F. Plasticity index

1. Liquid limit
2. Plastic limit

5. ION-EXCHANGE ANALYSES

A. Cation-exchange capacity
1. NH_4OAc, pH 7.0, Büchner funnel* (CEC-7)
   a. Direct distillation*
   b. Displacement, distillation*

5. NH_4OAc, pH 7.0, automatic extractor
   a. Uncorrected (extractable)
   b. Corrected (exchangeable)

C. Base saturation

1. NH_4OAc, pH 7.0
2. NaOAc, pH 8.2*
3. Sum of cations, TEA, pH 8.2

D. Exchangeable Sodium Percentage (ESP)

1. NaOAc, pH 8.2*
2. NH_4OAc, pH 7.0

E. Sodium-adsorption ratio

F. Calcium saturation*

1. NH_4OAc, pH 7.0*

G. Aluminum saturation

1. Bases plus aluminum

a. $FeSO_4$ titration*
b. CO_2 evolution, gravimetric
c. $FeSO_4$ titration, automatic titrator

2. Dry combustion
   a. CO_2 evolution I*
   b. CO_2 evolution II*
   c. CO_2 evolution III
3. Peroxide digestion*
   a. Weight loss*
4. Sodium pyrophosphate extraction
   a. CO_2 evolution, gravimetric

B. Nitrogen

1. Kjeldahl digestion I
   a. Ammonia distillation*
   b. Ammonia distillation, automatic titrator
2. Semimicro Kjeldahl*
   a. Ammonia distillation*
3. Kjeldahl digestion II
   a. Ammonia steam distillation, automatic titrator

C. Iron

1. Dithionite extraction*
   a. Dichromate titration*
   b. EDTA titration*
2. Dithionite-citrate extraction
   a. Orthophenanthroline colorimetry*
   b. Atomic absorption
3. Dithionite-citrate-bicarbonate extraction*
   a. Potassium thiocyanate colorimetry*
4. Pyrophosphate-dithionite extraction*
5. Sodium pyrophosphate extraction I*
   a. Atomic absorption*
6. Ammonium oxalate extraction*
   a. Atomic absorption*
7. HF dissolution
   a. Atomic absorption
   c. Weight loss*
   d. Weight gain*
   e. Titrimetric*
   f. Warburg method*
   g. Manometer, electronic
2. Sensitive qualitative method*
   a. Visual, gas bubbles*
3. H_2SO_4 treatment*
   a. Weight gain*
4. <20-mm basis

F. Gypsum

1. Water extract
   a. Precipitation in acetone
   b. Indirect estimate
   c. Chromatograph
2. Weight loss
3. Correction for crystal water
4. <20-mm basis
5. Gypsum requirement

G. Aluminum

1. KCl extraction I, 30 min*
   a. Aluminon I*
   b. Aluminon II*
   c. Aluminon III*
   d. Fluoride titration*
   e. Atomic absorption*
2. KCl extraction II, overnight*
   a. Aluminon I*
3. NH_4OAc extraction*
   a. Aluminon III*
4. NaOAc extraction*
   a. Aluminon III*
5. Sodium pyrophosphate extraction I*
   a. Atomic absorption*
6. Ammonium oxalate extraction*
   a. Atomic absorption*
7. Dithionite-citrate extraction
   a. Atomic absorption

2. $BaCl_2$-triethanolamine II*
   a. Back-titration with HCl*
3. KCl-triethanolamine*
   a. Back-titration with NaOH*
4. $BaCl_2$-triethanolamine III
   a. Back-titration with HCl, automatic titrator
5. $BaCl_2$-triethanolamine IV, automatic extractor
   a. Back-titration with HCl, automatic titrator

I. Carbonate

1. Saturation extract
   a. Acid titration*
   b. Acid titration, automatic titrator

J. Bicarbonate

1. Saturation extract
   a. Acid titration*
   b. Acid titration, automatic titrator

K. Chloride

1. Saturation extract
   a. Mohr titration*
   b. Potentiometric titration*
   c. Ion chromatograph

L. Sulfate

1. Saturation extract
   a. Gravimetric, $BaSO_4$*
   b. EDTA titration*
   c. Ion chromatograph
2. NH_4OAc extraction*
   a. Gravimetric, $BaSO_4$*

M. Nitrate

1. Saturation extract
   a. Phenoldisulfonic (PDS) acid colorimetry*
   b. Diphenylamine*
   c. Ion chromatograph

N. Calcium

1. Saturation extract
   a. EDTA titration*
   b. Atomic absorption
2. NH_4OAc extraction
   a. EDTA-alcohol separation*
   b. Oxalate-permanganate I*
   c. Oxalate-permanganate II* Fe, Al, and Mn removed*
   d. Oxalate-cerate*
   e. Atomic absorption
3. NH_4Cl-EtOH extraction*
   a. EDTA titration*
4. KCl-TEA extraction*
   a. Oxalate-permanganate*
   b. EDTA titration*
   c. Atomic absorption*
5. HF dissolution
   a. Atomic absorption

O. Magnesium

1. Saturation extract
   a. EDTA titration*
   b. Atomic absorption
2. NH_4OAc extraction
   a. EDTA-alcohol separation*
   b. Phosphate titration*
   c. Gravimetric, $Mg_2P_2O_7$*
   d. Atomic absorption
3. NH_4Cl-EtOH extraction*
   a. EDTA titration*
4. KCl-TEA extraction*
   a. Phosphate titration*
   b. EDTA titration*
   c. Atomic absorption*
5. HF dissolution
   a. Atomic absorption

P. Sodium

1. Saturation extract
   a. Flame photometry*
   b. Atomic absorption
2. NH_4OAc extraction
   a. Flame photometry*
   b. Atomic absorption
3. HF dissolution
   a. Atomic absorption

Q. Potassium

1. Saturation extract
   a. Flame photometry*
   b. Atomic absorption
2. NH_4OAc extraction
   a. Flame photometry*
   b. Atomic absorption
3. HF dissolution
   a. Atomic absorption

R. Sulfur

1. $NaHCO_3$ extract, pH 8.5*
   a. Methylene blue*
2. HCl release (sulfide)*
   a. Iodine titration*
3. SO_2 evolution
   a. KIO_3 titration

S. Phosphorus

1. Perchloric acid digestion*
   a. Molybdovanadophosphoric acid colorimetry*
2. Adsorption coefficient

T. Boron

1. Saturation extract
   a. Carmine colorimetry

U. Fluoride

1. Saturation extract
   a. Ion chromatograph

V. Silicon

1. HF dissolution
   a. Atomic absorption

7. MINERALOGY

A. Instrumental analysis

1. Preparation*
   a. Carbonate removal*
   b. Organic-matter removal*
   c. Iron removal*
   d. Particle-size fractionation*
   e. PSDA pretreatment*
2. X-ray diffraction
   a. Thin film on glass, solution pretreatment*
   b. Thin film on glass, resin pretreatment I*
   c. Thin film on glass, $NaPO_3$ pretreatment I*
   d. Thin film on tile, solution pretreatment*
   e. Thin film on tile, resin pretreatment*
   f. Thin film on tile, $NaPO_3$ pretreatment*
   g. Powder mount, diffractometer recording*
   h. Powder mount, camera recording*
   i. Thin film on glass, resin pretreatment II
   j. Thin film on glass, $NaPO_3$ pretreatment II
   k. Powder mounts
3. Differential thermal analysis
4. Thermal gravimetric analysis
5. Infrared analysis

B. Optical analysis

1. Grain studies
   a. Grain mounts, epoxy
   b. Grain mounts, Canada balsam
2. Electron microscopy*

C. Total analysis

1. Na_2CO_3 fusion*
2. X-ray emission spectrography*
3. HF dissolution

D. Surface area

1. Gylcerol retention*
2. Ethylene glycol monoethyl ether (EGME) retention

8. MISCELLANEOUS

A. Saturated paste, mixed

1. Saturation extract*
   a. Conductivity*
   b. Conductivity, quick test*
2. Bureau of Soils cup, conductivity*
3. Saturation extract, automatic extractor
   a. Conductivity, digital bridge

B. Saturated paste, capillary rise*

1. Saturation extract*
   a. Conductivity*

C. Reaction (pH)

1. Soil suspensions
   a. Water dilution*
   b. Saturated paste
   c. KCl*
   d. NaF
   e. $CaCl_2$*
   f. Water dilution and $CaCl_2$, automated system
   g. KCl, automated system
2. Organic materials
   a. $CaCl_2$

D. Ratios and estimates

   1. To total clay
   2. To noncarbonate clay
   3. Ca to Mg (extractable)
   4. Estimated clay percentage
   5. Estimated total salt
   6. Sodium pyrophosphate iron and aluminum (spodic horizon)
   7. Index of accumulation (spodic horizon)

E. Soil resistivity

   1. Saturated paste

F. Mineral content

   1. Loss on ignition (400° C)

G. Fiber volume

   1. Water dispersed

H. Pyrophosphate color

I. Salt prediction

ECHAW

SAMPLED AS: SANDY, SILICEOUS, THERMIC ENTIC HAPLOHUMOD

S 80SC-051 -003 SAMPLE NOS. 80P2737 - 2746 DATE 11/08/82

COASTAL PLAIN STUDY (NC-SC) USGS

GENERAL METHODS 1B1A, 2A1, 2B

U. S. DEPARTMENT OF AGRICULTURE
SOIL CONSERVATION SERVICE
NATIONAL SOIL SURVEY LABORATORY
LINCOLN, NEBRASKA

-1- -2- -3- -4- -5- -6- -7- -8- -9- -10- -11- -12- -13- -14- -15- -16- -17- -18- -19- -20-

SAMPLE NO.	HZN NO.	DEPTH (CM)	HORIZON	TOTAL CLAY LT .002	TOTAL SILT .002 -.05	TOTAL SAND .05 -2	CLAY FINE LT .0002	CLAY CO3 LT .002	SILT FINE .002 -.02	SILT COARSE .02 -.05	SAND VF .05 -.10	SAND F .10 -.25	SAND M .25 -.50	SAND C .5 -1	SAND VC 1 -2	COARSE FRACTIONS (MM) WEIGHT 2 -5	5 -20	20 -75	>2MM .1- 75	WT PCT OF WHOLE SOIL
				<- PCT OF <2MM (3A1) ->												<- PCT OF <75MM (3B1) ->				
802737	1S	0- 23	AP	2.4	3.6	94.0			0.8	2.8	1.1	68.5	23.2	1.2	TR	--	--	--	93	--
802738	2S	23- 37	A21	2.4	6.3	91.3			2.8	3.5	0.9	65.6	23.6	1.2	--	--	--	--	90	--
802739	3S	37- 55	A22	4.8	5.2	90.0			2.0	3.2	1.2	66.5	21.1	1.2	TR	TR	--	--	89	--
802740	4S	55- 73	B21T	12.5	4.4	83.1			1.2	3.2	1.2	61.1	19.8	1.0	--	TR	TR	--	82	TR
802741	5S	73- 91	B22T	8.5	3.1	88.4			--	3.1	1.1	64.4	21.8	1.0	0.1	TR	1	--	87	1
802742	6S	91-105	A'2	1.6	2.4	96.0			--	2.4	0.6	69.8	24.7	0.9	--	--	--	--	95	--
802743	7S	105-126	BH1	--	2.4	97.6			--	2.4	0.4	67.7	27.9	1.6	TR	--	--	--	97	--
802744	8S	126-146	BH21	--	2.1	97.9			--	2.1	0.5	72.2	24.1	1.1	TR	--	--	--	97	--
802745	9S	146-169	BH22	0.8	1.4	97.8			0.8	0.6	0.4	70.5	26.3	0.6	TR	--	--	--	97	--
802746	10S	169-190	BH3	--	1.3	98.7			--	1.3	0.4	76.6	19.2	2.5	--	--	--	--	98	--

SAMPLE NO.	HZN NO.	ORGN C 6A1C	TOTAL N 6B1B	EXTR P 6S1A	TOTAL S 6R3A	EXTRACTABLE FE 6C2B	EXTRACTABLE AL 6G7A	EXTRACTABLE MN 6D2A	CEC 8D1	15 BAR 8D1	ATTERBERG LL 4F1	ATTERBERG PI 4F	BULK DENSITY FIELD MOIST 4A3A	BULK DENSITY 1/3 BAR 4A1D	BULK DENSITY OVEN DRY 4A1H	COLE WHOLE SOIL 4D1	WATER CONTENT FIELD MOIST 4B4	WATER CONTENT 1/10 BAR 4B1C	WATER CONTENT 1/3 BAR 4B1C	15 BAR 4B2	WRD WHOLE SOIL 4C1
		<- PCT			OF <2MM						PCT <0.4MM		<- G/CC ->			CM/CM	<- PCT OF <2MM ->				CM/CM
802737	1	0.73	0.045			0.1	0.1	--	0.92	0.58				1.60						1.4	
802738	2	0.23	0.018			0.2	0.1	--	0.46	0.58				1.63	1.64	0.002			4.7	1.4	0.05
802739	3	0.17	0.014			0.2	0.1	--	0.23	0.33				1.55	1.55	--			5.6	1.6	0.06
802740	4	0.24	0.024			0.7	0.3	--	0.19	0.37		NP		1.57	1.57	--			7.4	4.6	0.04
802741	5	0.18	0.018			0.6	0.2	--	0.18	0.39				1.60						3.3	
802742	6	0.36	0.020			0.3	0.2	--	0.81	0.81				1.68	1.68	--			3.8	1.3	0.04
802743	7	0.34	0.011			0.1	0.2	--						1.70						0.8	
802744	8	0.68	0.016			0.1	0.3	--						1.70	1.75	0.010			4.3	1.2	0.05
802745	9	0.68	0.022			TR	0.2	--	2.38	1.38				1.67	1.67	--			3.7	1.1	0.04
802746	10	0.39	0.008			--	0.1	--						1.67	1.67	--			4.0	0.7	0.06

SAMPLE NO.	HZN NO.	NH4OAC EXTRACTABLE BASES CA 6N1A	MG 6N1A	NA 6P1A	K 6Q1A	SUM	ACIDITY 6H1A	EXTR AL 6G2A	CEC SUM	CEC NH4	BASES	AL SAT	BASE SUM	SAT NH4	CO3 AS CACO3	RES. OHMS	PH CACL2	PH H2O
		<- MEQ / 100 G ->																
802737	1	0.3	0.1	--	0.1	0.5	1.4	0.5	1.9	2.2	1.0	50	26	23			4.1	4.4
802738	2	0.1	TR	TR	TR	0.1	1.1	0.5	1.2	1.1	0.6	83	8	9			4.3	4.6
802739	3	0.1	TR	--	TR	0.1	1.3	0.6	1.4	1.1	0.7	86	7	9			4.2	4.6
802740	4	TR	0.1	--	0.1	0.2	3.5	1.3	3.7	2.4	1.5	87	5	8			4.1	4.6
802741	5	--	TR	--	TR	TR	1.9	0.9	1.9	1.5	0.9	100		1			4.3	4.7
802742	6	TR	TR	--	--	TR	3.5	0.4	3.5	1.3	0.4	100		2		6700	4.8	5.2
802743	7	0.1	TR	--	--	0.1	2.7	0.3	2.8	1.0	0.4	75		10			5.0	5.4
802744	8	TR	TR	--	--	TR	5.9	0.4	5.9	2.0	0.4	100		1			4.9	5.4
802745	9	--	--	--	--	--	5.5	0.4	5.5	1.9	0.4	100		1			4.9	5.3
802746	10	TR	TR	--	--	TR	3.3	0.3	3.3	1.0	0.3	100		2			4.9	5.3

SAMPLE NO.	HZN NO.	C 6A4A	FE 6C5A	AL 6G5A	FE+AL DI-CI PCT FE+AL	FE+AL PCT CLAY	AL+C/BI PCT CLAY	OP ACCUM	K2O 6Q3A	Fe 6C7A	X-RAY 7A2B	X-RAY 7A2B	X-RAY 7A2B	X-RAY 7A2B	DTA 7A3	DTA 7A3	TOTAL DOM RES 7B1A	TOTAL DOM WEATH 7B1A
		<- PCT OF <2MM->							<- PCT ->		<- RELATIVE AMOUNTS ->				<- PCT ->			
802737	1	2.3	0.1	0.1	1.0	0.1	1.0	16	0.5	2.5								
802738	2	2.9	0.2	0.3	1.7	0.2	1.3											
802739	3	0.1	0.2	0.3	1.7	0.1	0.1		0.4	4.6					KK11	GI 6		
802740	4	0.1	0.4	0.4	0.8	0.1	TR											
802741	5	0.1	0.4	0.5	1.1	0.1	0.1		0.4	6.6					KK14	GI14		
802742	6	0.3	0.3	0.3	1.2	0.4	0.4	38	0.2	4.3					GI 2			
802743	7	1.7	0.2	0.2	1.3													
802744	8	0.7	0.1	0.3	1.0				---	---								
802745	9	0.4	--	0.3	1.5	0.4	0.9	117										
802746	10	0.3	--	0.2	2.0				0.4	---								

Series: Echaw Taxadjunct Date: 8/81
Pedon No: S80SC-051-003
Taxonomy: Sandy, siliceous, thermic Entic Haplohumod.
Latitude: N33 Deg. 48 Min. Longitude: W079 Deg. 06 Min.
MLRA: 153 A Atlantic Coast Flatwoods
Physiography: Barrier bar in coastal plain
Geomorphic Position: On the crest interfluve summit
Slope and Aspect: 1 pct plane Elevation:
Microrelief: none
Air Temp. 17C Summer: 26C Winter: 8C
Precipitation: 137 cm Udic moisture regime.
Water Table: Not observed
Drainage: Moderately well drained Permeability:
Stoniness:
Land Use: Cropland
Erosion or Deposition:
Parent Material: moderately weathered, marine from unspecified material
Described by: D. C. Hallbick and R. T. Eppinette
Sampled by M. J. Mausbach. Pedon is a taxadjunct because of clay increase in Bt.

AP 0 — 23 cm. Dark gray (10YR 4/1) loamy sand; single grain structure; common fine roots; pH = 5.6, medium acid; clear smooth boundary.
80P2737

A21 23 — 37 cm. Light yellowish brown (10YR 6/4) fine sand; a few fine distinct very pale brown (10YR 7/3) mottles; single grain structure; common fine roots; pH = 5.6, medium acid; clear smooth boundary.
80P2738

A22 37 — 55 cm. Very pale brown (10YR 7/4) loamy sand; common fine faint brownish yellow (10YR 6/6) mottles; single grain structure; a few fine roots; pH = 5.6, medium acid; clear smooth boundary.
80P2739

B21t 55 — 73 cm. Light yellowish brown (10YR 6/4) loamy sand; common medium faint brownish yellow (10YR 6/6) and a few fine distinct yellowish brown (10YR 5/8) mottles; massive parting to weak coarse subangular blocky structure; a few fine roots; pH = 5.2, strongly acid; clear smooth boundary
80P2740

B22t 73 — 91 cm. Light yellowish brown (10YR 6/4) loamy sand; common medium faint brownish yellow (10YR 6/6) and a few fine distinct yellowish brown (10YR 5/8) mottles; massive parting to weak coarse subangular blocky structure; a few fine roots; pH = 5.2, strongly acid; clear smooth boundary.
80P2741

A'2 91 — 105 cm. Brown to dark brown (10YR 4/3) sand; single grain structure; common fine roots; silica concretions; clear smooth boundary.
80P2742

B1h 105 — 126 cm. Dark brown (7.5YR 3/4) sand; common medium distinct dark grayish brown (10YR 4/2) mottles; single grain structure; a few fine roots; fine ironstone nodules; pH = 5.8, medium acid; clear wavy boundary.
80P2743

B2h 126 — 169 cm. Black (10YR 2/1) sand; massive structure; a few fine roots; pH = 6.0, slightly acid; gradual smooth boundary.
80P2744 126 to 146 cm, 80P2745 146 to 169 cm. Few uncoated sand grains.

B3h 169 —190 cm. Very dark brown (10YR 2/2) sand; massive structure.
80P2746

Figure 3.—Site and pedon description for Echaw S80SC-051-003 from Horry County, South Carolina.

