

Soil Survey Investigations Report No. 1

**SOIL SURVEY
LABORATORY METHODS AND
PROCEDURES FOR
COLLECTING SOIL SAMPLES**

Soil Conservation Service
Washington, D.C.

U.S. Department of Agriculture
Revised April 1972

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PREFACE

This publication is the first of a new U.S. Department of Agriculture series—Soil Survey Investigation Reports. This series was established to make available technical information from cooperative laboratory and field investigations of soils of the United States, Puerto Rico, and the Virgin Islands. Many volumes will include only soil descriptions and physical, chemical, and mineralogical data from the soil survey laboratories. Others will be reports of completed studies of soil genesis.

The laboratory data in succeeding volumes in this series have been collected for a variety of purposes for about two decades. They have been distributed in unpublished form to those immediately concerned with specific problems. Some have appeared in technical journals, regional bulletins, USDA technical bulletins, and in published soil surveys. But because most of them were not published, they have been unavailable to many. We intend to publish in this series all data from the soil survey laboratories that form reasonably complete characterizations of soils. Fragmentary data collected as reference points for specific soil surveys will not be included.

There were several reasons for sampling these soils. Some were sampled to study soil genesis, some to facilitate classification, and some to obtain data to permit more useful interpretations. The soils sampled for genesis or classification studies do not always fit neatly into our present concepts of soil series. Partly because of these studies, our concepts of some soil series have changed. As a consequence, the name assigned a soil series at the time of sampling is not always the name that would be assigned today. In these publications the soil series names are being changed to follow the current series definitions.

Ways of describing soils were changing while these data were being assembled. Soil descriptions have become explicit on more and more features. The systems for designating horizons and for classifying soils have been changed. The soil descriptions being published were written at the time the samples were collected. The soil scientists who wrote them had no idea that they would be published; they prepared them as working documents to meet a specific need of a soil survey. Field textual estimates have been retained, even though sometimes at variance with the laboratory data, because the field estimates themselves are important data.

While these data were being assembled, there were many changes in laboratory methods. Consequently, laboratory data for different soils cannot be directly compared without allowance for the methods used to obtain them. This publication describes methods that have been used in the soil survey laboratories and procedures for sampling soils. Not all the methods described are in current use because methods are changed as our knowledge changes.

"Soil Survey Laboratory Methods and Procedures for Collecting Soil Samples" - Soil Survey Investigations Report No. 1

Errata

- Code pages are reversed.
- Method 4F2. Should read "Plastic limit"
- Method 8A2. Should read "Bureau of Soils cup, resistance"
- Page 21. Method 4F. Plasticity index Should read "The plasticity index is the difference between the plastic limit and the liquid limit."
- Method 4F2. Should read "Plastic limit"
- Page 27. Method 6A2a. Organic carbon, dry combustion Calculations: Should read "Report on oven-dry basis as in 6A1b."
- Page 29. Method 6A2b. Organic carbon, dry combustion Calculations: Should read "Report on oven-dry basis as in 6A1b."
- Page 58. Method 8A2. Should read "Bureau of Soils cup, resistance" Calculations: Should read "Convert to resistance at 60° F by referring to table 2. Use this value and table 9, p. 349, Soil Survey Manual. to estimate percentage of soluble salt."

- Page 60. Method 8E1. Soil resistivity, saturated paste Should read "Multiply resistance at 60° F (method 8A2) by the cell constant (approximately 4 for Bureau of Soils cup) to convert to resistivity, ohms per centimeter."

Addendum

- Page 24. NH_4Cl 5A7
- Procedure
- Proceed as in 5A6 except substitute 1N NH_4Cl for pH 7 NH_4OAc .
- Direct distillation 5A7a
- Determine ammonia by Kjeldahl distillation as described in 5A1a.

INTRODUCTION

The methods and procedures described in this publication are those in current use or that have been used in the soil survey laboratories of the Soil Conservation Service. 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 wish to read more about the theory or technique of a method. Therefore, references to the literature are included.

In subsequent volumes of this series, headings for columns of laboratory data include a symbol that refers to a method described in this volume, which is identified on a code sheet to be included in each volume. This code sheet (fig. 1) may give enough information to someone who wants only a general idea of the method used. For some methods it is necessary to refer to several sections to learn the complete procedure. An example of how the symbols are used is a data sheet for Hayter silt loam (fig. 2), which indicates that oven-dry bulk density was determined by a method having the symbol 4A1h. The code sheet shows that 4A1h describes the calculation of bulk density of oven-dry clods coated with Saran. Anyone wishing to learn more of the details of this method and preparation of the clods must read sections 4A and 4A1 as well as the brief statement in 4A1h.