WEISER

SAMPLED AS: LOAMY-SKELETAL, CARBONATIC THERMIC TYPIC CALCIORTHID

S 80NV-003 -008 SAMPLE NOS. 80P1260 - 1265 DATE 10/04/82

CLARK COUNTY

U. S. DEPARTMENT OF AGRICULTURE
SOIL CONSERVATION SERVICE
NATIONAL SOIL SURVEY LABORATORY
LINCOLN, NEBRASKA

GENERAL METHODS 1B1A, 2A1, 2B

SAMPLE NO.	HZN NO.	DEPTH (CM)	HORIZON	(- - TOTAL - - -) CLAY LT .002	SILT .002 -.05	SAND .05 -2	(- -CLAY- -) FINE LT .0002	CO3 LT .002	(- -SILT- -) FINE .002 -.02	COARSE .02 -.05	(- - - - -SAND- - - - -) VF .05 -.10	F .10 -.25	M .25 -.50	C .5 -1	VC 1 -2	(-COARSE FRACTIONS(MM)-) WEIGHT 2 -5	5 -20	20 -75	.1- 75	(>2MM) WT PCT OF WHOLE SOIL
				<- - -	- - -	- - -	- - -	PCT OF <2MM	(3A1)	- - -	- - -	- - -	- - -	- - -	- ->	<- PCT OF	<75MM	(3B1)	->	
				-4-	-5-	-6-	-7-	-8-	-9-	-10-	-11-	-12-	-13-	-14-	-15-	-16-	-17-	-18-	-19-	-20-
801260	1S	0- 5	A11	8.5	19.4	72.1		1	9.7	9.7	36.8	30.2	2.3	1.4	1.4	4	19	8	55	31K
801261	2S	5- 10	A12	8.1	19.3	72.6		1	6.1	13.2	38.6	29.4	2.8	1.7	0.1	5	11	2	46	18K
801262	3S	10- 44	2C1CA	8.1	17.2	74.7		1	6.5	10.7	28.7	26.7	4.2	5.5	9.6	9	34	32	86	75K
801263	4S	44- 61	2C2CA	8.9	12.4	78.7		1	4.9	7.5	23.7	25.6	6.1	10.4	12.9	17	36	7	82	60K
801264	5S	61-107	3C3	16.4	20.9	62.7		TR	11.5	9.4	25.7	22.7	3.0	4.2	7.1	8	24	37	80	69K
801265	6G	107-165	4C4CAM	5.7	15.9	78.4		2	9.3	6.6	10.6	12.0	11.1	23.7	21.0	—	—	—	68	--P

SAMPLE NO.	HZN NO.	ORGN C 6A1C	TOTAL N 6B1B	EXTR P 6S1A	TOTAL S 6R3A	(- - DITH-CIT - -) EXTRACTABLE FE 6C2B	AL 6G7A	MN 6D2A	(RATIO/CLAY) CEC 8D1	15 BAR 8D1	(ATTERBERG) - LIMITS - LL 4F1	PI 4F	(- BULK DENSITY -) FIELD MOIST 4A3A	1/3 BAR 4A1D	OVEN DRY 4A1H	COLE WHOLE SOIL 4D1	(- - -WATER CONTENT - -) FIELD MOIST 4B4	1/10 BAR 4B1C	1/3 BAR 4B1C	15 BAR 4B2	WRD WHOLE SOIL 4C1
		<- - -	- - -	-PCT OF <2MM	- - -	- - -	- - -	- ->			PCT <0.4MM		<- - G/CC	- - -	- ->	CM/CM	<- - -	-PCT OF <2MM	- - -	- ->	CM/CM
801260	1	0.26	0.032						0.65	0.47										4.0	
801261	2	0.16	0.027						0.59	0.46										3.7	
801262	3	0.12							0.51	0.62										5.0	
801263	4	0.08							0.44	0.51										4.5	
801264	5	0.10							0.25	0.48										7.9	
801265	6	0.08							0.30	0.54										3.1	

SAMPLE NO.	HZN NO.	(- NH4OAC EXTRACTABLE BASES -) CA 5B4A 6N2E	MG 5B4A 6O2D	NA 5B4A 6P2B	K 5B4A 6Q2B	SUM BASES	ACID-ITY 6H2B	(- -CEC- -) SUM CATS 5A3A	NH4-OAC 5A6A	EXCH NA 5D2	SAR 5E	BASE SATURATION SUM 5C3	NH4OAC 5C1	CARBONATE AS CACO3 <2MM 6E1B	<20MM	CASO4 AS GYPSUM <2MM 6F1C	<20MM 6F1C	(- - - -PH - - -) SAT PASTE 8C1B	CACL2 .01M 8C1F 1:2	H2O 8C1A 1:1
		<- - -	- - -	- - -	-MEQ / 100 G	- - -	- - -	- - -	- ->	PCT		<- -PCT- >		<- -PCT ->		<- -PCT ->				
801260	1K		1.8	TR	0.5				5.5					20	31				8.0	8.5
801261	2K		1.8	—	0.5				4.8					15	23				8.0	8.6
801262	3K		3.0	TR	0.6				4.1					19	50				8.1	8.6
801263	4K		2.9	0.4	0.7				3.9	7	4			20	49			8.4	8.2	8.8
801264	5K		4.0	5.9	0.8				4.1	47	30			19	44	--		8.2	8.4	8.6
801265	6		1.8	2.4	0.3				1.7	43	19			55		—		8.5	9.2	9.4

SAMPLE NO.	HZN NO.	(- - -WATER EXTRACTED FROM SATURATED PASTE- - -) CA 6N1B	MG 6O1B	NA 6P1B	K 6Q1B	CO3 6I1B	HCO3 6J1B	CL 6K1C	SO4 6L1C	NO3 6M1C	H2O 8A	TOTAL SALTS EST. 8D5	ELEC. COND. 8A1A MMHOS /CM	(- - -MINERALOGY - - -) (- - CLAY - -) (- - X-RAY - -) (- - <2U - -) 7A2B	7A2B	7A2B	7A2B	(- -DTA - -) 7A3	7A3	TOTAL RES 7B1A	DOM WEATH 7B1A
		<- - -	- - -	- - -	-MEQ / LITER	- - -	- - -	- - -	- - -	- ->	<- -PCT	->		<- RELATIVE	AMOUNTS	->		<- - -	- - -	-PCT -	- ->
801260	1												0.19								
801261	2												0.13	MT 1	MI 1	KK 1	CA 1				
801262	3												0.15								
801263	4	1.3	0.9	4.7	0.6	--	2.4	0.3	4.4	0.5	30.3	TR	0.81								
801264	5	7.1	10.4	87.4	1.6	—	1.3	59.3	48.6	8.0	45.6	0.3	9.16	PG 4	MT 1	CL 1					
801265	6	23.5	8.1	76.5	2.0	—	0.9	39.5	79.5	6.5	22.3	0.2	8.62								

FAMILY CONTROL SECTION: DEPTH 25-100 PCT CLAY 12 PCT .1-75MM 82

MMHOS/CM OF 1:2 WATER EXTRACT (8I) FOR LAYERS 1, 2, 3,

ANALYSES: S= ALL ON SIEVED <2MM BASIS G= <2MM ON GROUND <75MM BASIS K=CACO3 ON 20-2 AND <2MM FRACTION

MINERALOGY: KIND OF MINERAL MT MONTMORILL MI MICA KK KAOLINITE CA CALCITE PG PALYGORSKITE CL CHLORITE

RELATIVE AMOUNT 6 INDETERMINATE 5 DOMINANT 4 ABUNDANT 3 MODERATE 2 SMALL 1 TRACE

Series: Weiser Date: 8/81
Pedon No: S80NV-003-008
Taxonomy: Loamy-skeletal, carbonatic, thermic Typic Calciorthid.
Latitude: N36 Deg. 17 Min. Longitude: W115 Deg. 02 Min.
Location: Clark Co. 70M S & 54M W of the NE corner, sec.21,T.19S.,R.60S.
75 M SE of gravel road south of railroad
MLRA: 30 Sonoran Basin and Range
Physiography: Fan in level to undulating upland
Geomorphic Position: On lower third hillside toeslope
Slope and Aspect: 2 pct plane Elevation:
Microrelief: none
Air Temp. 18C Summer: 29C Winter: 7C
Precipitation: 10-cm Aridic moisture regime.
Water Table: Not observed
Drainage: Permeability:
Stoniness:
Land Use: Desert shrubland, ungrazed
Erosion or Deposition:
Parent Material: slightly weathered, alluvium from metamorphic-sedimentary
A11 0 — 5 cm. Pale brown (10YR 6/3) gravelly fine sandy loam, brown (10YR 5/3, when moist); moderate medium platy structure; friable, soft; common fine and common very fine roots; common fine vesicular and

Aluminum cases are used for shipping sampling equipment, supplies, and samples to and from field locations. Cardboard boxes, designed to fit the aluminum cases are used for shipping natural clod samples for bulk-density (4A1), water-retention (4B1), and thin-section (4E1) studies. Bulk samples are collected in 8-mil plastic bags, 23 × 51 cm (9 × 20 in), that are closed before shipping by stapling the folded opening. The samples are then shipped in canvas laundry bags.

Field Sampling 1A

Site Selection 1A1

Site selection, descriptions of the site and soil, and careful sample collection are requisite to successful soil analysis. Locate the site away from roads, fence rows, old farmsteads, and any other features that may have caused aberrant properties.

If duplicate (paired) pedons are to be sampled, the sites should be 1.5 to 30 km (1 to 20 mi) apart, and in different mapping delineations. If a transect is to be sampled, care must be taken to ensure that the variable being tested can be adequately assessed after considerations of other variables in the transect. Satellite pedons can be used very effectively to augment data from a heavily studied central site. Distances apart may be a matter of meters or kilometers for these pedons, depending upon the nature of the test being undertaken.

Pedon Sampling 1A2

Sample freshly dug pits. Dig the pit at least 1 by 2 m across and at least 2 m deep, or into the C horizon, whichever is deeper, or to bedrock if shallower than 2 m. Make supplemental borings or excavations if necessary to assess and describe pedons larger than the pit. In laterally uniform pedons, sample in a vertical pattern 30 to 50 cm wide. Each sample should represent the entire cross section of each horizon. Place a 3- to 5-kg sample, representative of the horizon, in an 8-mil, 4L plastic bag (about three-fourths full). Fold top of bag, place tag in fold and staple. Mark depth, horizon, and pedon number on the tag. Clods for determining bulk density and water retention are normally too small to cover the entire cross section of the horizon and should be taken from the center. Clods for thin sections should come from the feature of interest. Clods can be packaged in plastic bags and placed in cardboard clod boxes for transport. If horizons of a pedon are discontinuous or vary greatly in thickness or degree of expression, collect samples from different parts of the pedon (or different locations on the pit face). If convenient, start sampling at the bottom of the pit to minimize contamination.

If rock fragments >20 mm are present, follow procedures outlined in 1A2a. For contrasting soil materials, estimate the proportions of each component and record in the pedon description. Sample components separately, if reasonable. Make arbitrary subhorizons if morphologically recognizable horizons are more than 30 cm thick in the upper part of the pedon or more than 60 cm in the lower part. Consider the requirements of the classification system in locating subhorizon boundaries.

If for some reason a pit cannot be dug, samples can be taken with a probe core technique as in 1A2c. Bulk and clod samples can be taken from probe cores; however, cores smaller than 5 cm are not suitable for bulk density and water content determination.

For fabric analyses, take samples from the most representative part of the horizon.

Soils with rock fragments 1A2a

Size limits.—The upper size limit for rock fragments is the size of the pedon. The term "rock fragments" refers to particles 2 mm in diameter or larger and includes all sizes that have horizontal dimensions less than the size of a pedon. It is not the same as coarse fragments, which excludes stones and boulders larger than 250 mm. Coarse fragments are 2 to 250 mm. Laboratory data sheets normally report size classes up to 75 mm or 250 mm. The largest size sent to the laboratory with 3- to 5-kg samples is 20 mm.

Volume estimates.—Record in the pedon description the volume percentage estimates of rock fragments >250 mm (10 in), 75 to 250 mm (3 to 10 in), and 20 to 75 mm (¾ to 3 in). Collect a 3- to 5-kg samaple of the <20-mm soil material, and store it in an airtight plastic bag if field moisture content is to be determined. Calculate the volume percentages of coarse fragments (3B2).

Weight estimates.—Estimate and record the volume percentage of the >75-mm fraction as outlined in volume estimates. Collect and weigh a 15- to 20-kg sample of the <75-mm fraction. Sieve out and weigh the 20- to 75-mm fraction. Record the weights of the 20- to 75-mm fraction and the <75-mm fraction.

Collect a 3- to 5-kg sample from the <20-mm fraction, and store it in an airtight plastic bag for determination

sieve. Sieve out the >2-mm fractions (2 to 5 mm and 5 to 20 mm), weigh, and set aside. Continue rolling

membrane with filter candles. Knead the membrane to mix the sample and place it in contact with ethanol to desiccate further. Remove the sample from the membrane and air-dry.

If the buffer-treated sample contains particles larger than 2 mm, wet sieve the sample through a 2-mm sieve. Then dry sieve the material remaining on the sieve (>2 mm) and add the <2-mm fraction from this sieving to the <2-mm fraction separated by the wet sieving. Remove most of the water from the <2-mm fraction with filter candles. Use ethanol to transfer the samples to shallow pans and dry. Ethanol prevents aggregation of clay into durable flakes during drying.

Discussion

The time required for carbonate removal varies greatly, depending on particle size, percentage and type of carbonate, and sample size. Samples from horizons strongly cemented by carbonate have required as long as 2 months. The concentration of alkaline-earth ions in the buffer greatly affects the rate of carbonate removal. Changing the buffer in the reservior well before the buffer capacity has been exhausted, thereby keeping the alkaline-earth ion concentration low, increases the rate markedly. Desalting usually takes about 4 days.

For carbonate-cemented horizons, the whole sample, not just the <2-mm material, must be buffer treated. Furthermore, for horizons without carbonate cementation, buffer treatment of the whole sample has the advantage of washing the >2-mm skeletal material free of adhering fines and organic material. This problem is considered further in 1B4.

For very gravelly horizons, large samples (several kilograms) are necessary for buffer treatment because of the small amount of <2-mm material. Using large samples also increases precision of the >2-mm percentage.

Reference

Grossman and Millet (1961).

Coarse Fragments (2-20 mm) 1B5

2 to 20 mm 1B5a

Crush 2 to 20 mm coarse fragments from 1B1 to <2 mm in a Denver Fire Clay Crusher.

<20 mm 1B5b

Mix thoroughly crushed material from 1B5a with fine earth material from 1B1a.

Whole Soil 1B6

Spread the field samples on trays and air-dry at 30° to 35° C. Use a Denver Fire Clay Crusher and reduce all material to pass a 2-mm sieve.

CONVENTIONS 2

Size Fraction Base for Reporting Data 2A

<2mm 2A1

Unless otherwise specified, all data are reported on a <2-mm base.

>2 mm, Specified Size 2A2

The maximum coarse fragment size for the >2-mm base varies with the relative proportions of the coarse fractions. The base usually includes the entire fraction <75 mm (<3 in) and sometimes the <250-mm (<10-in) fraction. The percentage of those 75 to 250 mm is usually recorded in the field and not measured in the laboratory.

Record the maximum particle size of the base in the data sheet column heading; for example, <75 mm. The base used to calculate the >2-mm percentages reported in the column includes all material in the sample smaller than the size recorded in the heading.

Data Sheet Symbols 2B

The following conventions are used for trace and zero quantities and for samples not tested.

Tr = Trace, either not measurable by quantitative procedure used or less than reportable amount.

— = Analysis run but none detected.

blank = Analysis not run.

PARTICLE-SIZE ANALYSES 3

An automated balance system, consisting of a Radio Shack Model II microcomputer interfaced to a Mettler PL2000 electronic balance (for sand) and a Mettler AE160 electronic balance (for silt and clay), is used for determining, storing, and processing sample weights.

Particles <2 mm (Pipet Method) 3A

Air-Dry Samples 3A1

Apparatus

Fleaker, 300 ml (tare to 1 mg).

Pasteur-Chamberlain filter candles, fineness "F."

Shaker, horizontal, 120 oscillations per minute.

Cylinders, 1,000 ml.

Stirrer, motor-driven.

Stirrer, hand. Fasten a circular piece of perforated plastic to one end of a brass rod.

Shaw pipet rack.

Pipets, 25 ml automatic (Lowy with overflow bulb).

Polyurethane foam, pipe-insulating cover

Shaker with ½-in vertical and lateral movements and 500 oscillations per minute. Accommodates a nest of sieves.

Wide-mouth glass pill bottles with screw caps, 90 ml (tare to 1 mg).

Electronic balance (0.1-mg sensitivity).

Set of sieves. Square mesh woven phosphor bronze wire cloth. U.S. Series and Tyler Screen Scale equivalent designations as follows:

Opening (mm)	U.S. no.	Tyler mesh size
1.0	18	16
0.5	35	32
0.25	60	60
0.105	140	150
0.046	300	300

Reagents

Hydrogen peroxide (H_2O_2), 30 to 35 percent.

Sodium hexametaphosphate $(NaPO_3)_6$. Dissolve 35.7 grams of $(NaPO_3)_6$ and 7.94 grams of Na_2CO_3 per liter of water.

Demineralized water.

Procedure

Removing organic matter.—Place about 10 g air-dry soil containing no particles larger than 2 mm in a tared Fleaker. Add about 50-ml of demineralized water (referred to subsequently as water) and then add 5 ml of H_2O_2. Cover the Fleaker with a watchglass. If a violent reaction occurs, repeat the cold H_2O_2 treatment periodically until no more frothing occurs. Heat the Fleaker to about 90° C on an electric hot plate. Add H_2O_2 in 5-ml quantities at 45-min intervals until the organic matter is destroyed, as determined visually. Continue heating for about 30 min to remove any excess H_2O_2.

Removing cementing agents (optional).—Treat the sample with about 200 ml of 1*N* sodium acetate buffered at pH 5 to remove carbonates. When CO_2 bubbles are no longer evident, wash free of salts with a filter candle system. Highly calcareous samples may need a second treatment.

Remove siliceous cementing agents by soaking the sample overnight in 0.1*N* NaOH. Iron oxide cementing agents are removed by shaking overnight in sodium dithionite (6C2). Wash free of salts with filter candle system before proceeding.

Removing dissolved mineral and organic components.—After the H_2O_2 treatment, place the Fleaker in a rack and add about 150 ml of water in a jet strong enough to stir the sample well. Filter the suspension through a short Pasteur-Chamberlain filter of "F" fineness. Five such washings and filterings are usually enough except for soils containing much coarse gypsum. Remove soil adhering to the filter by gentle back pressure; use finger as policeman. Dry the sample overnight in an oven at 105° C, cool in a desiccator, and weigh to the nearest milligram. Use the weight of the overdry, H_2O_2-treated sample as the base weight for calculating percentages of the various fractions.

Dispersing the sample.—Add 10 ml of sodium hexametaphosphate dispersing agent to the Fleaker containing ovendry treated sample. Make the volume to approximately 200 ml. Stopper and shake overnight on a horizontal reciprocating shaker at 120 oscillations per minute.

Separating sands from silt and clay.—Wash the dispersed sample with water on a 300-mesh sieve. Silt and clay pass through the sieve into a 1-L cylinder. Use a clamp and stand to hold the sieve above the cylinder. Avoid using jets of water in washing the soil. It is important to wash all particles of less than 20μ diameter through the sieve. Remove the sieve from the holder, wash the sands into an evaporating dish with water, and dry at 105° to 110° C. Bring the silt and clay suspension in the cylinder to 1 L with water and cover with a watchglass.

Pipetting.—First pipet the <20-μ fraction at a 10-cm depth. Vary sedimentation times according to temperature. Next, pipet the <2-μ fraction after a predetermined settling time (usually 4½ to 6½ hr). Vary depth according to time and temperature. Use a Lowy 25-ml automatic pipet and regulate filling time to about 12 s. Before each pipetting, stir material in the sedimentation cylinder for 6 min with a motor-drive stirrer (8 min if suspension has stood for more than 16 hr). Remove from stirrer, slip a length of pipe-insulating cover over sedimentation cylinder, and stir the suspension for 30 s with a hand stirrer, using an up-and-down motion. Note the time at completion of stirring. About 1 min before sedimentation is complete, lower the tip of the pipet slowly into the suspension to the proper depth with a Shaw pipet rack. At the appropriate time, fill the pipet and empty into a 90-ml, wide-mouth bottle. Rinse the pipet into the bottle once. Dry in an oven overnight at 105° C. Cool in a desiccator containing phosphorus pentoxide (P_2O_5). Weigh.

Sieving and weighing the sand fractions.—Transfer the dried sands to a nest of sieves. Shake for 3 min on a shaker that has ½-in vertical and lateral movements and oscillates at 500 strokes per minute. Record the weights of the individual sand fractions.

Calculations

Pipetted fractions:

Percentage of pipetted fractions = $(A - B)KD$

where

A = weight (g) of pipetted fraction

B = weight correction for dispersing agent (g)

$$K = \frac{1{,}000}{\text{ml in pipet}}$$

$$D = \frac{100}{}$$

Sand fractions:

Percentage of sieved fractions = weight (g) of fraction on sieve times D.

Coarse silt fraction:

Obtain by difference. Subtract the sum of the percentages of sand plus the <20-μ fraction from 100.

References

Kilmer and Alexander (1949), Kilmer and Mullins (1954), Tyner (1939), and Grossman and Millet (1961).

Carbonate and noncarbonate clay I **3A1a**

Apparatus

Warburg manometer.
¼-oz (5-ml) gelatin capsules.
30-ml plastic cups.

Reagents

Hydrochloric acid (HCl), 6*N*.

Procedure

If carbonate is present, use the glass bottle containing clay residue from regular pipet analysis and [illegible]

Fine clay (<0.2μ) **3A1b**

Apparatus

International No. II centrifuge, head No. 976.
500-ml centrifuge bottles.
Shaw pipet rack.
125-ml Erlenmeyer flasks or weighing bottles (tared to 1 mg).

Procedure

Mix contents of cylinder following pipet procedure. Allow to stand for 15 min and transfer enough suspension to fill a 500-ml centrifuge bottle to the 8-cm depth (≈ 250 ml). Mix contents of bottles and centrifuge at 1,500 rpm, adjusting the time according to the temperature as follows:

Temperature (°C)	Minutes
20	42
21	41
22	40
23	39
24	38
25	37
30	33

Adjust pipet assembly for 3-cm sampling depth. Gently move centrifuge under pipet rack and lower pipet directly into a centrifuge bottle. Withdraw 25 ml in 12 to 15 s (avoid turbulence) and transfer to tared

1¼-inch machined PVC caps with self-sealing, quick-connect fitting, O-ring seal.

¼-oz (5-ml) gelatin capsules.

Reagents

Hydrochloric acid (HCl), 6*N*.

Procedure

Use a glass bottle containing clay residue from regular pipet analysis (3A1a) and determine calcium carbonate equivalent as in method 6E1g.

Sample selection is based on measured carbonate content (6E1g) of the <2-mm sieved sample or reaction (effervescence) of the <2-mm sample to 1*N* HCl.

Calculations

Carbonate clay (pct. <2 mm) (Cc)

Noncarbonate clay percentage <2 mm (Nc)

$$Cc = \frac{(\text{reading} - \text{blank}) \times \text{factor}^*}{\text{total sample weight (3A1)}} \times 100$$

Nc = Total clay – Cc.

** Factor derived from standard curve and includes pipette volume*

References

Shields and Meyer (1964).

Moist Samples **3A2**

If drying affects dispersion of treated sample, ovendrying may be avoided by removal of a pipet sample to estimate the total weight of the sample. Pipet 50 ml at a depth of 20 cm at time zero while the suspension is still turbulent. Use the ovendry weight of the aliquot to calculate the total weight of the

and discarded. The ovendry weight of the second sample is substituted in the calculations for the first sample.

Fine clay (<0.2μ) **3A2b**

Proceed as in 3A1b except use moist samples.

Water-dispersible clay **3A2c**

Proceed as in 3A1c except use moist samples.

Carbonate and noncarbonate clay II **3A2d**

Proceed as in 3A1d except use moist samples.

Particles >2 mm 3B

Fractions >2 mm can be reported on different bases, e.g., fractions <20 mm, <75 mm, or <250 mm, etc. Unless otherwise specified, individual fractions <75 mm are reported on a <75-mm basis and the total >2-mm fraction is reported on a whole-soil (<250-mm) basis. Details on reporting procedures are given in 2A.

Weight Estimates **3B1**

By field or laboratory weighing **3B1a**

Sieve the entire horizon sample through a 75-mm screen and discard the >75-mm fraction. Weigh the <75-mm fraction and sieve through a 20-mm screen. Weigh the 75- to 20-mm fraction. Calculate the <20-mm fraction by subtraction. Take a subsample of the <20-mm fraction for laboratory processing. Determine the air-dry weight of the <20-mm sample and correct the total sample weight for the loss in water from field conditions. Separate and weigh the 2- to 5- and 5- to 20-mm fractions. Report these fractions as percentage of <75 mm. The percentage <250 mm can be reported where applicable from field volume

From volume and weight estimates **3B1b**

If visual field volume estimates are made for fractions >20 mm and fragments of 2 to 20 mm are weighed in the laboratory, convert the weight percentages to volume percentages by applying bulk density and particle density data. Combine these calculated volume percentages with the visual field volume estimates to give a combined volume estimate. Use the following equation to convert all fractions to weight percentages of the combined fractions. The terms are defined in 3B2.

$$\text{Percentage } >2 \text{ mm (weight basis)} = \frac{100Dp\,(x)}{Dp(x) + Db_m\,(1-x)}$$

This equation can be used to calculate any individual fragment fraction >j mm by substituting an appropriate value of Db_m representing the fabric <j mm (where j may be any size fraction).

Volume Estimates **3B2**

Record the estimated volume percentage of material >20 mm (¾ in), >75 mm (3 in), or >250 mm (10 in) in the pedon description. If larger stones or boulders are not included in making the volume estimates, note this in the pedon description.

To arrive at a value for the volume percentage of all material >2 mm, convert the weight estimates of the fine earth to volume estimates and then combine.

$$Cm = \frac{\text{Vol. moist <2-mm fabric}}{\text{Vol. moist whole soil}^1} =$$

$$\frac{Dp(1-y)(1-x)}{Dp(1-y) + Db_m(y)}$$

where

Cm = Coarse-fragment conversion factor
Dp = Density of particles (assumed to be 2.65 g/cm³ unless noted)
Db_m = Bulk density of the moist fine-earth fabric (4A1)

$$x = \frac{\text{vol. fragments >i mm}}{\text{vol. whole soil}}$$

$$y = \frac{\text{wt material between 2 mm and i mm}}{\text{wt material <i mm}}$$

i = the size above which volume estimates are made and below which weight percentages are determined, usually 75 mm or 250 mm

The coarse-fragment conversion factor has been reported on some data sheets and is used to convert laboratory data on a <2-mm weight base to a whole soil moist volume base: Cm × Db_m × laboratory datum. Cm times 100 is the volume percentage of fine-earth fabric in the whole soil. Volume percentage of <2-mm material is the difference between this value and 100.

>2 (pct. by vol.) = 100 (1 − Cm)

For some soils the weight of the fine earth per unit volume of the whole soil is reported. It is calculated by multiplying Cm by Db_m. Values for *x* are reported in the pedon description and those for *y* on the data sheets.

[1] *"Volume moist whole soil" refers to the fine earth plus the coarse fragments in the >2-mm base.*

Two automated balance systems, consisting of two Mettler PC-2000 electronic balances interfaced to Radio Shack Model II and Model IV microcomputers, are used for determining, storing, and processing sample weights.

Bulk Density 4A

Density is defined as mass per unit volume. Soil density as commonly used differs from most density measurements in that the volume of interparticle space is included but the mass of the liquid phase is excluded. Therefore, soil density has been called bulk density, Db, to distinguish it from the more usual density that is based on intraparticle volume only. Since the volume of a shrinking-swelling soil changes with a change in its water content, subscripts are added to designate the mositure condition when the measurement was made. Thus, Db_m is the bulk density of a moist sample, $Db_{1/3}$ is the bulk density of a clod sample equilibrated at 1/3-bar tension, and Db_d is the bulk density of a dry sample.

Saran-Coated Clods 4A1

Regeants

Methyl ethyl ketone.

Dow Saran S310 resin.—The Saran resin dissolves readily in acetone or methyl ethyl ketone. In this method, methyl ethyl ketone is used as a solvent because it is less soluble in water than is acetone and there is less penetration of the Saran-solvent solution into a moist clod. However, acetone is adequate for a first (field) coat and is more readily available. Saran-solvent ratios of 1:4 and 1:7 are used, depending on the porosity of the soil to be coated.

Coating solution.—To prepare the solution, fill a weighted container with solvent to about three-fourths its volume. From the weight of the solvent, calculate the weight of resin required to obtain a predetermined resin-solvent ratio and add to the solvent. Since the solvent is flammable and its vapors form explosive mixtures with air, mix the components with an air-powered or nonsparking electric stirrer under an exhaust hood.[2] If a high-speed stirrer is used, the resin dissolves in about 1 hr. In the field, mix with a wooden stick. Metal cans (1 gal) are statisfactory containers for mixing and storing the plastic. Keep the containers tightly closed to prevent evaporation of the solvent.

Procedure

Collect natural clods (three per horizon) of about 100 to 200 cm^3 in volume (fist-sized). Remove a piece of soil larger than the clod from the face of a sampling pit with a spade. From this piece prepare a clod by gently cutting or breaking off protruding peaks and material sheared by the spade. If roots are present, they can be cut conveniently with scissors or side cutters. In some soils, clods can be removed directly from the face of a pit with a knife or spatula. No procedure for taking clod samples fits all soils; the procedure must be adjusted to meet the conditions in the field at the time of sampling.

The clods are tied with fine copper wire or placed in hairnets and suspended from a rope or string, hung out like a clothesline. Moisten dry clods with a fine mist spray. The suspended clods are dipped by raising a container of the dipping mixture upward around each clod, so it is immersed momentarily. The Saran-coated clods should be allowed to dry for 30 min or longer.[3] For convenience, either of two concentrations of plastic solution is usually used—a 1:7 solution for most soil samples or a 1:4 solution for clods that have large pores. If bulk density at field-moisture content is desired, store the clods in waterproof plastic bags as soon as the coating dries since the coating is permeable to water vapor. Although the coating keeps the clods intact, they may be crushed in transport unless they are packed in rigid containers.

In the laboratory, additional coatings of plastic are applied to make the clod waterproof and to prevent its disruption during wetting. Then weigh the clod, either in its natural moisture condition or in an adjusted

[2] *Information on the safe handling and use of methyl ethyl ketone is available in Chemical Safety Data Sheet SD-83, Manufacturing Chemists' Association, Inc., 1825 Connecticut Avenue NW, Washington, D.C.*

The threshold limits of methyl ethyl ketone are 200 ppm as given in OSHA standards, Part 2, Section 1910.93, table G1.

[3] *Clods coated in this way can be transported to the laboratory and examined microscopically in an undisturbed state.*

moisture condition (e.g., ⅓-bar tension) in air and in water to obtain its volume by Archimedes' principle. Subsequent changes in moisture condition and volume of the soil sample can be followed by reweighing the coated clod in air and in water. Finally, weigh the

Be careful not to lose any soil material because the weight of material lost is calculated as soil moisture, and calculated bulk densities depend on the final ovendry weight of the clod.

Bulk-density values determined by this method are

that may contain particles >2 mm; however, after the measurements are made, the weight and volume of the coarse fraction are subtracted. The remainder consists of the weight of the <2-mm material and the volume of these fine-earth particles and the pore space associated with them.

Db_{od} = bulk density of <2-mm fabric at ovendryness in grams per cubic centimeter

$W_{1/3}$ = the weight percentage of water retained at ⅓-bar tension

wt $clod_{od}$ = weight of ovendry coated clod

wt $clod_{1/3}$ = weight of coated clod equilibrated at ⅓-bar tension

vol $clod_{1/3}$ = volume of coated clod equilibrated at ⅓-bar tension

vol >2 mm = volume of material >2 mm separated from clod after ovendrying

References

Brasher et al. (1966).

Field state (Db_f) **4A1a**

When Saran is dry (approximately 1 hr for most clods—maybe overnight for porous clods), record weight of the clods suspended in air and in water. The difference is the "field-state" volume. Determine ovendry weight and calculate bulk density as described in 4A1.

⅓-Bar desorption I ($Db_{⅓}$) **4A1d**

Remove a patch of Saran coating from a flat face of each clod or cut a flat surface with a diamond saw. Place the exposed area in firm contact with a tension table of porous firebricks covered with very fine sand and equilibrated to 5-cm water tension. When clods have reached a constant weight (7 to 14 days), remove them and record the 5-cm weight in air. This value must be greater than the ⅓-bar or 1/10-bar desorption value. Cover a pressure plate with a 0.5-cm layer of silt and saturate with water. Place a layer of industrial tissue (Kim-wipe) over the silt and press the exposed faces of the clods onto the tissue. Desorb at ⅓-bar suction until water ceases to discharge (about 7 to 14 days). Apply four more coats of Saran plastic and measure volume of clods and calculate bulk density as described in 4A1.

Ovendry (Db_d) **4A1h**

After determining moist volume, ovendry the clods at 105° C for about 48 hr. To reduce cracking of clods that have high-volume change potential, dry overnight at 40° C before drying at 105° C. Record the weight of ovendry clods suspended in air and in water. The difference is ovendry volume. Calculate bulk density as described in 4A1.

Rewet (Db_r) **4A1i**

After measuring the ⅓-bar volume (4A1d), the Saran clods are again desorbed to ⅓-bar as in 4A1d and volume measurements of the clod are made and bulk density is calculated as described in 4A1.

Cores **4A3**

Field moist **4A3a**

To collect cores, prepare a flat surface in the sampling pit, either horizontal or vertical, at the desired depth. Press the core sampler into the soil, using due caution to prevent compaction. Remove the core in the aluminum liner and place both in a moisture can for transport to the laboratory. If the soil is too loose to remain in the liner, use the core sampler without liner and deposit the entire sampler in a moisture can. Moisture cans can be pushed directly into a prepared face. The sample is trimmed even with the top of the can and retained in the can for transport and analysis. For fibrous organic materials, trim sample to fit snugly in a moisture can. If the core contains <5 percent coarse fragments more than 2 mm in diameter and there are no large cracks in the horizon, measured bulk density is used directly.

Calculations

$$\text{Bulk density (g/cm}^3\text{)} = \frac{\text{ovendry wt core (g)}}{\text{vol core (cm}^3\text{)}}$$

Water Retention 4B

If clay within a fragment swells, it tends to push the structure apart, creating more pore spaces that may hold water at low tension. In a natural fragment of reasonable size, this swelling is limited to some extent by the overall coherence of the unit. In very small units, such as the <2-mm fragments of a sieved sample, there are few restraints to such swelling. The result is that moisture-release curves show that sieved samples hold more water at 1/10 or ⅓ bar than larger

References

Young and Dixon (1966).

Pressure-Plate Extraction **4B1**

Apparatus

Pressure-plate apparatus.

Retainer rings, approximately 1 cm high and 4 cm in diameter to hold teaspoon-size samples (10 to 15 g).

Balance, drying oven, and moisture cans.

Air-dry <2 mm (0.06, 1/10, or 2 bar) **4B1a**

Procedure

Place duplicate teaspoon-size samples in retainer rings on a porous plate. Cover the plate with water to wet the samples from below. Cover with a sheet of plastic and let stand overnight. Remove excess water and place the plate in a pressure cooker or other extraction chamber. Apply the specified pressure until no more water is extracted (usually 6 days). After extraction, quickly transfer the samples to moisture cans, weigh, dry at 105° C, weigh again, and report moisture content as percentage of ovendry <2-mm weight.

References

Richards (1954).

Natural clods (0.06, 1/10, 1/3, or 1 bar) **4B1c**

Determine moisture content of clods prepared for bulk-density measurement (4A1 and 4A1d). For precise measurement it may be necessary to correct for the weight of the Saran coating.

To determine 1/10- and 1/3-bar moisture on the same clods, remove the clods from the pressure apparatus when no more water is extracted at 1/10 bar, weigh, and reseat on the silt-covered extraction plate to be equilibrated at 1/3 bar.

For 1-bar measurements without bulk density, samples are prepared similar to those for bulk density measurements (4A1). After equilibrating at 1 bar, samples are quickly transferred to moisture cans and the moisture content is determined. Report as percentage of ovendry <2-mm weight.

Cores **4B1d**

Proceed as in 4B1a except use core samples in their collecting rings.

Rewet **4B1e**

Determine moisture content of clods prepared for rewet bulk-density measurements (4A1i). It is necessary to correct for the weight and volume of the Saran coating. Report as percent of ovendry, <2-mm weight.

Pressure-Membrane Extraction **4B2**

Apparatus

Pressure-membrane apparatus with sausage-casing membrane.

Retaining rings—rubber soil-retaining rings 1 cm high and about 4 cm in diameter that hold about 12 g soil.

Balance, drying oven, and moisture cans.

Air-dry <2 mm (15 bar) **4B2a**

Procedure

Install a wet cellulose membrane in the pressure apparatus. Add water and fill the retaining rings with <2 mm of soil. Cover with a plastic sheet, and allow samples to soak overnight. Remove excess water and close the apparatus. Gradually apply pressure in increments of 25 psi at intervals of about 15 min to the pressure desired. After a few hours to 1 day, apply a 4-psi pressure differential to the rubber diaphragm at the top of the chamber. Remove samples when cessation of outflow indicates that equilibrium has been reached (2 to 6 days). Quickly transfer samples to moisture cans and determine moisture content. Report as percentage of ovendry weight.

References

Richards (1954).

Field moist <2 mm (15 bar) **4B2b**

Proceed as in 4B2a except use field-moist samples instead of air-dry samples.

Field State 4B4

Clods, cores, or mixed samples taken directly into the laboratory, with due precaution to prevent moisture loss, can be used to determine water content at time of sampling. Determine moisture content by ovendrying at 105° C.

Air-Dry 4B5

Determine moisture content of air-dry samples by ovendrying at 105° C.

Water Retention Difference (WRD) 4C

Between 1/3-Bar and 15-Bar Tension 4C1

$$WRD = \frac{(W_{1/3} - W_{15})(Db_{1/3})Cm}{100}$$

where

WRD = weight (g) of water retained in 1 cm^3 of whole soil between 1/3-bar and 15-bar tension and is reported as centimeter per centimeter (numerically equivalent to inches of water per inch of soil).

$W_{1/3}$ = weight percentage of water retained at 1/3-bar tension (4B1c)

W_{15} = weight percentage of water retained at 15-bar tension (4B2)

$Db_{1/3}$ = bulk density of the <2-mm fabric at 1/3-bar tension (4A1)

$$Cm = \frac{\text{Vol moist <2-mm fabric } (cm^3)}{\text{Vol moist whole soil } (cm^3)} \quad (3B2)$$

Between 1/10-Bar and 15-Bar Tension 4C2

Substitute 1/10- for 1/3-bar value, and calculate as in 4C1.

Between 1/3-Bar Rewet and 15-Bar (Air-Dry) Tension 4C3

Substitute 1/3 bar rewet for 1/3-bar value, and calculate as in 4C1.

Coefficient of Linear Extensibility (COLE) 4D

Coefficient of linear extensibility denotes the fractional change in clod dimension from a dry to a moist state. It has also been expressed as LE (linear extensibility). LE = COLE × 100. When coarse fragments are absent:

$$COLE = \frac{[L_m - L_d]}{L_d} = \frac{L_m}{L_d} - 1 = \left(\frac{Db_d}{Db_m}\right)^{1/3} - 1$$

where

L_d = length of clod, dry

L_m = length of same clod, moist

Db_d = bulk density of clod, dry

Db_m = bulk density of clod, moist

Air-Dry or Ovendry to 1/3-Bar Tension 4D1

Coefficient of linear extensibility can be estimated from laboratory bulk-density data and the coarse-fragment conversion factor (Cm).

$$COLE = \left(\frac{1}{Cm\left(\frac{Db_m}{Db_d}\right) + (1 - Cm)}\right)^{1/3} - 1$$

where

$$Cm = \frac{\text{Vol moist <2-mm fabric}}{\text{Vol moist whole soil}} \quad (3B2)$$

Db_m = bulk density of the <2-mm fabric at 1/3 bar (4A1)

Db_d = bulk density of the <2-mm fabric at ovendryness or air-dryness (4A1)

If there is no coarse material, Cm = 1 and the equation reduces to

$$COLE = \left(\frac{Db_d}{Db_m}\right)^{1/3} - 1$$

COLE calculated for the fine-earth fabric alone can be referred to as $COLE_f$ (or LE_f).

Nomographs for these calculations have been published by Holmgren (1968).

References

Franzmeier and Ross (1968), Grossman et al. (1968), and Holmgren (1968).

Micromorphology 4E

Thin Sections 4E1

Preparation 4E1a

Apparatus

Micro-Petrolab, thin sectioning center[4].
Metallographic polisher with cast-iron laps.

Reagents

Epo-Kwick epoxy[5].
Scotchcast resin[6].

Collecting samples.—Samples can be collected by any procedure that does not disturb the natural structure. Core samplers are commonly used. Another satisfactory procedure for some soils is using a knife or trowel to carve a clod that fits a tin box or round ice-cream container. Clods selected from bag samples can be used if orientation is of no interest. Large clods can be shipped if packed tightly in boxes. Fill any space around irregular clods in the container with loose soil from the same horizon. For most samples, it is unnecessary to prevent moisture loss unless fragility is affected, since samples usually must be dried before impregnation. The whole core or clod can be impregnated, but better results generally are obtained with specimens that are 5 cm^3 or smaller. Remove specimens from the sample with a small chisel, a probe like an ice pick, an ordinary hacksaw, or a jeweler's hacksaw.

Preparing samples.—First dry a soil sample in an oven, preferably overnight or longer at about 80° C in a disposable heat-and chemical-resistant container. This treatment may cause some change in structure, but thus far good impregnation seems impossible unless the sample is thoroughly dried. If freeze-drying equipment is available, the natural structure of the soil samples can be better preserved by this technique. Impregnation of the freeze-dried samples may be

conditions for air bubbles escaping from the sample

Final grinding.—In preparation for the final grinding,

min), remove the container from the desiccator, reheat it to 80° C, and then return it to the desiccator. Repeat the heating-evacuation cycle three or four more times. Then cure the plastic in the oven at 105° C overnight. The block is then ready for sectioning. Disposable containers can be cut with the cooled samples during sectioning.

saw. The sample can be hand held or a slide holder can be used. If the vacuum chuck and the Micro-Petrolab are used, make certain that the glass is clean and is seated in the chuck. The Micro-Petrolab cuts off the excess sample and then grinds the sample down to approximately 30μ. If the lap is to be used, the mounted sample—now about 0.050 to 0.100 mm

these dense materials. Place pieces of air-dry soil material in xylene and evacuate. Then submerge the xylene-saturated soil material in molten Aroclor 5460. Hold the sample in the Aroclor at about 200° C for 1 to 2 days. Remove impregnated soil material, allow it to cool, and prepare thin sections by dry-grinding.