Figure 3 is the profile description of Hayter silt loam that accompanies the data sheet. It illustrates some of the volume estimates of materials of different size made in the field, which are discussed in section 3B.

Values reported on the data sheets are either primary or derived. A primary value is the result of direct calculations for a particular determination from which the derived values can be calculated. For example, carbon percentage and nitrogen percentage are primary values; the carbon-nitrogen ratio is derived by dividing carbon percentage by nitrogen percentage.

How other derived values reported for the B1 horizon (5 to 10 inches) of Hayter silt loam were calculated is shown in the following list, which includes references (in parentheses) to the appropriate sections of this publication.

Values related to bulk density:

Primary values:

Moist bulk density	
(D_b_m)	1.35 g per cubic centimeter (4A1e)

Dry bulk density (D_b_s)	1.39 g per cubic centimeter (4A1h)
Particle density (D_p)	2.65 g per cubic centimeter (assumed)
Volume (pct.) of >75-mm material based on unit volume of whole soil (x)	25 percent or 0.25 (3B2)
Weight (pct.) of 2-mm to 75-mm material based on unit weight of <75-mm material (y)	22+4 percent or 0.26 (3B1)
Water content at 1/2-bar tension (W_1)	26.8 percent (4B1c)
Water content at 15-bar tension (W_{15})	11.5 percent (4B2)
Coarse fragment conversion factor (cm)	0.64 (3B2)

Derived values:

Volume (pct.) of >2mm material of whole soil

$$\begin{aligned}
 >2 = 100 \left[1 - \frac{D_p(1-y)(1-x)}{D_p(1-y) + D_b_m(y)} \right] \\
 &= 100 \left[1 - \frac{2.65(0.74)(0.75)}{2.65(0.74) + 1.35(0.26)} \right] \\
 &= 36 \text{ percent} \quad (3B2)
 \end{aligned}$$

Weight (pct.) of >2mm material of whole soil

$$\begin{aligned}
 >2 = \frac{100 D_p(x)}{D_p(x) + D_b_m(1-x)} \\
 &= \frac{100(2.65)(0.36)}{2.65(0.36) + 1.35(1-0.36)} \\
 &= 52 \text{ percent} \quad (3B1)
 \end{aligned}$$

Linear extensibility (LE)

$$\begin{aligned}
 LE(\text{pct.}) &= 100 \left[\left(\frac{1}{C_m \left(\frac{D_b_m}{D_b_s} \right) + (1-C_m)} \right)^t - 1 \right] \\
 &= 100 \left[\left(\frac{1}{0.64 \left(\frac{1.35}{1.39} \right) + (1-0.64)} \right)^t - 1 \right] \\
 &= 0.6 \quad (4D1)
 \end{aligned}$$

Water-retention difference (WRD)

$$\begin{aligned}
 WRD &= \frac{(W_1 - W_{15})(D_b_m)C_m}{100} \\
 &= \frac{(26.8 - 11.5)(1.35)(0.64)}{100} \\
 &= 0.13 \quad (4C1)
 \end{aligned}$$

Cation-exchange capacity and base saturation:

Primary values:

Extractable calcium	1.6 meq per 100 g (6N2d)
Extractable magnesium	1.1 meq per 100 g (6O2b)
Extractable sodium	0.1 meq per 100 g (6F2a)
Extractable potassium	0.2 meq per 100 g (6Q2a)
Extractable acidity	10.8 meq per 100 g (6H2a)

6. CHEMICAL ANALYSES (cont.)

- d. Weight gain
- e. Titrimetric
- f. Warburg method
- 2. Sensitive qualitative method
 - a. Visual, gas bubbles
- 3. H_2SO_4 treatment
 - a. Weight gain
- F. Gypsum
 - 1. Water extract
 - a. Precipitation in acetone
 - b. Indirect estimate
- 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
 - a. Atomic absorption
 - 6. Ammonium oxalate extraction
 - a. Atomic absorption
 - 7. Dithionite-citrate extraction
 - a. Atomic absorption
- H. Extractable acidity
 - 1. $BaCl_2$ -triethanolamine I
 - a. Back-titration with HCl
 - 2. $BaCl_2$ -triethanolamine II
 - a. Back-titration with HCl
 - 3. KCl-triethanolamine
 - a. Back-titration with NaOH
- I. Carbonate
 - 1. Saturation extract
 - a. Acid titration
- J. Bicarbonate
 - 1. Saturation extract
 - a. Acid titration
- K. Chloride
 - 1. Saturation extract