References

Lockwood (1950), Rogers and Kerr (1933), Reed and Mergner (1953), Cady (1965), and Innes and Pluth (1970).

Interpretation **4E1b**

It is desirable to become familiar with the overall features of the section first. Many studies requiring thin sections are concerned with movement of clay or other substances and differences between horizons as a result of soil-forming processes and weathering. Thus, it is helpful to scan all the sections from a profile or all connected with a particular problem to see what important features may be worth the most emphasis. Different kinds of illumination should be used with each magnification. Strong convergent light with crossed polarizers brings out structures in dense or weakly birefringent material that may appear opaque or isotropic. Structures in translucent specimens become more clearly visible if plain light is used and the condensers are stopped down. Everything should be viewed in several positions of the stage or during slow rotation with crossed polarized light.

A thin section is a two-dimensional slice through a three-dimensional body. Mineral grains and structural features are seen in one plane and the shapes seen must be extrapolated to their true shapes. A grain that appears needle shaped may be a needle or the edge of a flat plate. An elliptical pore may be an angular slice through a tube. A circular unit is probably part of a sphere. Repeated viewing of similar features that appear to be cut at different angles with the three-dimensional appearance in mind is the best way to accustom oneself to a volume rather than a planar interpretation of shape. An observer must also keep thickness of the section in mind. A well-prepared section is 20μ to 30μ thick. Grains smaller than this are stacked up and cannot be seen as individuals, and pores much smaller than this cannot be seen clearly.

Sand and silt grains in thin sections are identified by standard methods given in petrography texts. The general approach is the same as for grain studies (7B1) except that refractive index can be used only roughly and more weight is given to other optical and morphological properties. It is seldom necessary to be concerned with minerals that occur in small quantities or to attempt quantitative mineralogical analysis. If identification and mineralogical analysis are important to the problem being studied, it is best to do them on separate size fractions and to use the thin sections mainly for information about arrangement of the components. Recognition of aggregates, concretions, secondary pseudomorphs, and weathered grains is more important in thin-section studies than in sand and silt petrography. It can be easier because interior structures are exposed. Although grains of this kind may be important in studying soil genesis, they are often destroyed or eliminated by sample-preparation procedures that separate sand, silt, and clay.

By far the greatest interest in micromorphology, in the United States at least, has centered on the arrangement of clay. Clay occurs not only in the form of aggregates but also in massive interstitial fillings, coatings, bridges, and general groundmass. Even though the particles are submicroscopic, the clay can be described and characterized and sometimes identified; at least the 1:1 and 2:1 lattice types can be distinguished. Completely random-arranged clay of less than 1μ exhibits no birefringence and appears isotropic in crossed polarized light. The clay in a soil is seldom all random and isotropic. It develops in oriented bodies during formation or becomes oriented by pressure or translocation. If enough plate-shaped particles are together and oriented in a body large enough to see, birefringence can be observed.

The silicate clay minerals in soils, except halloysite, are platy. The *a* and *b* crystallographic axes are within the plane of the plate, and the *c* axis is almost perpendicular to this plane. The crystals are monoclinic, but the distribution of stems along the *a* and *b* axes is so nearly the same and the *c* axis is so nearly perpendicular to the other axes that the minerals are pseudohexagonal. The optical properties of clay as well as its crystal structure and general habit are analogous to those of the micas, which can be used as a model in thinking about and describing the properties of clays.

Since the speed of light traveling in the direction of the *c* axis and vibrating parallel to the *a* axis is almost the same as that vibrating parallel to the *b* axis, the refractive indices are very close. Hence, interference effects seen in crossed polarized light are small when the observer is looking along the *c* axis. Light vibrating

parallel to the *c* axis travels faster than in other directions and hence the refractive index is lower. If the edge of the crystal or aggregate of crystals is viewed along the *a-b* plane between crossed polarizers, there are two straight extinction positions and interference colors will show in other positions. If a concentration of clay is organized so that most of the plates are parallel, the optical effects can be observed. How completely or satisfactorily they can be observed depends on the purity and continuity of the clay body and on the process that oriented it.

Kaolinite has low birefringence and refractive indices slightly higher than quartz. In the average thin section, its interference colors are gray to pale yellow; in residual soils it often occurs as booklike and accordionlike aggregates of silt and sand size.

Halloysite, because of its tubular habit, should not show birefringence even though it can form oriented aggregates. It may show very faint, patternless birefringence caused by impurities or refraction of light at interfaces between particles.

The 2:1 lattice minerals have high birefringence and show bright intermediate-order interference colors if the edges of the aggregates are viewed. It is seldom possible to distinguish between clay-size montmorillonite, mica, vermiculite, and chlorite in thin section. These clay minerals seldom occur pure in soils; they are usually mixed and in many soils are stained by and mixed with iron oxide and organic matter.

Residual clay has been in place since its formation by weathering or since deposition of a transported soil parent material. Local adjustment of position may have occurred, but such clay has not moved separately. It may be random, completely unoriented, and thus isotropic, but more often it shows some birefringence. In transported materials, silt-size flakes and other small aggregates are common. In many residual materials, clay is arranged in forms pseudomorphic after rock minerals or in crystal aggregates in definite bodies such as the vermicular or accordionlike kaolin books. Regular, intact arrangement of these materials generally is diagnostic for residual material.

Clay becomes rearranged by stress applied differentially to produce shear. Platy particles become oriented by slipping along a plane like the slickenside faces in a Vertisol or in heavy glacial till; they are also oriented inside the blocks. Root pressure, mass movement, slump, and creep can produce stress orientation.

Stress orientation can be inferred if the faces seen on structural units are smooth and do not have a separate coating; otherwise, it cannot be observed in plain light. In plain light, clay in the section may be homogeneous and featureless. In crossed polarized light the pattern of orientation is reticulate, consisting of bright lines showing aggregate birefringence often intersecting at regular angles. The effect is that of a network in a plaid pattern. There may be numerous sets of these slip planes that appear in different positions as the stage is turned. Stress-oriented clay may appear around rigid bodies like quartz grains or along root channels. It is often strongly developed on ped faces. Stress can also orient mica flakes and any other small platy grains.

Translocated clay has several features that distinguish it from residual clay. It occurs in separate bodies, usually having distinct boundaries, and is located on present or former pore walls, channel linings, or ped faces. It is more homogeneous than matrix clay and is usually finer. It may have a different composition from the matrix, especially if it came from another horizon. It shows lamination, indicating deposition in successive increments. Finally, these bodies of translocated clay show birefringence and extinction, indicating that they are oriented aggregates. If they are straight, they have parallel extinction; if curved, a dark band is present wherever the composite *c* axis and composite *a* and *b* axes are parallel to the vibration planes of the polarizers. These dark bands sweep through the clay aggregate when the stage is rotated.

Other substances may form pore linings and ped coatings. The more common are goethite, gibbsite, carbonate minerals, and gypsum; they can be identified by their mineralogical properties.

Amorphous coatings of organic matter with or without admixed iron and aluminum are common, especially in spodic horizons. This material is dark brown to black, isotropic or faintly birefringent, and often flecked with minute opaque grains. It occurs as the bridging and coating material in B horizons of sandy Spodosols and as a thin coating or stain on faces of pores and peds in other soils.

References

Cady (1965).

Plasticity Index 4F

The plasticity index is the difference in water content between the plastic limit and the liquid limit. Details are available in ASTM method D 424.

References

American Society for Testing and Materials (1970).

Liquid Limit **4F1**

See ASTM method D 423.

Plastic Limit **4F2**

See ASTM method D 424.

Cation-Exchange Capacity 5A

By Summation **5A3**

Sum of cations (CEC-8.2) **5A3a**

Procedure

Compute the cation-exchange capacity (CEC-8.2) from the sum of the extractable bases (NH_4OAc extract, method 6N2, 602, 6P2, 6Q2) and the extractable acidity obtained by titrating the triethanolamine (TEA) extract (6H). Values for exchange capacity by this method are not valid if significant quantities of soluble salts or calcium carbonate are present in the soil.

References

Peech et al. (1947).

Effective cation exchange capacity (ECEC) **5A3b**

Sum the extractable bases by ammonium acetate, pH 7, and the KCl-extractable aluminum.

NH_4Cl **5A7**

Procedure

Proceed as in 5A6 except substitute 1 *N* NH_4Cl for 1*N* NH_4OAc.

Direct distillation **5A7a**

Determine ammonia by Kjeldahl distillation as described in 5A8a.

NH_4OAc, pH 7.0—Automatic Extractor (CEC-7) **5A8**

Apparatus

Automatic extractor, 24 place.

Syringes, 60 cc, polypropylene. Use one sample tube, one reservoir tube, and one tared extraction syringe for each sample.

Reagents

Ammonium acetate (NH_4OAc), 1*N*, pH 7.0.

—Mix 68 ml ammonium hydroxide (NH_4OH), specific gravity of 0.90, and 57 ml of 99.5-percent acetic acid

(CH_3COOH) per liter of solution desired. Cool, dilute with water to the specified volume, and adjust to pH 7.0 with CH_3COOH or NH_4OH.

Optionally, prepare from NH_4OAc reagent salt and adjust pH.

Ethanol (CH_3CH_2OH), 95-percent, U.S.P.

Procedure

Prepare sample tubes by tightly compressing a 1-g ball of filter pulp into bottom of syringe barrel with plunger. Weigh to the nearest 0.01 g, approximately 2.5 g soil sample and place in tube.

Place sample tube in upper disc of extractor and connect to inverted, tared extraction syringe, the plunger of which is inserted in slot of stationary disc of extractor. Fill to 25-ml mark with NH_4OAc above soil, and let stand for 20 min.

Put reservoir tube on top of sample tube, extract rapidly until NH_4OAc is at a depth of 0.5 to 1.0 cm above sample. Turn off extractor, add about 45 ml NH_4OAc to reservoir tube, turn on extractor, extract overnight.

Weigh syringes containing NH_4OAc extract to the nearest one hundredth of a gram. Mix, and then set aside a portion of extract for determination of extractable cations (or bases, 5B5), and discard the remainder. See analytical procedures for calcium (6N2e), magnesium (602d), sodium (6P2b), or potassium (6Q2b).

Attach syringes to sample tubes, rinse sides of sample tubes with ethanol from wash bottle, fill sample tubes to 25-ml mark, stir, and let stand for 15 to 20 min. Place reservoir tube in sample tube. Extract at a setting of 25 to 35 until the level of liquid is 0.5 to 1.0 cm above sample. Turn off extractor and add enough ethanol to reservoir to assure an excess over the capacity of the syringe. Extract at a setting of 12—approximately 45 min.

Extract with ethanol a second time but omit stirring the samples.

References

Holmgren, Juve, and Geschwender (1977).

Direct distillation **5A8a**

Reagents

Sodium chloride (NaCl).

Antifoam mixture.—Mix equal parts of mineral oil and n-octyl alcohol.

Sodium hydroxide (NaOH), 1*N*.

Hydrochloric acid (HCl), 0.2*N*, standardized.

Boric acid (H_3BO_3), 4 percent.

Procedure

Transfer the soil plus filter pulp from methods 5A8 or 5A9 to a Kjeldahl flask. Add 400 ml water and about 10 g NaCl, 5 drops antifoam mixture, a gram or two of granular zinc, and 40 ml of 1*N* NaOH. Connect the flask with the condenser and distill 140 ml into 50 ml of 4-percent H_3BO_3 solution in 250-ml titrator beaker. Titrate with automatic titrator to end point pH setting of 4.60.

Calculations

$$\text{CEC (meq/100g)} = \frac{\text{ml HCl}}{\text{g sample}} \times \text{normality of acid} \times 100$$

Report on ovendry basis.

References

Peech et al. (1947).

Steam distillation **5A8b**

Apparatus

Steam distillation/titration apparatus, Kjeltec auto 1030 analyzer.

Alphacom 40 printer.

Digestion tubes, 250-ml straight neck.

Reagents

Sodium chloride

Antifoam, spray can silicone.

Sodium hydroxide (NaOH), 1*N*.

Hydrochloric acid (HCl), 0.1*N*, standardized.

Boric acid 4% (W/V) aqueous solution with Bromocresol Green-Methyl Red Indicator[7].

[7] *Ricca Chemical Company, Arlington, Texas.*

Procedure

Transfer the soil plus filter pulp from method 5A8 or 5A9 to a 250-ml digestion tube, using a minimum amount of water. Add 6 to 7g of sodium chloride. Refer to the instructional manual for operation of the Kjeltec auto 1030 analyzer. Spray antifoam into the digestion tube and connect to the distillation unit. Close the safety door. The distillation, titration, and calculation are performed automatically in about 4 min. Result is printed in milliliters of titrant.

Calculations

$$\text{CEC(meq/100g)} = \text{ml HCl} \times \frac{\text{normality of Acid} \times 100}{\text{g sample}}$$

Report on ovendry basis.

NH_4Cl, Automatic Extractor — 5A9

Procedure

Proceed as in 5A8, except substitute 1*N* NH_4Cl for 1*N* NH_4OAc.

Direct distillation — 5A9a

Determine ammonia by Kjeldahl distillation as described in 5A2a.

Uncorrected (extractable) — 5B5a

If a soil does not contain soluble salts or carbonates, the extractable bases are presumed to equal the exchangeable bases. They are, however, reported as extractable bases.

Corrected (exchangeable) — 5B5b

If a soil contains soluble salts, estimate their amount from the saturation extract as follows. Multiply cation concentration in the saturation extract (meq/L) by the saturation percentage (divided by 1,000) to convert to milliequivalents per 100 g. Subtract this quantity from the concentration of the extracted cation. This procedure is not valid for calcium and magnesium in the presence of carbonates that contain those elements, or for calcium in the presence of gypsum, because these compounds are soluble in NH_4OAc.

Base Saturation — 5C

NH_4OAc, pH 7.0 — 5C1

Divide sum of NH_4OAc-extracted bases by the exchange capacity determined by the NH_4OAc method (CEC-7).

Sum of Cations, TEA, pH 8.2 — 5C3

Divide the sum of NH_4OAc-extracted bases by the sum of cations determined by the method 5A3a

$$SAR = \frac{[Na^+]}{\sqrt{\frac{[Ca^{++}] + [Mg^{++}]}{2}}}$$

where $[Na^+]$, $[Ca^{++}]$, and $[Mg^{++}]$ refer to the concentration of these cations expressed in millequivalents per liter in the saturation extract (8A3).

References

Richards (1954).

Aluminum Saturation 5G

Bases Plus Aluminum 5G1

Divide the KCl-extracted aluminum by the sum of NH_4OAc-extracted bases plus the KCl-extracted aluminum (ECEC).

The following instruments or devices are used at the NSSL for the indicated procedures:

(1) *Sample Weighing*

Mettler Model PC220 interfaced with a Radio Shack Model IV microcomputer. A Mettler Model AE160 analytical balance is also used for more precise weighings.

(2) *Sample Extraction*

Samples are extracted with solutions of NH_4OAc, KCl, NH_4Cl, or water with a Concept Engineering Mechanical Extractor. The extractor has a capacity of twenty-four 60-cc syringe barrels for smaller samples or 12 specially-designed funnels for saturated pastes. Six of these units are used in the characterization laboratory.

(3) *Atomic Absorption*

[redacted]

automatic sample changer and Model 3600

(6) *Digestion and Distillation*

A Tecator Digestion System 20-1005 heating unit is combined with a Tecator 1030 analyzer for automatic distillation and titration for determination of total nitrogen and cation exchange capacity.

(7) *Ion Chromatograph*

A Dionex Model 2110i Ion Chromatograph with Advanced Chromatography Module and automatic sample changer are used for the determination of chloride, nitrate, and sulfate in water extracts.

(8) *Manometer*

Datameterics Model 1174 Barocel Electronic Manometer with pressure sensor and thermal base is used for determination of calcium carbonate equivalent.

(9) *Conductivity Meter*

Markson 4405 Digital Conductivity Analyzer with temperature-compensating cell is used for determination of electrical conductivity of water extracts.

(10) *Diluting/Dispensing*

Hamilton Digital Diluter for rapid dispensing of fluids.

Organic Carbon 6A

Acid-Dichromate Digestion 6A1

CO_2 evolution, gravimetric 6A1b

Reagents

Digestion-acid mixture.—Mix 600 ml concentrated H_2SO_4 and 400 ml 85-percent H_3PO_4.

Potassium dichromate ($K_2Cr_2O_7$), reagent grade.

Potassium iodide (KI).—Dissolve 100 g KI in 100 ml water.

Silver sulfate (Ag_2SO_4), saturated aqueous solution.

Concentrated sulfuric acid (H_2SO_4).

Other reagents.—indicarb or Mikohbite, soda lime, 30-mesh zinc, and anhydrone (anhydrous magnesium perchlorate).

Apparatus

See figure 6.

Procedure

Place a soil sample containing 20 to 40 mg carbon (usually 0.5 to 3 g ovendry soil) in digestion flask and add 1 to 2 g $K_2Cr_2O_7$. Wash the neck of the flask with 3 ml water and connect the flask to reflux condenser. Attach the weighed Nesbitt bulb to the system and open the valve at the top. Pour 25 ml digestion-acid mixture into funnel, let it enter the flask, and close the stopcock immediately to prevent loss of CO_2. Use digestion-acid mixture to lubricate the funnel stopcock. The tip of the air-delivery tube should extend about 0.5 cm below the surface of the acid during digestion. Adjust the "carrier stream" to a flow rate of one or two bubbles per second and maintain this rate during digestion. Heat with a gas flame of sufficient intensity to bring the sample to boiling in 3 to 4 min. Continue gentle boiling for a total heating period of 10 min (avoid excessive frothing). Heating is too rapid if white fumes of SO_3 are visible above the second bulb of the reflux condenser during boiling. At the end of the digestion period remove the flame and aerate for 10 min at the rate of six to eight bubbles per second. Then close the stopcock on the Nesbitt bulb, disconnect the bulb from the system, and weigh.

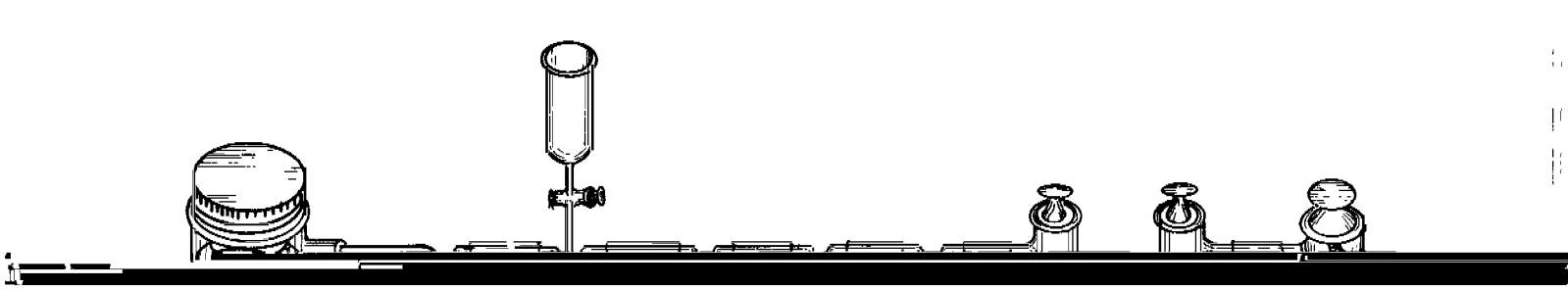
Calculations

$$\text{Organic carbon (pct.)} = \frac{\text{final wt bulb (g)} - \text{initial wt bulb (g)}}{\text{g sample}} \times 27.29$$

Report on ovendry basis.

References

Allison (1960).



Calculations

Organic carbon (pct.) =

$$\frac{FeSO_4 \text{ blank (ml)} - FeSO_4 \text{ sample (ml)}}{\text{g sample}} \times \text{normality of } FeSO_4 \times \frac{0.30}{0.77}$$

0.77 is the recovery factor proposed by Walkley (1935).

Report on ovendry basis.

References

Peech et al. (1947) and Walkley (1935).

Dry Combustion **6A2**

CO_2 evolution III **6A2c**

Apparatus

LECO 70-second carbon analyzer, model 750-100.

LECO induction furnace, model 521-000.

Reagents

Manganese dioxide.

Antimony.

1-g standard sample rings containing 0.870 percent carbon.

1-g standard sample rings containing 0.073 percent carbon.

Metal accelerator.

Iron chip accelerator.

Anhydrone.

Procedure

For noncalcareous soils, weigh approximately ½ g of <2-mm soil into crucibles in duplicate. Add to the soil in the crucibles one scoop of copper accelerator and one scoop of iron chip accelerator. Mix by stirring. Add an additional scoop of iron chips to the stirred mixture. Four standard soils containing 0.8, 2.1, 3.5, and 6.5 percent organic carbon are run with each group of soils. Follow LECO instruction manuals for instrument operation. Record readings from digital voltmeter as percent carbon.

References

Tabatabai and Bremner (1970).

Sodium Pyrophosphate Extraction **6A4**

This extraction procedure is used if organic carbon as well as pyrophosphate-extractable Fe and Al are to be determined. The Fe and Al are extracted by method 6C8 in any case.

Reagents

Sodium pyrophosphate ($Na_4P_2O_7$), 0.1*M*.

Superfloc solution, 0.2 percent in water.

Procedure

Place 2 g soil into 500-ml centrifuge bottle. Add 236 ml 0.1*M* $Na_4P_2O_7$, stopper and shake overnight in reciprocating shaker. Remove stoppers, add 10 drops Superfloc, replace stoppers, and shake each bottle by hand for 15 s. Remove stoppers, and centrifuge for 30 min at 1,900 rpm.

CO_2 evolution, gravimetric **6A4a**

Procedure

Proceed as in 6A1b except that in place of soil sample, pipet 100-ml aliquot of extract into 100-ml Kjeldahl flask, add 1 ml of digestion-acid mixture and evaporate to dryness in boiling water bath.

Calculations

Organic carbon (pct.) =

$$\frac{\text{final wt bulb (g)} - \text{initial wt bulb (g)}}{\text{g sample}} \times 27.29 \times \text{dilution}$$

Report on ovendry basis.

References

Allison (1960).

Nitrogen 6B

Kjeldahl Digestion I 6B1

Reagents

Concentrated sulfuric acid (H_2SO_4).

Salt mixture:

Potassium sulfate (K_2SO_4), 1,000 g.

Ferrous sulfate (anhydrous) $FeSO_4$, 55 g.

Copper sulfate (anhydrous) $CuSO_4$, 32 g.

Hengar granules (selenized).

Procedure

Weigh 5 g soil into 800-ml Kjeldahl flask, add 20 ml distilled water and let stand overnight. Add 10 g salt mixture, 2 or 3 Henger granules, and 30 ml H_2SO_4. Digest on Kjeldahl digestion heaters, rotating flasks frequently. Continue digestion 1 hr after mixture is clear.

References

Association of Official Agricultural Chemists (1945).

Ammonia distillation, automatic titrator 6B1b

Reagents

solution. Connect flask to condenser and distill ammonia into 250-ml titrator beaker containing 50 ml H_3BO_3 solution. Titrate with standard HCl to end point pH setting of 4.60 on automatic titrator.

Calculations

$$\text{N (pct.)} = \frac{\text{HCl sample (ml)} - \text{HCl blank (ml)}}{\text{g sample}} \times \text{normality of HCl} \times 1.4$$

Report on ovendry basis.

Kjeldahl Digestion II 6B3

Apparatus

Aluminum digestion block, 20 places for 250-ml digestion tubes.

Reagents

Concentrated sulfuric acid (H_2SO_4).

Salt mixture:

Potassium sulfate (K_2SO_4), 1,000 g.

Ferrous sulfate (anhydrous) $FeSO_4$, 55 g.

[redacted]

Hengar granules (selenized).

Procedure

Weigh 3 g soil or less into 250-ml digestion tubes.

Ammonia steam distillation, automatic titrator **6B3a**

Apparatus

Steam distillation/titration apparatus, Kjeltec auto 1030 analyzer.

Alphacom 40 printer.

Digestion tubes, 250-ml.

Reagents

Sodium Hydroxide (NaOH), 50 percent solution.

Boric acid (H_3BO_3) 4 percent (W/V) aqueous solution

[redacted]

Procedure

Refer to instruction manual for operation of Kjeltec auto 1030 analyzer. From procedure 6B3, connect digestion tube to distillation unit. Close the safety door. The distillation, titration, and calculation are performed automatically in about 4 min. Result is printed in percent nitrogen.

citrate. Make up to 4 oz with water, and shake overnight in a reciprocating shaker. Add 2 ml Superfloc solution to the suspension, make up to 8 oz with water, shake vigorously for 15 s and allow to settle for at least 1 hr. This extract is used for analysis of iron (6C2b), aluminum (6G7a), and manganese (6D2a).

References

Holmgren (1967).

Atomic absorption **6C2b**

[redacted]

Atomic absorption spectrophotometer.

Diluter.

Reagents

Iron solution 1,000 mg/L, standard (optionally 2,000 mg/L).

Atomic absorption 6C7a

Reagents

Standard Fe solutions, 100 and 200 mg/L.

Procedure

Dilute HF extracts from 7C3 and Fe standards fivefold to twentyfold with water. Compare absorbance of samples with that of standards at 372.0 nm. The concentration of most cations must be kept nearly constant to eliminate ionization interferences.

Calculations

$$\text{Fe (pct.)} = \frac{\text{Fe (mg/L)} \times 10}{\text{mg clay}} \times \text{dilution}$$

$$Fe_2O_3 \text{ (pct.)} = \text{Fe (pct.)} \times 1.43$$

Report on ovendry basis.

Sodium Pyrophosphate Extraction II 6C8

Reagents

Sodium pyrophosphate ($Na_4P_2O_7$), 0.1*M*.

Superfloc solution, 0.2 percent in water.

Procedure

Place 2 g soil into 8-oz shaking bottle. Add 0.1*M* $Na_4P_2O_7$ to 7 oz mark, stopper and shake overnight in reciprocating shaker. Add 4 ml Superfloc and make up to 8 oz volume with 0.1*M* $Na_4P_2O_7$. Shake and let stand several days to allow suspension to settle. This extract is used for analysis of iron (6C8a) and aluminum (6G10a).

Atomic absorption 6C8a

Apparatus

Atomic absorption spectrophotometer.

Reagents

Standard Fe solution, 50 mg per 8 oz.

Procedure

Measure absorbance of sample (6C8) at 372.0 nm and compare with absorption of standard prepared by pipetting 50 ml of 1,000 mg/L Fe solution into 8-oz shaking bottle and carrying through extraction procedure as in 6C8. The concentration of most cations must be kept nearly constant to eliminate ionization interference.

Calculations

$$\text{Fe (pct.)} = \frac{\text{Fe (mg/8 oz)}}{\text{g sample}} \times \frac{\text{dilution}}{10}$$

Report on ovendry basis.

Manganese 6D

Dithionite-Citrate Extraction 6D2

Extract soil sample as described in 6C2.

Atomic absorption 6D2a

Apparatus

Diluter.

Atomic absorption spectrophotometer.

Reagents

Manganese solution 1,000 mg/L, standard.

Superfloc flocculating agent, 0.2 percent in water.

Procedure

Add 2 ml Superfloc to dithionite-citrate treated soil suspension, (from 6C2) bring volume to 8 oz with water and shake vigorously. Allow to settle until supernatant is clear. Dilute the supernatant fivefold to twentyfold with the diluter, dispensing into polyethylene sample vials of the automatic sample changer. Dilute standards in the same ratio.

Determine the flame absorption of the sample at 403.1 nm. Calibrate instrument to read directly in milligrams per 8 oz.

To prepare the standards, pipet 10-, 25-, 50-, and 100-ml aliquots of standard manganese solution (1,000 mg/L) into 8-oz shaking bottles, making to 8 oz after adding reagents and treating as in 6C2.

Calculations

$$\text{Mn (pct.)} = \frac{\text{Mn (mg/8 oz)}}{\text{g sample}} \times \frac{\text{dilution}}{10}$$

Report on ovendry basis.

Calcium Carbonate 6E

HCl Treatment 6E1

Vary sample weight according to $CaCO_3$ content as follows: For <25 percent $CaCO_3$, use 2 g soil; for 25 to 50 percent $CaCO_3$, 1 g soil; and for >50 percent $CaCO_3$, 0.5 g soil.

For trace amounts, add a few drops 6*N* HCl to soil and observe under binocular microscope. Evolution of gas bubbles indicates the presence of $CaCO_3$.

References

Williams (1948).

Manometer, electronic 6E1g

Apparatus

Manometer, electronic, digital, 0-1000 Torr.
Pressure sensor with thermal base.
Wide-mouth bottle, round, 120 ml (4 oz).
Machined cap for bottle, PVC, with self-sealing

<20-mm Basis **6E4**

Determine carbonate content of 2- to 20-mm fraction (1B5a) by one of the above methods. The carbonate in the 2- to 20-mm fraction and that in the <2-mm fraction (1B1a) are combined and converted to a <20-mm soil basis.

Calculations

Carbonate as $CaCO_3$ (pct.) = (A × B) + (C × (1–B))

where

A = $CaCO_3$ (pct.) in <2-mm material

B = wt material <20 mm / wt material 20 to 2 mm

wt material <20 mm

C = $CaCO_3$ (pct.) in 20- to 2-mm material

Note: Sample weights for B are obtained during sample preparation (1B1 and 3B1a).

Gypsum 6F

Water Extract 6F1

Precipitation in acetone 6F1a

Reagents

Acetone (CH_3COCH_3).

Procedure

Qualitative.—Pour approximately 5 ml saturation extract into a 15-ml conical centrifuge tube. Add about 5 ml acetone and invert the tube several times to mix the contents. Let stand for 30 min in a test-tube rack; run quantitatively all samples in which a precipitate forms, an indication of gypsum in the soil.

Quantitative.—Weigh 20 g air-dry soil into an 8-oz nursing bottle and add 100 ml water (1:5 ratio). If gypsum content is more than 10 meq per 100 g soil suspension on folded No. 12 Whatman 18.5-cm filter paper, using a 90-mm funnel. The first few milliliters of filtrate are usually cloudy and should be caught in a waste beaker and discarded. Pipet a 5-ml (or 20-ml) aliquot into a 15-ml (or 50-ml) conical centrifuge tube, add 5 ml (or 20 ml) acetone with a buret, stopper, and mix by inverting the tube. Let stand for at least 10 min to allow the precipitate to flocculate and centrifuge at 2,000 rpm for 5 min. Decant and discard the supernatant liquid. Invert and drain the tube on filter paper or toweling for about 5 min. Add 5 ml (or 10) acetone, replace stopper, and shake until precipitate disintegrates. Remove stopper and centrifuge at 2,000 rpm for 5 min. Decant and drain as before. Add 10 ml (or 40) water, stopper, and shake until precipitate dissolves. Measure electrical conductivity with a Solu-bridge, using a 2-ml-pipet conductivity cell.

Quantities in parentheses are those used in the laboratory at Riverside, Calif. Adjust instrument to temperature of solution and read concentration of $CaSO_4$ from standard curve.

This curve can be constructed by means of the following data from the International Critical Tables.

$CaSO_4$ concentration (meq/L):	Electrical conductivity at 25° C (mmhos/cm)
1	0.121
2	.226
5	.500
10	.900
20	1.584
30.5	2.205

Calculations

$CaSO_4$ in aliquot (meq) = $CaSO_4$ from curve (meq/L) × $\frac{H_2O \text{ to dissolve ppt (ml)}}{1{,}000}$

$CaSO_4$ in soil (meq/100 g) = $\frac{CaSO_4 \text{ in aliquot (meq)}}{\text{soil:water ratio}} \times \frac{100}{\text{aliquot (ml)}}$

References

Richards (1954).

Indirect estimate **6F1b**

Add a weighed quantity of soil to enough water to dissolve all the gypsum by overnight shaking. The concentration of sulfate in this dilute soil:water extract should be <10 meq/L. Gypsum can be estimated by method 6F2. If crystals are observed or estimated gypsum content is >5 percent, the <2-mm sample should be ground to approximately 80 mesh. Determine total sulfate in this extract by any appropriate procedure. Also determine Ca and SO_4 in a saturation extract by any appropriate procedure.

Calculations

$$\text{Gypsum} = (SO_4)_{DE} - (SO_4)_{\text{non-gypsum SE}}$$

$$\text{but } SO_{4\ \text{non-gypsum SE}} = (SO_4)_{SE} - (SO_4)_{\text{gypsum SE}}$$

$$\therefore \text{gypsum} = (SO_4)_{DE} + (SO_4)_{\text{gypsum SE}} - (SO_4)_{SE}$$

where

$(SO_4)_{DE}$ = SO_4 in dilute water extract

$(SO_4)_{SE}$ = SO_4 in saturation extract

$(SO_4)_{\text{gypsum SE}}$ = 30 meq/L if SO_4 and Ca are $\geq$ 30 meq/L

= $(SO_4)_{SE}$ if $(Ca)_{SE} > (SO_4)_{SE}$

= $(Ca)_{SE}$ if $(Ca)_{SE} < (SO_4)_{SE}$

Apparatus

DIONEX Model 2110i ion chromatograph
Recorder (1 volt input).
Voltage stabilizer.

Reagents

0.1*M* Na_2CO_3.
0.003*M* $NaHCO_3$.
0.0024*M* Na_2CO_3.
1*N* H_2SO_4.

Mixed standard solution

Fluoride 0.0125 to 5.0 meq/L.
Chloride 0.01 to 4.0 meq/L.
Nitrate 0.025 to 10.0 meq/L.
Sulfate 0.05 to 20.0 meq/L.

All solutions are filtered through a polycarbonate membrane having 0.4μm pore size. Soil extracts are filtered with a disposable filter unit (Millix™) having 0.22 μm pore size.

Procedure

The soil extract is obtained as described in 6F1a. Fill a plastic syringe (3 to 10 cc) with a solution having a concentration within the range of the sulfate standard. Baseline is established using a full-scale μmhos setting of 3 before each determination. This setting is adjusted as needed, keeping in the range used for making the determinations on the mixed standard. Peak height readings are made on the mixed standard using eight concentrations. A curve fitting linear regression equation [$y(\text{meq/L}) = a_1 (PKH) + a_0$] is established for the sulfate standards. Sulfate concentration in the soil extracts is determined by this equation.

Calculations

Aluminum dish.
Balance, 0.001-g sensitivity.

Reagents

Phosphorus pentoxide (P_2O_5).

Procedure

Place about 10 g of soil in a tared (Wt A) aluminum dish. Saturate sample with water and let stand overnight to air-dry. Place in a vacuum desiccator with P_2O_5 desiccant. Evacuate desiccator and allow to stand 48 hr. Remove dish from desiccator and weigh (Wt B), then place in oven at 105° C for 24 hr. Allow dish to cool in desiccator and weigh (Wt C).

Calculations

$$\text{Gypsum (pct.)} = \frac{(\text{Wt B} - \text{Wt C})\ (100)}{(\text{Wt B} - \text{Wt A})\ (0.1942)}$$

The theoretical crystal water content of gypsum is 20.91 percent. However, Nelson et al. have determined that, in practice, this content averages 19.42 percent.

References

Nelson, Klameth, and Nettleton (1978).

Correction for Crystal Water **6F3**

Soil properties are expressed on an ovendry-weight basis. Gypsiferous soils are a special case because gypsum ($CaSO_4 \cdot 2H_2O$) loses nearly all of its two molecules of water at 105° C.

Chemical properties of gypsiferous soils reported on an ovendry weight basis should be converted to include the weight of crystal water in gypsum. This can be done conveniently by converting the air-dry (AD) to ovendry (OD) ratio, used to convert chemical properties to an ovendry basis, to a crystal-water-containing basis (equation 1).

When reporting water content of gypsiferous soils, the crystal water content must be subtracted from the total ovendry water content (equation 2).

Calculations

(1) $\frac{\text{AD}}{\text{OD}}$, corrected basis = $\frac{\frac{\text{AD}}{\text{OD}}, \text{ uncorrected}}{1 + (\text{A} \times 0.001942)}$

(2) Water content, corrected basis =

$$\frac{\text{Percent total water} - (\text{A} \times 0.1942)}{1 + (\text{A} \times 0.001942)}$$

where

A = gypsum (pct.)

References

Nelson, Klameth, and Nettleton (1978).

<20-mm Basis **6F4**

Determine gypsum content by appropriate method on 2- to 20-mm fraction (1B5a). This gypsum and that determined on the fine earth (1B1a) are combined and converted to a <20-mm soil basis.

Calculations

Gypsum (pct.) = A × B + (C × (1 − B))

where

A = gypsum (pct.) in <2 mm material

$$\text{B} = \frac{\text{wt material <20 mm} - \text{wt material 20 to 2 mm}}{\text{wt material <20 mm}}$$

C = gypsum (pct.) in 20 to 2 mm material

Gypsum Requirement **6F5**

The amount of gypsum needed to replace all of the sodium on the exchange complex with calcium is the gypsum requirement.

Reagents

Saturated gypsum solution.—Place about 25 g gypsum ($CaSO_4 \cdot 2H_2O$) in 5 L water in a large flask, stopper, and shake by hand periodically for 1 hr or more. Let settle and decant through a filter into storage bottle. Determine calcium concentration by titration of an aliquot with standard EDTA solution using Eriochrome black T as indicator.

EDTA solution.—Dissolve 1.25 g di-sodium ethylenediamine tetraacetate in water and dilute to 1 L. Standardize against solutions containing known concentrations of Ca and Mg.

Buffer solution.—Dissolve 6.75 g ammonium chloride in about 400 ml water. Add 570 ml concentrated ammonium hydroxide and dilute to 1 L with distilled water.

Eriochrome black T indicator.—Dissolve 1 g Eriochrome black T in 100 ml triethanolamine.

Procedure

Weigh 5 g soil into flask, add 100 ml saturated gypsum solution, stopper, and shake for 5 min in mechanical shaker. Filter through folded filter paper, discarding the first few milliliters of filtrate, which may be cloudy.

Pipet a 5-ml aliquot of filtrate into a 125-ml Erlenmeyer flask and dilute to 25 or 30 ml with distilled water. Add 10 drops of buffer solution, 2 drops Eriochrome black T indicator, and titrate with standard EDTA solution to blue end point.

Calculations

Gypsum requirement (meq/100 g) =
[(Ca conc of gypsum soln (meq/L)) – (Ca + Mg conc of filtrate (meq/L))] × 2

References

Richards (1954).

Aluminum 6G

Dithionite-Citrate Extraction 6G7

Prepare extract as described in 6C2.

Atomic absorption 6G7a

Apparatus

Atomic absorption spectrophotometer.

Reagents

Standard Al solutions, 30, 60, 120 mg/8 oz.

Procedure

Measure absorbance of samples from 6C2 at 309.3 nm and compare with that of standard solutions.

Calculations

$$\text{Al (pct.)} = \frac{\text{Al (mg/8 oz)}}{\text{g sample}} \times \frac{\text{dilution}}{10}$$

Report on ovendry basis.

NH_4Cl, Automatic Extractor 6G8

Prepare extract as described in 5A9.

Atomic absorption 6G8a

Apparatus

Atomic absorption spectrophotometer.

Reagents

Standard Al solutions, 0 to 6 meq/L

Procedure

Compare absorbance of samples from 5A9 with that of standards at 309.3 nm, diluting if necessary.

Calculations

$$\text{Al (meq/100 g)} = \frac{\text{Al (meq/L)}}{\text{g sample}} \times$$

$$\text{dilution} \times \frac{\text{ml extract}}{10}$$

Report on ovendry basis.

KCl, Automatic Extractor 6G9

Equipment

Automatic extractor, 24 place.

60-ml plastic syringes and syringe barrels; use one

sample tube, one reservoir tube, and one tared extraction syringe.

Reagents

KCl solution, 1*N*.

Procedure

Prepare sample extraction tubes by forcing a 1-g ball of filter pulp into bottom of barrel with syringe plunger. Weigh 2.5 g soil into tube. Place sample tubes in extractor, attach tared extraction syringe, rinse sides of tube and stirring rod with KCl and fill to 20-ml mark. Attach reservoir tube and let stand 30 min.

Extract at a setting of 20 to 25 until a depth of 0.5 to 1.0 cm of KCl remains above sample Turn off extractor and add 45 ml KCl to the reservoir. Extract at a setting of 15 (approximately 40 min).

Remove and weigh syringes and reserve a portion of extract for determination of aluminum.

Atomic absorption **6G9a**

Equipment

Atomic absorption spectrophotometer.

Reagents

Standard Al solution, 0 to 6 meq/L.

Procedure

Measure absorption of samples at 309.3 nm and compare with that of standard solutions.

Calculations

Density factor for 1*N* KCl = 1.0412

$$\text{Al (meq/100 g)} = \frac{(\text{wt syringe + extract) (g) } - \text{ tare (g)}}{1041.2} \times \frac{100}{\text{sample wt (g)}} \times 0.1 \times \text{Al from curve (meq/L)} \times \text{dilution}$$

Report on ovendry basis.

Sodium Pyrophosphate Extraction **6G10**

Prepare extract as described in 6C8.

Atomic absorption **6G10a**

Apparatus

Atomic absorption spectrophotometer.

Reagents

Standard Al solutions, 0, 30, and 60 mg per 8 oz (237 ml).

Procedure

Measure absorbance of sample (from 6C8) at 309.3 nm and compare with absorption of standards prepared by pipetting 30 and 60 ml of 1,000 mg/L Al solution into 8-oz shaking bottle and carrying through extraction procedure as in 6C8.

Calculations

$$\text{Al (pct.)} = \frac{\text{Al (mg/8 oz)}}{\text{g sample}} \times \frac{\text{dilution}}{10}$$

Report on ovendry basis.

HF Dissolution **6G11**

Prepare extract as in 7C3.

Atomic absorption **6G11a**

Apparatus

Diluter.

Atomic absorption spectrophotometer.

Reagents

Standard Al solutions, 100, 200 mg/L.

Procedure

Dilute HF extracts from 7C3 and Al standards fivefold to twentyfold with water. Compare absorbance of samples with that of standards at 309.3 nm.

Calculations

$$\text{Al (pct.)} = \frac{\text{Al (mg/L)} \times 10 \times \text{dilution}}{\text{mg sample}}$$

$$Al_2O_3 \text{ (pct.)} = \text{Al (pct.)} \times 1.89$$

Report on ovendry basis.

Extractable Acidity 6H

$BaCl_2$—Triethanolamine III 6H4

Apparatus

60-ml plastic syringe barrels.

Reagents

Buffer solution.—Barium chloride, 0.5*N*, and triethanolamine, 0.2*N*. Add 1*N* HCl (about 90 ml/L) to adjust pH to 8.2. Protect the buffer solution from CO_2 of the air by attaching a drying tube containing soda lime (sodium calcium hydrate) to the air opening at the top of the solution bottle.

Replacement solution.—Barium chloride, 0.5*N*. Add 5 ml of above buffer solution per liter. Protect the replacement solution from CO_2 of the air by attaching a drying tube similar to that used for the buffer solution.

"Celite" filter pulp.

References

Peech (1947).

Back-titration with HCl, automatic titrator 6H4a

Reagents

Hydrochloric acid (HCl), 0.33*N*, standardized.

Procedure

Titrate the leachate contained in the 250-ml beaker to an end-point pH setting of 4.60 with automatic titrator. Carry reagent blank through procedure.

Calculations

$$\text{Extractable acidity (meq/100 g)} = \frac{\text{HCl blank (ml)} - \text{HCl sample (ml)}}{\text{g sample}} \times \text{normality of HCl} \times 100$$

Report on ovendry basis.

$BaCl_2$—Triethanolamine IV, Automatic Extractor 6H5

Apparatus

Automatic extractor, 24 place.

Syringes, 60 cc, polypropylene; use one sample tube and one extraction syringe per sample.

"Repipet" dispenser, 10 ml and 20 ml.

Sample diluter (optional).

Reagents

Procedure

Prepare sample extraction tubes by tightly compressing a 1-g ball of filter pulp into bottom of syringe barrel with plunger. Weigh 2.00 g soil sample into tube.

Place sample tube in upper disc of extractor and connect to inverted extraction syringe, the plunger of which is inserted in slot of stationary disc of extractor. Wash down sides of tube with small amount of water from wash bottle. Add a 10-ml aliquot of buffer solution with sample diluter, directing the stream of solution into sample for thorough mixing. Allow to stand for 30 min.

Set extractor for 30-min rate and extract until only a small volume of solution remains above sample. If necessary, shut off extractor to prevent soil from becoming dry. Add a second 10-ml aliquot of buffer solution and continue extracting until nearly all solution has been pulled through sample. Add replacement solution from sample diluter in two 20-ml aliquots, passing the first aliquot through the sample before adding the next. Total time for replacement should be close to 30 min.

References

Peech (1947).

Back-titration with HCl, automatic titrator **6H5a**

Reagents

Hydrochloric acid (HCl), 0.13*N*, standardized.

Procedure

Transfer extract to titrator beaker, add 100 ml distilled water and titrate with automatic titrator to an end-point pH setting of 4.60.

Establish a blank titer for each new batch of extraction reagents by taking the mean of titrations of 12 blanks, each containing 20 ml buffer solution and 40 ml replacement solution.

Calculations

$$\text{Extractable acidity (meq/100 g)} = \frac{\text{HCl blank (ml)} - \text{HCl sample (ml)}}{\text{g sample}} \times \text{normality of HCl} \times 100$$

Report on ovendry basis.

Carbonate 6I

Saturation Extract **6I1**

Prepare saturation extract as directed in 8A3.

Acid titration, automatic titrator **6I1b**

Apparatus

Titrator, automatic.
Titrator beakers, 250 ml.

Reagents

Sulfuric acid (H_2SO_4) 0.015*N*, standardized.
Phenolphthalein.

Procedure

Pipet 5 ml or other suitable aliquot of saturation extract from 8A3 into 250-ml titrator beaker and dilute with 70 ml boiled, distilled water. Add a drop of phenolphthalein. If pink color develops, set pH end point of titrator to 8.2 and titrate sample to this end point. Retain sample for measurement of bicarbonate.

Calculations

$$\text{Carbonate (meq/L)} = \frac{H_2SO_4 \text{ (ml)}}{\text{aliquot (ml)}} \times \text{normality of } H_2SO_4 \times 2{,}000$$

Bicarbonate 6J

Saturation Extract 6J1

Prepare saturation extract as directed in 8A3.

Auto sampler.
Recorder (1-volt input).
Anion analytical column HPIC-AS3 4×250.
Anion guard column HPIC-AG3 4×50.
Fiber anion supressor.
[illegible]

Acid titration, automatic titrator 6J1b

Apparatus

Titrator, automatic.
Titrator beakers, 250 ml.

Reagents

Sulfuric acid (H_2SO_4) 0.015*N*, standardized.

Procedure

Use solution remaining from carbonate titration (6J1b). Set pH end point to 4.01, and titrate sample to this end point. Titrate blanks of boiled distilled water to same end point.

Calculations

Bicarbonate (meq/L) =

[(ml H_2SO_4 + ml H_2SO_4 from 6I1b) – (blank) – (2 × ml H_2SO_4 from 6I1b)] ×

$$\frac{\text{normality of } H_2SO_4 \times 1{,}000}{\text{aliquot (ml)}}$$

Chloride 6K

Saturation Extract 6K1

Prepare saturation extract as directed in 8A3.

Chromatograph 6K1c

Apparatus

Dionex System 2110i chromatograph.
Advanced chromatography module.
Analytic pump.
[illegible]

Reagents

Sodium bicarbonate ($NaHCO_3$), 0.003*M*.
Sodium carbonate (Na_2CO_3), 0.0024*M*.
Sulfuric acid (H_2SO_4), 0.025*N*.

Standard solution concentration ranges:
Nitrite—0.05 to 0.07 .

Mixed standards—six standard solutions with the following four anions in each solution:

Fluoride—	
0.0125 to 1.875 meq/L	0.025 to 1.875 meq/L.
Chloride—	
.01 to 1.5 meq/L	.02 to 1.5 meq/L.
Nitrate—	
.03 to 4.5 meq/L	.06 to 4.5 meq/L.
Sulfate—	
.05 to 7.5 meq/L	.1 to 7.5 meq/L.

All solutions are made with distilled water which has been filtered through a 0.2–μ filter. Soil extracts are filtered.

Procedure

The saturation extract is obtained from the saturated paste (8A3), is filtered, and its conductance is measured. The estimated sum of cations is calculated from the conductance using the relationship of meq/L saturation extract = $10^{(1.08805 \times \log EC + 1)}$. If chloride is the expected dominant anion, a dilution is selected so the resulting concentration will be <0.3 meq/L chloride. If sulfate is the expected dominant anion, a dilution is selected so the resulting concentration will be <5 meq/L sulfate. Dilutions are made using the eluent solution as the diluent. Fill a plastic syringe (3 to

A baseline is established using a full-scale micromho setting of 3 before each determination. This setting is adjusted as needed, keeping within the range used for making the determinations on the mixed standard. Peak height readings are made on the mixed standard using selected concentrations. A curve fitting the linear regression equation (y (meq/L) = a_1 (peak heights) + a_o) is established for each ion, r_2 = >0.98.

A qualitative (NO_2-NO_3) test is made by placing 1 drop of the sample and 4 drops of diphenylamine solution on a spot plate. The amount present is indicated by the intensity of the blue color. This independent test is an aid in the identification of peaks. If NO_2 is present in any sample, this standard is run and its curve-fitting linear regression equation established. These values are reported as NO_3 + NO_2 meq/L.

Calculations

[redacted]

a_1 (peak height) + a_0 where a_1 = slope and a_0 = intercept.

Sulfate 6L

Saturation Extract 6L1

Prepare saturation extract as directed in 8A3.

Chromatograph 6L1c

Determine sulfate as described in 6K1c.

Nitrate 6M

Saturation Extract 6M1

Prepare saturation extract as directed in 8A3.

Atomic absorption 6N1b

Apparatus

Atomic absorption spectrophotometer.
Diluter.

Reagents

Standard Ca solutions, 0 to 30 meq/L.

Lanthanum stock solution, 65,000 ppm. Slowly dissolve 76.2 g La_2O_3 in 300 ml 6*N* HCl and dilute to 1 L. Dilute stock solution 1:10 with water for use as diluent when diluting sample solutions and standards.

Procedure

Prepare standards and sample extracts from 8A3 with 0.65 percent (6,500 ppm) lanthanum. Measure absorbance of samples at 422.7 nm and compare with [redacted]

Calculations

Ca (meq/L) = Ca (meq/L) × dilution

NH_4OAc Extraction 6N2

Obtain extract as in 5A8.

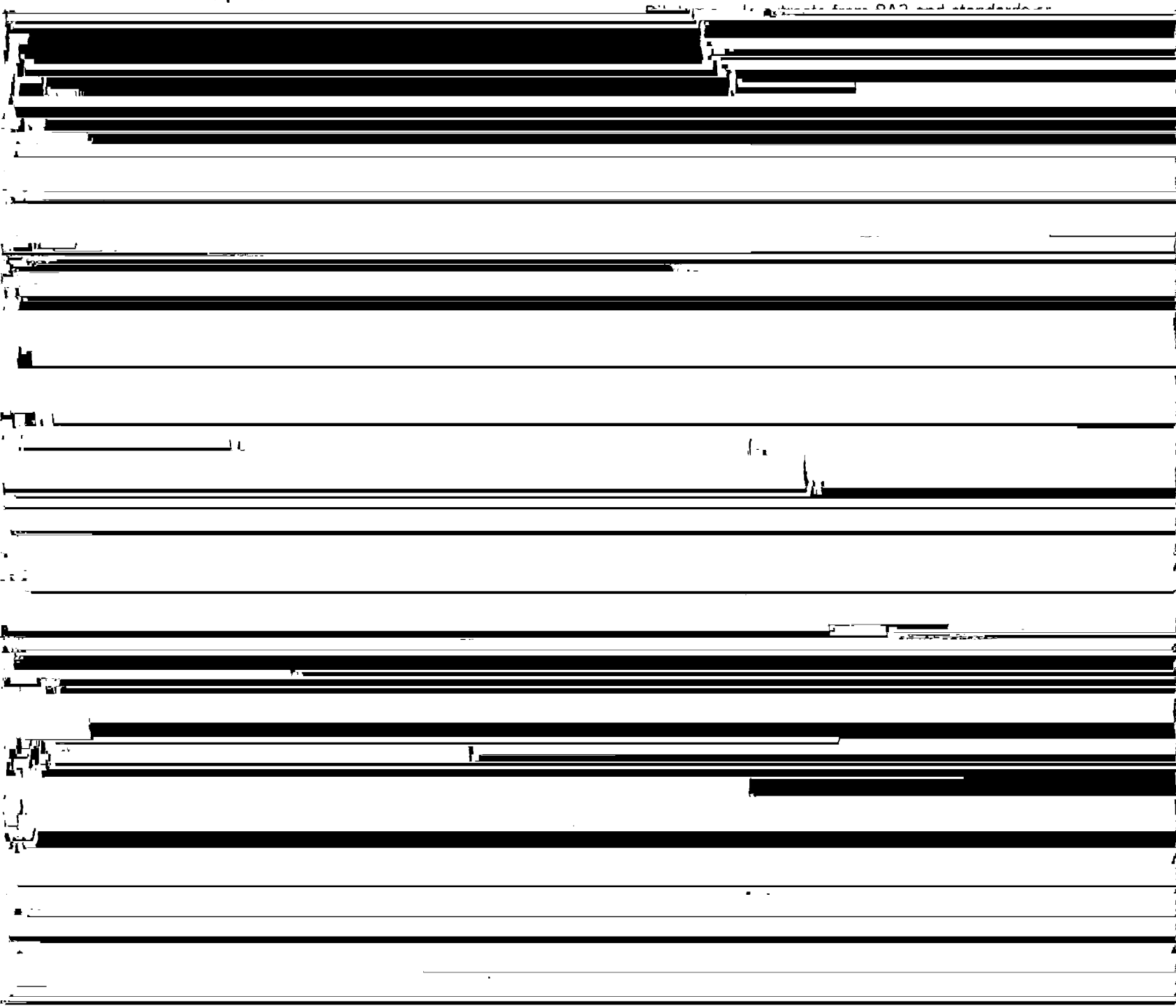
Atomic absorption 6N2e

Procedure

Proceed as in 6N1b except use samples from NH_4OAc extract, method 5A8.

Calculations

$$\text{Ca (meq/100 g)} = \frac{\text{Ca (meq/L)}}{\text{g sample}} \times \frac{\text{ml extract}}{}$$



Diluter.
Atomic absorption spectrophotometer.

Reagents
Standard Ca solutions, 0 to 30 meq/L.

Procedure
Dilute HF extracts from 7C3 and Ca standards

absorbance of samples at 285.2 nm and compare with that of standards.

Calculations
Mg (meq/L) = Mg (meq/L) × dilution

NH_4OAc Extraction **602**
Obtain extract as in 5A8

Procedure

Dilute HF extracts from 7C3 and Mg standards fivefold to twentyfold with water. Compare absorbance of samples with that of standards at 285.2 nm.

Calculations

Mg (pct.) =

$$\frac{\text{Mg (meq/L)} \times 10 \times \text{dilution} \times 12.16\ \text{(mg/meq)}}{\text{mg sample}}$$

$$\text{MgO (pct.)} = \text{Mg (pct.)} \times 1.66$$

Report on ovendry basis.

Sodium 6P

Saturation Extract 6P1

Prepare saturation extract as described in 8A3.

Atomic absorption 6P1b

Apparatus

Atomic absorption spectrophotometer.
Diluter.

Reagents

Standard Na solutions, 0 to 20 meq/L in water.

Procedure

Dilute samples from 8A3 and standards and compare absorption of samples at 589 nm with that of standard solutions.

Calculations

Na (meq/L) = Na (meq/L) × dilution

NH_4OAc Extraction 6P2

Obtain extract as in 5A8.

Atomic absorption 6P2b

Proceed as in 6P1b except use samples from NH_4OAc extract, method 5A8.

Calculations

$$\text{Na (meq/100 g)} = \frac{\text{Na(meq/L)}}{\text{g sample}} \times \text{dilution} \times \frac{\text{ml extract}}{10}$$

Report on ovendry basis.

HF Dissolution 6P3

Obtain extract as in 7C3.

Atomic absorption 6P3a

Apparatus

Diluter.
Atomic absorption spectrophotometer.

Reagents

Standard Na solutions, 0 to 20 meq/L in HF and boric acid.

Procedure

Dilute HF extracts from 7C3 and Na standards fivefold to twentyfold with water. Compare absorbance of samples with that of standards at 589 nm.

Calculations

Na (pct.) =

$$\frac{\text{Na (meq/L)} \times 10 \times \text{dilution} \times 23.00\ \text{(mg/meq)}}{\text{mg sample}}$$

$$Na_2O\ \text{(pct.)} = \text{Na (pct.)} \times 1.35$$

Report on ovendry basis.

Potassium 6Q

Saturation Extract 6Q1

Prepare saturation extract as described in 8A3.

Atomic absorption **6Q1b**

Apparatus
Atomic absorption spectrophotometer.
Diluter.

Reagents
Standard K solutions. 0 to 5 meq/L in water.

Procedure
Dilute sample extracts from 8A3 and standards, if necessary, and compare absorption of samples at 766.5 nm with that of standard solutions.

Calculations
K (meq/L) = K (meq/L) × dilution

Atomic absorption **6Q3a**

Apparatus
Atomic absorption spectrophotometer.
Diluter.

Reagents
Standard K solutions. 0 to 5 meq/L in HF and boric acid.

Procedure
Dilute HF extracts from 7C3 and K standards fivefold to twentyfold with water. Compare absorbance of samples at 766.5 nm with that of standards.

Calculations

K (pct.) =

Reagents

Potassium iodate (KIO_3).
Potassium iodide (KI).
Arrowroot starch.
Hydrochloric acid (HCl) 7.7*N*.
Hydrochloric acid (HCl) 0.18*N*.
Magnesium oxide (MgO).

Copper metal accelerator.

Procedure

Into a tared crucible, weigh approximately ½ g of 60-mesh soil, recording gross weight. Where high sulfur content might be present, either ¼ or ⅒ g sample should be run. Add 2 scoops of MgO and a scoop of iron chips. Mix thoroughly. Add a half scoop of copper accelerator and a scoop of iron chips. Magnesium oxide scoops are heaping; all others are level. A cover is placed on the crucible, which is placed on the pedestal and raised into the combustion tube for ignition. The LECO instruction manual is followed in setting up the furnace and titrator. The timer is set to 8 min and grid tap switch to midposition. These settings should be adjusted as needed to get complete fusion of the mixture in the

Phosphorus 6S

Adsorption Coefficient 6S2

Apparatus

Syringes, 60 cc polypropylene; use one sample tube and one extraction syringe per sample.

Reagents

Extractant.—Dissolve 4.5 g ammonium fluoride (NH_4F) and 85.6 g ammonium chloride (NH_4Cl) in about 4 L of distilled water, add 92 ml glacial acetic acid and 10 ml concentrated HCl, make to 8 L and mix.

Sulfuric-molybdate-tartrate solution.—Dissolve 100 g ammonium molybdate [$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$] and 2.425 g antimony potassium tartrate [$K(SbO)C_4H_4O_6 \cdot ½H_2O$] in 500 ml distilled water, heating if necessary but not to exceed 60° C. Slowly add 1,400 ml concentrated H_2SO_4 and mix well. Cool, dilute to 2 L with water and store in refrigerator in polyethelene or Pyrex bottle.

Apparatus

Colorimeter.
Automatic extractor.
Shaker.

Procedure

A. Saturation

Weigh three 2-g subsamples of ovendried soil into 50-ml Erlenmeyer flasks.

To the first add 2 ml distilled water.

To the second add 2 ml 20 ppm P solution.

To the third add 2 ml 80 ppm P solution.

Let stand for 1 hr then place in oven at 60° C and dry overnight.

B. Extraction

To each of the dried samples in the 50-ml Erlenmeyer flasks, add 20 ml extractant reagent, and shake for 20 min (Burrell shaker). Extract samples using the automatic extractor.

C. Developing the color

Standard curve—Using 50-ml Erlenmeyer flasks, pipette aliquots from the phosphorus working standards as follows:

Flask 1—2 ml extractant
Flask 2—2 ml 2 ppm P
Flask 3—2 ml 4 ppm P
Flask 4—2 ml 6 ppm P
Flask 5—2 ml 8 ppm P
Flask 6—2 ml 10 ppm P

Samples—For each sample extracted in part B, pipette 2 ml of extract into clean 50-ml Erlenmeyer flasks corresponding with sample numbers.

To all flasks, standards, and samples, add 25 ml of color solution, swirl to mix, and let stand for 15 min to allow color to develop. After color has developed fully, transfer to colorimeter tubes.

D. Reading the color

Using a wavelength setting of 880 μm, set colorimeter to 100 percent transmittance with No. 1 standard containing 2 ml extractant. Read percent transmittance of remaining standards and samples.

Generally, the standard curve is around the following values:

0	2	4	6	8	10	ppm
100	77	59	45	33	24	%t

(t = transmittance).

E. Calculations

1. Develop standard curve by the least squares analysis using concentration of standards as a f(ln%t).

This results in the equation:

concentration = m(ln%t) + b

2. Use this equation to determine solution concentrations of unknowns (leachate).

Concentration of leachate × 10 is desorbed P in ppm of dry soil.

3. P retained of that added = P added − (desorbed P at that conc. minus desorbed P at zero P addition).

4. Pα (adsorption coefficient) is the slope of the least square regression of P retained as a function of phosphorus added, f(P added).

Example

0	2	4	6	8	10	ppm—standard concentrations
100	77	59	45	33	24	%t
			0	20	80	P added (ppm)
			84	73	45	%t

1. Concentration = −7.023(ln%t) + 32.5158
2. Concentration =

−7.023 (ln84) + 32.5158 = 1.40
(ln73) 2.38
(ln45) 5.78

Desorbed P (ppm) of dry soil = 1.40 × 10 = 14.0
= 2.38 × 10 = 23.8
= 5.78 × 10 = 57.8

3. P (ppm) retained of that added =
   0 − (14.0 − 14.0) = 0
   20 − (23.8 − 14.0) = 10.2
   80 − (57.8 − 14.0) = 36.2

4. y = 0.4478(P added) + 0.5083
   $P\alpha$ = 0.4478

References
Mehlich (1978).

Boron 6T

Saturation Extract 6T1

Carmine colorimetry 6T1a

Refer to USDA Handbook 60, method 17 (p. 100) and method 73b (p. 142).

Fluoride 6U

Saturation Extract 6U1
Prepare saturation extract as directed in 8A3.

Ion chromatograph 6U1a
Determine fluoride as described in 6K1c.

Silicon 6V

HF Dissolution 6V1
Obtain extract as in 7C3.

Atomic absorption 6V1a

Apparatus
Diluter.
Atomic absorption spectrophotometer.

Reagents
Standard Si solutions, 400 and 800 mg/L in HF.

Procedure
Dilute HF extracts from 7C3 and Si standards fivefold to twentyfold with water. Compare absorbance of samples with that of standards at 252 nm.

Calculations

$$\text{Si (pct)} = \frac{\text{Si (mg/L)} \times 10 \times \text{dilution}}{\text{mg sample}}$$

$$SiO_2 \text{ (pct)} = \text{(pct)} \times 2.14$$

Report on ovendry basis.

Instrumental Analyses 7A

The following instruments are used at the NSSL for mineralogical analyses:

(1) Philips Automated Powder Diffractometer System (APD 3501) interfaced with a Texas Instruments dual cassette electronic data terminal (TI-733). Philips manual X-ray diffraction unit.

(2) Columbia Scientific Industries Differential Thermal Analysis System Model 200, comprising furnace platform Model P-202 and recorder-controller cabinet Model RC-202G.

(3) Columbia Scientific Industries Thermogravimetric Analysis Instrument Model 1000B used with recorder-controller unit in (2).

(4) Perkin-Elmer Infrared Spectrophotometer Model 283.

X-ray Diffraction 7A2

Clay suspensions are dried as thin films so that the plates are parallel to one another (preferred orientation). This results in greater X-ray diffraction peak intensities for the diagnostic 001 spacings. Identification and semiquantitative estimation of nonplaty minerals such as quartz, feldspars, and crystalline iron and aluminum oxides can be made with randomly oriented dry powder samples. Refer to SSIR No. 1 (1972) for discussion of interpretation of results.

Thin film on glass, resin pretreatment II 7A2i

Apparatus

Glass slides 27 × 46 mm or 14 × 19 mm.
Hypodermic syringes (plastic, 10 cc).
80-mesh screen.
International No. 2 centrifuge with a No. 240 head.
100-ml centrifuge tubes (plastic).

Reagents

Potassium chloride (KCl), 1*N*.
Magnesium chloride ($MgCl_2$), 1*N*.
Glycerol-water mixture (1:8 glycerol-water).
Cation exchange resin, Rexyn 101 (H)[8]

Procedure

Preparation of Mg and K resin syringes.—Place a small circle of 80-mesh screen in syringe and add 3 cc of exchange resin. Saturate with Mg or K by drawing 2 ml of the appropriate 1*N* salt solution ($MgCl_2$ or KCl) into the syringe. After equilibration, expel the salt solution. Repeat twice. Finally, wash three times with 5 to 8 ml of distilled water to remove excess salt solution by drawing water into the syringe, rinsing it around, and expelling the solution.

Preparation of sample slides.—Place a teaspoon of <2-mm air-dry soil into a 100-ml centrifuge tube, add 5 ml of sodium metaphosphate solution and bring to a total volume of 50 ml with distilled water. Shake overnight. Centrifuge for 3.0 min at 750 rpm for a 10-cm suspension depth and decant to separate the clay, which is Na saturated. Draw 1 cc of clay suspension into the resin-loaded syringe. Expel approximately 0.2 cc clay suspension, containing about 13 mg clay in a 20- × 27-mm band across the middle of the 47- × 27-mm slide, or expel approximately 0.1 cc of clay suspension containing approximately 6 mg clay onto and covering a 14- × 19-mm slide. Prior to the deposition of the clay suspension, one [a single] small drop of glycerol-water mixture is placed on the slide to solvate the sample. Prepare four slides for X-ray diffraction: 1) Mg^{++}—*room* temperature, 2) Mg^{++}—solvated, 3) K^{+}—heated 2 hr at 300° C, and 4) K^{+}—heated 2 hr at 500° C.

Draw distilled water into the syringe and expel three times to remove all of the clay suspension.

After the tenth sample, the resin should be recharged with the appropriate ion as described in "preparation of Mg and K resin syringes."

Use the Na-saturated clay for DTA (7A3), IR (7A5), or HF (7C3) analyses.

Thin film on glass, $NaPO_3$ pretreatment II 7A2j

Apparatus

Hypodermic syringe (1.0 cc).
Glass slides 24 × 46 mm or 14 × 19 mm.
International No. 2 centrifuge with a No. 240 head.
100-ml centrifuge tubes (plastic).

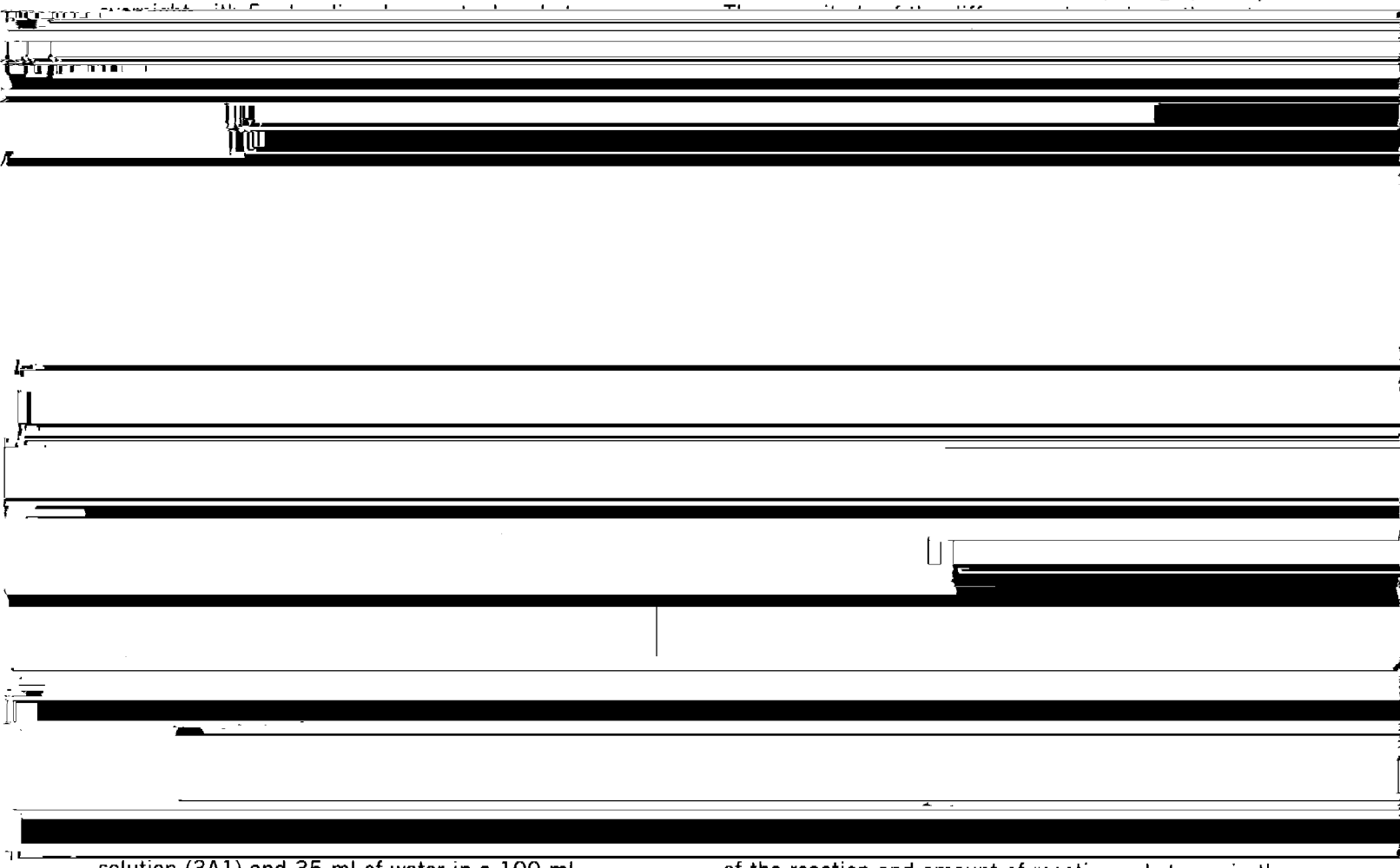
[8] *Fisher Scientific Company, Pittsburgh, PA 15219.*

Reagents

Glycerol-water mixture (1:8 glycerol-water).
Sodium hexametaphosphate solutions.

Procedure

Shake approximately 5 g ovendried soil (<2 mm)



solution (3A1) and 35 ml of water in a 100-ml centrifuge tube, centrifuge at 750 rpm for 3 min for a 10-cm suspension depth, and decant clays. Draw about 0.5 cc of clay suspension into the syringe. Expel approximately 0.2 cc of the clay suspension onto an area approximately 20 × 27 mm in a band across the middle of a 46- × 27-mm slide or expel approximately 0.1 cc of clay suspension, containing approximately 6 mg of clay, onto and covering the 14- × 19-mm slide. Prior to the deposition of the clay suspension, one small drop of glycerol-water mixture is placed on the slide which is to be solvated. Prepare four slides for X-ray diffraction: 1) Na^+—room temperature, 2) Na^+—solvated, 3) Na^+—heated 2 hr at 300° C, and 4) Na^+—heated for 2 hr at 500° C.

sample of soil material and a thermally inert material as the two are heated simultaneously at a constant rate. Thermocouples are in contact with two platinum pans; one pan contains an unknown and the other pan contains an inert material of similar composition. If a reaction occurs, a difference in temperature is registered on a stripchart recorder or photographically.

of the reaction and amount of reacting substance in the unknown. The temperature at which the reaction occurs identifies the substance if enough is known about the sample to predict the possibilities.

Apparatus

Columbia scientific instrument (CSI) system 200.
Mortar and pestle.
Analytical balance.
Desiccator.

Reagents

Reference sample—calcined kaolinite, 2 to 20 μ.
Ethyl alcohol, 95 percent.
Magnesium nitrate ($Mg(NO_3)_2 \cdot 6H_2O$.

Endotherms at about 120° C indicate surface-adsorbed water. Montmorillonite produces a double peak at a low temperature if saturated with a divalent cation. The proportion of this mineral can be estimated if samples are kept in an atmosphere with a high (70 to 80 percent) relative humidity for 24 hr or more before analysis. Allophane has a broad endotherm at about 160° C.

Samples can be any well-powdered material—whole soil or separated fractions. Organic matter is objectionable because it produces irregular exothermic reactions that obscure the important peaks. If a clay separate is used, it must be washed free of hygroscopic salts or salts containing water of crystallization.

References

Grim (1968), McKenzie (1957), and Tan and Hajek in Dixon and Weed (1977).

Thermal Gravimetric Analysis 7A4

Thermal gravimetric analysis is the detection and measurement of weight changes in a sample of soil material as the sample is being heated or cooled over a specific temperature range.

Apparatus

CSI Stone Model 1000B used in conjunction with an RC-202 recorder-controller. Furnace is water cooled, with a rapid cooling Kanthal element. Furnace is

Reagents

Potassium bromide, spectroscopic grade.

Apparatus

Infrared spectrometer—Perkin Elmer Model 283.
Pellet die.
Hydraulic press.
Analytical balance.

Procedure

Mix 0.30 g KBr and 1 mg of sample in mortar and pestle. Transfer the mixture to the pellet die, and place die in hydraulic press. Apply 8 tons of pressure for 1 min. Place pellet in instrument holder and scan for 12 min. Peaks produced on chart recorder are used to identify the substance.

References

J. L. White in Dixon and Weed (1977).

Optical Analyses 7B

Grain Studies 7B1

Sample selection and mounting

For most

acetylene tetrabromide or bromoform. These liquids can be diluted with nitrobenzene, toluene, or other appropriate organic solvents to make separations in other density ranges, or to concentrate minerals such as mica or calcic plagioclase. A liquid with specific gravity of about 2.5 is useful to concentrate volcanic glass, plant opal, or sponge spicules. Specific gravity alone is usually adequate to separate grains larger than 0.10 mm in separatory funnels or various kinds of tubes. Separation of smaller grains requires centrifuging. A pointed, 15-ml centrifuge tube is generally satisfactory. A glass rod with a smooth bulb on the end can be used to close the end of the tube and the light minerals are poured off. Alternatively, the contents of the lower part of the tube can be frozen to keep the heavy minerals in place.

Micas are difficult to separate because of their shape and because a little weathering, especially in biotite, decreases their specific gravity measurably. It is possible to use the differences in density to concentrate weathered biotite in various stages of alteration.

To be effectively separated by heavy liquids, the grains must be clean. Organic matter may prevent wetting and cause grains to clump or raft together. Light coatings may cause heavy grains to float, and iron-oxide coatings may increase specific gravity. In some kinds of material, it is best to separate and weigh the magnetic fraction, either before or after the heavy-liquid separation. Wrapping a thin sheet of flexible plastic around the magnet helps make this separation quantitative.

Analysis and interpretation

First survey the slide under a low-power objective to become familiar with the grain assemblage and to make a rough estimate of the relative abundance of minerals and other grains. Identify the most abundant minerals first; they probably are the easiest to identify, and their elimination decreases the number of possibilities to consider when trying to identify the difficult ones. A preliminary survey also gives clues to the minor species that can be expected. One ultimately learns to identify minerals by combining familiarity with a few striking features and the process of elimination. The identification procedures used are described in standard references on sedimentary petrography.

For many purposes, listing the minerals is enough. It is easy to accompany such a list with an estimate of their relative abundance. To get volume percentages, minerals can be counted on arbitrarily or regularly spaced traverses. If the grains are large or sparsely distributed, all grains should be counted. If there are many small grains, all grains passed by a quadrant or all grains touched by a cross-hair intersect should be counted. If there are only a few species, counting 100 to 300 grains gives a good idea of the composition. As the number of species increases, the count should increase. It is seldom necessary to count more than 1,000 grains, and in most work 500 to 600 is enough.

It is often important to record the morphology and condition of the different grains in the sand and silt fractions. Wear during transport shows as rounding, especially in chemically resistant minerals that do not have good cleavage, such as quartz. Rounding can be observed nicely in crossed polarized light. A grain can have a round outline and still be a flat plate. If it is truly round, the interference colors rise smoothly, without steps or interruption, from low order at the periphery to high order in the thickest part.

Manifestations of weathering range from slight bleaching of color or slight lowering of the refractive index to replacement of one mineral by another or complete removal of a species. Weathering in single grains is observed best in at least two mounting media. A medium whose refractive index closely matches that of a grain enables one to see the interior of the grain well and shows up contrasting coatings. A medium whose refractive index is a few hundredths of a unit away from that of the grain shows the condition of the grain surfaces.

Minerals modified by weathering, secondary minerals, aggregate grains, and other grains not identifiable as specific minerals are common in many soils and may help to determine the important characteristics of those soils. These minerals and grains should be described accurately even if they cannot be identified. The following paragraphs illustrate some kinds of aggregates that may be present in sand and silt fractions. The aggregates are in several categories of significance. Some are nuisances but must be accounted for, and others have very real and important diagnostic value.

Rock fragments include chips of shale, slate, schist, and fine-grained igneous rocks such as rhyolite. Their identification depends on the recognition of structure and individual components.

Clay aggregates may be present in many forms. Silt and sand bound together into larger grains by a nearly isotropic brownish material usually indicate faulty dispersion. Clay skins may resist dispersion and appear as fragments in grain mounts. Such fragments are

usually brown or red and translucent and show wavy extinction bands; they have been mistaken for weathered biotite. Clay aggregates may be mineral pseudomorphs; e.g., kaolin is a pseudomorph of feldspar and montmorillonite aggregates are pseudomorphs of basic rock minerals. Montmorillonite in this form shows high birefringence and its extinction is mottled or patchy on a small scale. Coarse kaolinite flakes, books, and vermicular aggregates resist dispersion and may be abundant in sand and silt. Such materials may resemble muscovite, which is cloudy, shows no definite extinction, and has very low birefringence.

Allophane occurs in many soils that formed from volcanic ash. It seldom can be identified directly, but it can be inferred if sand and silt are cemented into aggregates by isotropic material that has a low refractive index, and especially if volcanic glass shards are present.

Opal is another isotropic material. It occurs in separate grains, as a cementing material, and in organic forms (plant opal, sponge spicules, diatoms). Its refractive index is very low (<1.45), lower than that of volcanic ash. Identification may depend in part on its form and occurrence.

Iron oxides may occur separately or as cementing agents and in mixtures with other minerals. They impart brown and red colors and raise the refractive index of the mixtures. Goethite is yellow to bright red. The refractive index and birefringence are higher in the red varieties, which seem to be more crystallized. Goethite often has a prismatic or fibrous habit, and aggregates have parallel extinction. In the more or less oriented aggregates, the interference colors often have a greenish cast. Hematite has a higher refractive index than goethite and in granular rather than prismatic

Glauconite occurs in the form of an aggregate of small micaceous grains that have high birefringence. If fresh, it is dark green and almost opaque, but it weathers to a brown and more translucent form. It is difficult to identify glauconite by optical evidence alone.

Titanium oxide aggregates have been tentatively identified in the heavy mineral separates of many soils. They have high refractive indices and birefringence, and their yellow to gray colors are similar to those of anatase. They are granular and rough surfaced, which suggests that they are secondary.

References

Cady (1965), Fry (1933), Krumbein and Pettijohn (1938), and Milner (1962).

Grain mounts, epoxy **7B1a**

Reagents

Petropoxy 154 resin.

Petropoxy curing agent.

Procedures

Heat hot plate to 125° C. Mix small amount of Petropoxy 154 resin and curing agent (ratio of 10:1) in plastic beaker provided with the reagents. Heat for 4 min at 125° C. Cool mixture to room temperature. Add resin mixture to glass slide, add specimen grains, stir to mix uniformly, put cover glass in place, and heat for 10 min at 125° C (yields index of refraction of

index of 1.68, which is close to that of many of the common heavy minerals, is best for mounting them.

Total Analysis 7C

HF Dissolution 7C3

Apparatus

Digestion vessel (Parr No. 4745 general purpose) consisting of stainless steel retainer with 25-ml Teflon cup and cover.
5-ml pipette.
2-ml pipette.
100-ml nalgene volumetric.
25- or 50-ml polyethylene container with cover.
Teflon stirring rod.

Reagents

Hydrofluoric acid (48 percent HF).
Boric acid.

Procedure

Surface Area 7D

Ethylene Glycol Monoethyl Ether (EGME) Retention 7D2

Apparatus

Analytical balance.
Vacuum desiccator.
Laboratory suction (0.65 to 0.75 bar).
EGME trap (anhydrous $CaCl_2$ in drying tube).
Syringe, plastic, 1 cc.

Reagents

Ethylene glycol monoethyl ether (EGME).
Phosphorus pentoxide (P_2O_5).
Calcium chloride ($CaCl_2$).

Procedure

Dry 3 to 5 g of <2-mm air-dried soil in a weighing dish for 2 days over P_2O_5 in a vacuum desiccator. Weigh the P_2O_5-dried sample. Saturate the soil with EGME using a 1-cc syringe and add 5 drops in excess. Place the soil-EGME mixture over anhydrous $CaCl_2$ (4 to 8 mesh) in a vacuum desiccator. Connect the

Calculated surface area (m^2/g) =

$$\frac{\text{Retention of EGME (mg/g)}}{0.286}$$

References

Heilman, Carter, and Gonzalez (1965).

Saturated Paste, Mixed 8A

Apparatus

Sixteen-ounce container with lid, such as a cottage cheese carton.

Procedure

Prepare saturated soil paste by adding water to a sample of soil and stirring with a spatula. From time to time tap the container on a workbench to consolidate the soil-water mixture. At saturation the soil paste glistens as it reflects light, flows slightly when the container is tipped, and slides freely and cleanly off the spatula except for soils containing much clay. Allow the sample to stand overnight, and then recheck the preceding criteria for saturation. Free water should not collect on the soil surface nor should the paste stiffen markedly or lose its glistening appearance on standing. If this occurs, remix with more water.

Soils puddle most readily when worked at a moisture content near field capacity. Since minimum puddling is desired, add enough water initially to bring the sample nearly to saturation. If the paste is too wet, add more dry soil.

Size of the soil sample depends on the number of determinations to be made, i.e., on the volume of extract desired. A 250-g sample is convenient to handle and provides enough extract for most purposes. Mixing is generally easier if the soil is first air-dried and passed through a 2-mm sieve.

Special precautions must be taken for peat and muck soils and very fine-textured soils. Dry peat and muck soils, especially if coarse textured or woody, require overnight wetting to get a definitive end point for the saturated paste. After the first wetting, pastes of these soils usually stiffen and lose their glisten on standing. Adding water and remixing usually gives a mixture that retains the characteristics of a saturated paste. To minimize puddling and thus obtain a more definite end point for fine-textured soils, add water with a minimum of stirring, especially in the early stages.

Ovendry a subsample overnight at 105° C to determine moisture at saturation.

References

Richards (1954).

Saturation Extract, Automatic Extractor — 8A3

Apparatus

Automatic extractor.
Filter funnels.
60-cc syringes.
Fine pore membrane filters, disposable.
Extract containers, 1-oz, disposable.

Procedure

Transfer the saturated soil paste to plastic filter funnel fitted with 9-cm filter paper. Place funnel on extractor. Attach syringe and extract at setting of about 20 to 25. Remove syringe from extractor, attach disposable filter unit to tip of syringe and filter extract into 1-oz container. Refer to methods 6I1b, 6J1b, 6K1c, 6L1c, 6M1c, 6N1b, 6O1b, 6P1b, 6Q1b, 6V1a for chemical analyses usually performed on the saturation extract.

References

Holmgren, Juve, and Geschwender (1977).

Conductivity, digital bridge — 8A3a

Apparatus.

Markson conductivity-analyzer, digital readout with temperature-compensating conductivity cell.

Reagents

KCl solution, 0.1*N*. Dissolve 0.7456 g dry KCl in water and make to 1 L at 25° C. This solution has an electrical conductivity at 25° C of 1,412 μmhos/cm.

Procedure

Calibrate conductivity analyzer with standard KCl solution. Read conductivity of unknown solutions directly in micromhos. No temperature compensation is necessary.

Reaction (pH) — 8C

Soil Suspensions — 8C1

Saturated paste — 8C1b

Apparatus

pH meter.

Procedure

Immerse electrodes in the saturated paste prepared by method 8A.

NaF — 8C1d

Apparatus

pH meter.
Stopwatch or watch with sweep second hand.

Reagents

Sodium fluoride (NaF) 1*N* (saturated). Add 1,000 ml water to 50 g NaF in a 1-L plastic bottle. Let stand for 2 days but shake occasionally. On the third day, after excess NaF has settled, measure 50 ml of the solution into a beaker. The pH should be between 7.2 and 8.1. Add 3 to 5 drops 0.25-percent phenolphthalein and titrate with 0.01*N* NaOH to a pink end point (pH 8.2 to 8.3). If the solution has a pH of more than 8.2 or if the titratable acidity exceeds 0.25 meq/L, try another source of NaF.

Procedure

Place 1 g air-dry soil in a 100-ml beaker. Add 50 ml 1*N* NaF. Record the time. Stir the suspension for 1 min. Place the glass and reference electrodes in the upper third of the suspension. Stir for 1 min and read the pH exactly 2 min after adding the NaF solution.

References

Fields and Parrott (1966).

Water dilution and $CaCl_2$, automated system 8C1f

Apparatus

Brinkman automatic pH unit with sample changer and Radio Shack Model IV microcomputer.

Paper cold drink cups, 4 oz.

Disposable coffee stirrers.

Titrator beakers, plastic, 250 ml.

20-L bottles.

Reagents

Calcium chloride ($CaCl_2$), 0.02*M*.

pH buffers (pH 4.1, 7.0).

Procedure

Weigh 20 g soil into 4-oz paper cups. Add 20 ml distilled water, stir, place cup with sample in 250-ml plastic beakers, and let stand 1 hr, stirring occasionally. Load beakers into 4-place sample magazines and arrange on sample changer table. Calibrate pH meter with fresh buffer solutions, following procedure in instruction manual.

Enter total number of samples to be run and the sample numbers through the keyboard of the computer. Start pH unit and collect measured values on a floppy disk and printer. Sample stirring, the waiting interval for readings, addition of $CaCl_2$ solution, and rinsing are controlled by the computer. The 0.02*M* $CaCl_2$ is dispensed from a 20-L bottle as is the distilled water for rinsing the electrode and stirrer. By adding an equal volume of 0.02*M* $CaCl_2$ to the soil suspension prepared for the water pH, the soil-solution ratio will be 1:2 at

Organic Materials 8C2

$CaCl_2$ 8C2a

Procedure

Place 2.5 cc of the sample prepared by method 8G1 in a 30-ml plastic container and add 4 ml of 0.015*M* $CaCl_2$ (gives final concentration of approximately 0.01*M* $CaCl_2$ with most packed moist organic materials). Mix, cover, and allow to equilibrate at least 1 hr. Uncover, immerse electrodes, and measure pH.

References

Soil Survey Staff (1975).

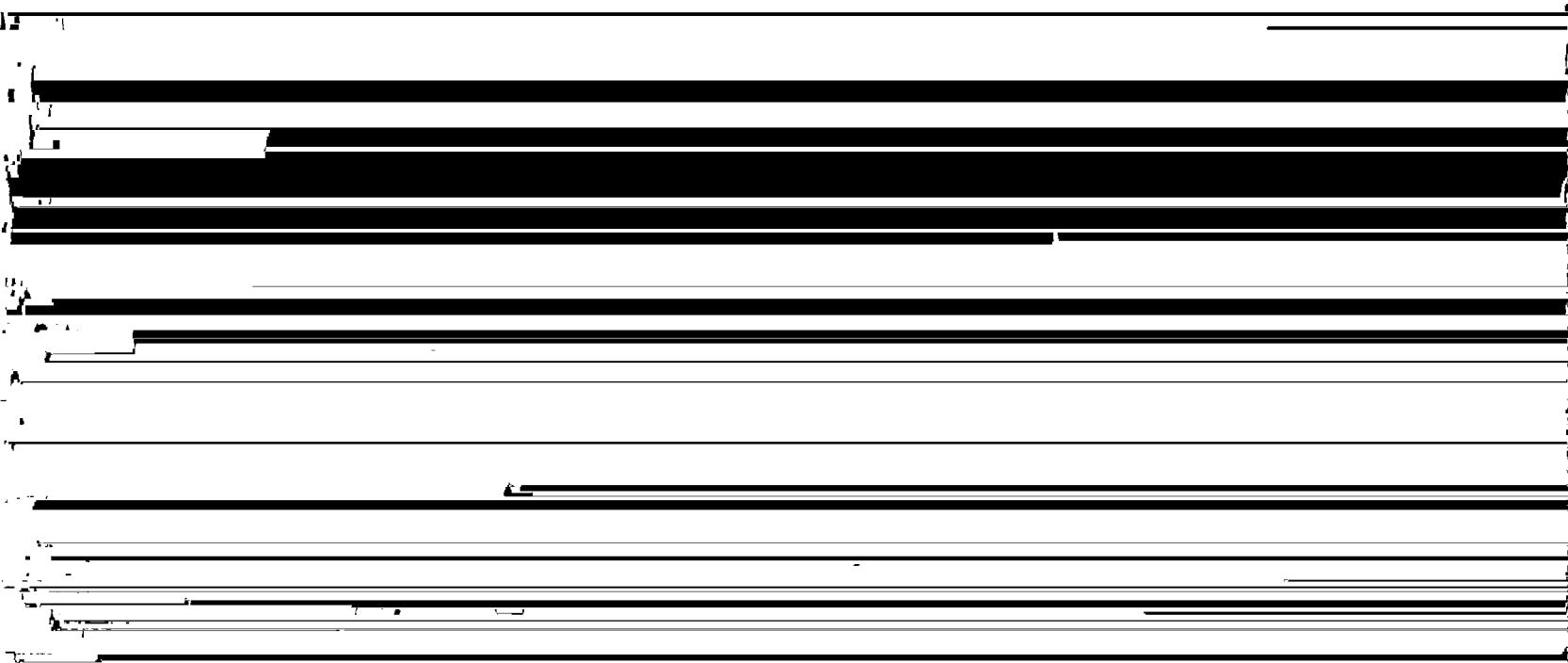
Ratios and Estimates 8D

To Total Clay 8D1

Divide cation-exchange capacity (CEC-7), extractable iron, 15-bar water retention, or other measurements by the total clay percentage. In the past, these ratios have been reported as milliequivalents per gram of clay (CEC) or grams per gram of clay (iron, water).

To Noncarbonate Clay 8D2

Divide cation-exchange capacity (CEC-7), extractable iron, or 15-bar water retention by the noncarbonate clay percentage determined by subtracting the



Estimated Total Salt **8D5**

Estimate total salt from the conductivity of the saturation extract by using charts and graphs available in Richards (1954). The essential relations are summarized in the following equations:

log total salt in soil (ppm) = 0.81 + 1.08 log EC(mmhos/cm) (method 8A3a) + log saturation percentage (method 8A)

total salt in soil (pct) = total salt (ppm) $\times 10^{-4}$

The equations work well for saturation extracts with conductivity less than 20 mmhos/cm, deviations occur at higher salt concentrations.

References

Richards (1954).

Iron Plus Aluminum—Pyrophosphate Extractable to [illegible] **8D6**

Divide the sum of the pyrophosphate-extractable iron plus aluminum by the sum of dithionite-citrate-extractable iron plus aluminum. A resulting ratio of 0.5 or greater is needed to meet one of the chemical criteria for spodic placement.

References

Soil Survey Staff (1975).

Index of Accumulation (Spodic Horizons) **8D7**

Subtract one-half the clay percentage of a subhorizon

Apparatus

Wheatstone bridge.

Electrode cup.

Procedure

Fill cup with soil paste, jar to remove air bubbles, and strike off excess paste so that cup is level full. Connect to bridge, record resistance (ohms), and measure temperature of paste (°C).

Calculations

Convert measured resistance in ohms to resistivity (ohms-cm) at 60° F as follows:

$$\text{Resistivity (ohms-cm)} = \frac{1{,}000}{\text{Coef}} \times R \times F$$

where

$$\text{Coef} = 5{,}137.82 - (215.73 \times \text{Temp}) + [4.6089 \times (\text{Temp})^2] - [0.046583 \times (\text{Temp})^3] + [0.000177 \times (\text{Temp})^4]$$

Coef 60° F = 1,018.052

R = resistance in ohms

$$F = \text{cell factor} \left(\frac{1}{\text{cell constant}} \right)$$

References

Romanoff (1957).

temperature to 400° C. Heat overnight (16 hr), cool, and weigh.

Calculations

Mineral content (pct.) =

$$\frac{\text{wt of residue after ashing}}{\text{wt of ovendry soil}} \times 100$$

Fiber Volume 8G

Water Dispersed 8G1

Apparatus

Half-syringe—a 6-ml plastic syringe cut longitudinally to form a half cylinder measuring device.
100-mesh sieve, 3-in diameter.
Eggbeater.
Microscope.
Electric mixer, Hamilton Beach No. 35.

Procedure

Sample preparation.—If the soil is dry, add water and let stand to saturate. Place 50 to 60 cc of a representative sample on a paper towel in a linear mound. Roll the towel around the sample and express water if necessary. Use additional paper towels as external blotters. Remove the sample and place on a fresh paper towel. The sample should be firm but saturated with water. Cut with scissors into segments 0.5 to 1 cm long. Use a random selection of segments for determination of fiber, solubility in pyrophosphate, and pH.

Unrubbed fiber.—Procedure involves a series of three steps designed to remove sapric[9] material by increasingly vigorous treatments. The percentage of sapric material remaining is estimated visually under a microscope or hand lens following each step. Estimate sapric component that remains within the following categories:

1. Clean (<1 percent sapric).
2. Nearly clean (1 to 10 percent sapric).
3. Some sapric (10 to 30 percent sapric).
4. Sapric (>30 percent sapric).

Step 1. Pack a half-syringe adjusted to 5-cc capacity (2.5-cc volume) level with the moist sample. Transfer all the soil material to a 100-mesh sieve and wash under a stream of cold tap water, adjusted to deliver 200 to 300 ml in 5 s, until the water passing through the sieve appears clean. Examine the sample under a microscope or hand lens to determine if it is free of sapric material. If the sample contains more than 10 percent sapric, go to step 2. If not, wash the residue to one side of the screen and blot from underneath with absorbent tissue to withdraw water. Repack the residue into a half-syringe and blot again with absorbent tissue. The moisture content should approximate that of the orginial sample. Measure the volume by inserting the plunger until the material fills the remaining half-syringe barrel, and record it as a percentage of the initial 2.5-cc volume. Proceed with the rubbed fiber determination.

Step 2. Transfer the residue obtained in step 1 to a 500-ml plastic container and fill about half full with water. Stir vigorously with an eggbeater for 1 min. Transfer to the 100-mesh sieve and repeat procedures in step 1. If sapric material remains, go to step 3.

Step 3. Transfer residue left from step 2 to an electric mixer container (malt mixer or blender) and fill about two-thirds full with water. Mix for 1 min. Transfer to a 100-mesh sieve and repeat step-1 washing procedure. Examine the residue under a microscope or hand lens and determine the percentage of sapric material, if any. Blot the sample and measure the residue volume. Proceed with the rubbed fiber determination.

Rubbed fiber.—Transfer the residue from the unrubbed fiber treatment to the 100-mesh sieve. Rub sample between thumb and fingers under a stream of cold tap water, adjusted to deliver 150 to 200 ml in 5 s, until water passing through the sieve is clean. Clean rubbed fibers will roll between the thumb and fingers rather than slide or smear. Blot sample and measure volume in half-syringe.

Calculations

Fiber volume (pct.) = reading on half-syringe (cc) × 20.

[9] *Sapric materials are the most decomposed class of organic materials defined in Soil Taxonomy. Sapric material passes through a 100-mesh sieve (0.15-mm openings). Fibers are retained on the sieve.*

References

Soil Survey Staff (1975).

Pyrophosphate Color 8H

Apparatus

Poly-Con container, 30-ml.

Chromatographic paper, Schleicher and Schuell No. 470 A-3.

Munsell Color Book, 10YR and 7.5YR pages required.

Reagents

Sodium pyrophosphate ($Na_4P_2O_7 \cdot 10H_2O$), ground to 2 mm.

Procedure

Dissolve 1 g (heaping ⅛ tsp) pyrophosphate in 4 ml water in a 30-ml poly-con. Allow to equilibrate for 5 min. Prepare soil material and moisten half syringe as described in 8G1. Transfer soil material cleanly into the container that holds the pyrophosphate solution. Mix thoroughly using a wooden coffee stirrer. Cover and let stand overnight.

Mix again next morning. Insert strip of chromatographic paper vertically about 1 cm with tweezers. Let stand until the paper test strip has wetted 2 cm above the slurry surface (about 5 min but may stand longer if cover is closed). Remove the paper test strip with tweezers and tear off and discard the portion to which soil adheres. Place the strip on a piece of blotting paper and press gently with tweezers to make even contact.

Remove paper strip with tweezers and compare color on side next to blotting paper with Munsell color charts.

Diluter.

Reagents

Potassium chloride (KCl), 0.0100*N*.

Procedure

Weigh 3 g of <2-mm soil into a 10- to 30-ml volume container with a lid. Add 6 ml of distilled water, mix, cap, and allow to equilibrate overnight. The KCl solution is used to standardize the analyzer to give a reading of 1,412 µmhos. Conductance of the supernatent solution is read directly. Samples with a conductance of <250 µmhos are considered as nonsalty and no further salt analyses are made.

LITERATURE CITED

Allison, L. E. 1960. Wet combustion apparatus and procedure for organic and inorganic carbon in soil. Soil Sci. Soc. Amer. Proc. 24:36-40.

American Society for Testing and Materials. 1970. Book of ASTM standards, pp. 212-219.

Association of Official Agricultural Chemists. 1945. Official and tentative methods of analysis, 6th ed. 932 pp. Washington, D.C.

Bascomb, C. L. 1968. Distribution of pyrophosphate-extractable iron and organic carbon in soils of various groups. J. Soil Sci. 19:251-268.

Berdanier, C. R., W. C. Lynn, and G. W. Threlkeld. 1978. Illitic mineralogy in Soil Taxonomy: X-ray vs. total potassium. Agronomy Abstracts 1978 Annual Meetings. 197 pp.

Brasher, B. R., D. P. Franzmeier, V. T. Valassis, and S. E. Davidson. 1966. Use of Saran resin to coat natural soil clods for bulk-density and water-retention measurements. Soil Sci. 101:108.

Brown, G., ed. 1961. The X-ray identification and crystal structures of clay minerals. 345 pp. Mineralogical Society (Clay Minerals Group), London.

Holmgren, George G. S. 1967. A rapid citrate-dithionite extractable iron procedure. Soil Sci. Soc. Amer. Proc. 31:210-211.

Holmgren, George G. S. 1968. Nomographic calculation of linear extensibility in soils containing coarse fragments. Soil Sci. Soc. Amer. Proc. 32:568-570.

Holmgren, George G. S., R. L. Juve, and R. C. Geschwender. 1977. A mechanically controlled variable rate leaching device. Soil Sci. Soc. Amer. J. 41:1207-1208.

Innes, R. P., and D. J. Pluth. 1970. Thin section preparation using an epoxy impregnation for petrographic and electron microprobe analysis. Soil Sci. Soc. Amer. Proc. 34:483-485.

Jackson, M. L. 1956. Soil chemical analysis, advanced course. 991 pp. Privately published. Madison, Wisconsin.

Kilmer, V. J., and L. T. Alexander. 1949. Methods of making mechanical analyses of soils. Soil Sci. 68:15-24.

Kilmer, V. J., and L. F. Mullins. 1954. Improved

[redacted]

Cady, J. G. 1965. Petrographic microscope techniques. *In* Methods of soil analysis, Monog. 9. v. 2, pp. 604-631. Amer. Soc. Agron., Madison, Wis.

Dixon, J. B. and S. B. Weed. (ed.). 1977. Minerals in soil environments. Soil Sci. Soc. of Amer. Madison, Wisconsin.

Fields, M., and K. W. Parrott. 1966. The nature of allophane in soils. Part 3. Rapid field and laboratory test for allophane. N.Z.J. Sci. 9:623-629.

Franzmeier, D. P., and S. J. Ross, Jr. 1968. Soil swelling: Laboratory measurement and relation to other soil properties. Soil Sci. Soc. Amer. Proc. 32:573-577.

stirring and pipetting apparatus for mechanical analysis for soils. Soil Sci. 77:437-441.

Krumbein, W. C., and F. J. Pettijohn. 1938. Manual of sedimentary petrography. 549 pp. D. Appleton-Century Co., New York.

Lagerwerff, J. V., G. W. Akin, and S. W. Moses. 1965. Detection and determination of gypsum in soils. Soil Sci. Soc. Amer. Proc. 29:535-540.

Lin, C., and N. T. Coleman. 1960. The measurement of exchangeable aluminum in soils and clays. Soil Sci. Soc. Amer. Proc. 24:444-446.

Lockwood, W. N. 1950. Impregnating sandstone specimens with thermosetting plastics for studies of

Reed, F. S., and J. L. Mergner. 1953. The preparation of rock thin sections. Amer. Miner. 38:1184-1203.
Richards, L. A., ed. 1954. Diagnosis and improvement of saline and alkali soils. U.S. Salinity Laboratory, U.S. Dept. Agr. Hbk. 60, 160 pp.
Rogers, A. F., and P. F. Kerr. 1933. Thin-section mineralogy. McGraw-Hill Book Co., New York.
Romanoff, Melvin. 1957. Underground corrosion. Nat. Bur. Stds. Cir. 579, 227 pp.
Schoonover, W. R. 1952. Examination of soils for alkali. University of California Extension Service. Berkeley, California. (mimeographed)
Schuman, G. E., M. A. Stanley, and D. Knudsen. 1973. Automated total nitrogen analysis of soil and plant samples. Soil Sci. Soc. Amer. Proc. 37:480-481.
Shields, L. G., and M. W. Meyer. 1964. Carbonate clay: measurement and relationship to clay distribution and cation exchange capacity. Soil Sci. Soc. Amer. Proc. 28:416-419.
Smith, R. M., W. E. Grube, Jr., T. Arkle, Jr., and A. Sobek. 1974. Mine spoil potentials for soil and water quality. EPA Environmental Protection Technology Series. 670/2-74-070:43-48.
Soil Survey Staff. 1975. Soil Taxonomy. U.S. Dept. Agr. Hbk. 436, 754 pp.
Switzer, G., J. M. Axelrod, M. L. Lindberg, and E. S. Larson. 1948. Tables of d spacings for angle 2Θ. U.S. Geol. Survey Cir. 29.
Tabatabai, M. A., and J. M. Bremner. 1970. Use of the Leco 70-second carbon analyzer for total carbon analysis of soils. Soil Sci. Soc. Amer. Proc. 34:608-610.
Tyner, E. H. 1939. The use of sodium metaphosphate for dispersion of soils for mechanical analysis. Soil Sci. Soc. Amer. Proc. 4:106-113.
Walkley, A. 1935. An examination of methods for determining organic carbon and nitrogen in soils. J. Agr. Sci. 25:598-609.
Williams, D. E. 1948. A rapid manometric method for the determination of carbonate in soils. Soil Sci. Soc. Amer. Proc. 13:127-129.
Young, K. K., and J. D. Dixon. 1966. Overestimation of water content at field capacity from sieved-sample data. Soil Sci. 101:104-107.