6. CHEMICAL ANALYSES (cont.)

- 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
- F. Sodium
 - 1. Saturation extract
 - a. Flame photometry
 - b. Atomic absorption
 - 2. NH_4OAc extraction
 - a. Flame photometry
 - b. Atomic absorption
- Q. Potassium
 - 1. Saturation extract
 - a. Flame photometry
 - b. Atomic absorption
 - 2. NH_4OAc extraction
 - a. Flame photometry
 - b. Atomic absorption
- R. Sulfur
 - 1. $NaHCO_3$ extract, pH 8.5
 - a. Methylene blue
 - 2. HCl release (sulfide)
 - a. Iodine titration
 - 3. Total phosphorus
 - 1. Perchloric acid digestion
 - a. Molybdovanadophosphoric acid colorimetry
- 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
 - c. Thin film on glass, $NaPO_3$ pretreatment
 - d. Thin film on tile, solution pretreatment
 - e. Thin film on tile, resin pretreatment

[The body of the page is almost entirely obscured by heavy black redaction bars and horizontal scanning artifacts. Only a few faint, illegible characters are visible through the noise.]

SOIL Hayter silt loam SOIL Nos. 863Ky-74-6 LOCATION McCreary County, Kentucky
SOIL SURVEY LABORATORY Beltsville, Maryland LAB. Nos. 63776 - 63781

Depth (in.)	Horizon	3A1											3B2 >2 Vol pct. of whole soil	3B1			
		1B1b				Size class and particle diameter (mm)								Coarse fragments 2-20 Pct. of whole soil	20-75 Pct. of whole soil		
		Total				Sand			Silt								
Sand (2-0.05) (0.05- 0.002)	Silt (0.05- 0.002)	Clay (= 0.002)	Very coarse (2-1)	Coarse (1-0.5)	Medium (0.5-0.25)	Fine (0.25-0.1)	Very fine (0.1-0.05)	0.05-0.02	Int. III (0.02- 0.002)	Int. II (0.2-0.02)	(2-0.1)						
1/2-5	Ap2	15.0	59.0	26.0	3.8	1.9	0.6	1.6	7.1	15.7	43.3	23.9	7.9	23	36	21	-
5-10	B1	15.0	57.4	27.6	3.0	1.9	.6	1.1	8.4	14.6	42.8	23.7	6.6	36	52	22	4
10-19	B21	15.6	57.0	27.4	3.4	1.9	.6	1.5	8.2	14.7	42.3	24.0	7.4	34	47	17	2
19-34	B22t	14.3	57.0	28.7	3.4	2.1	.8	1.2	6.8	13.4	43.6	20.9	7.5	26	37	12	-
34-48	B23t	15.9	53.7	30.4	4.8	2.9	1.0	1.6	5.6	9.2	44.5	15.7	10.3	47	59	16	18
48-60	B3t	24.1	50.8	25.1	8.1	4.9	1.7	2.8	6.6	9.5	41.3	17.8	17.5	51	62	27	7
Depth (in.)	6A1a Organic carbon Pct.	6B2a Nitrogen Pct.	C/N	Carbonate as CaCO ₃ Pct.	6C1a Ext. iron as Fe Pct.	Bulk density			Water content			6C1 WRD in %	pH				
						6A1b 1/3 bar g/cc	6A1h Oven dry g/cc	4D1 LE Pct.	6B1c 1/3 bar Pct.	6B2 15 bar Pct.	6C1 WRD in %		6C1c (1:1) KCl 1N.	6C1a (1:1) H ₂ O			
1/2-5	1.86	0.180	10		2.9	1.36	1.39	0.7	26.6	12.0	0.15		4.4	4.9			
5-10	.87	.100	9		2.9	1.35	1.39	0.6	26.8	11.5	.13		3.9	4.6			
10-19	.35	.058	6		2.3	1.54	1.61	0.9	21.6	11.1	.11		3.9	5.4			
19-34	.21				2.4	1.57	1.63	0.9	21.5	12.0	.11		3.9	5.4			
34-48	.22				2.5	1.60	1.63	0.3	21.7	12.4	.08		3.8	5.4			
48-60	.10				2.9	1.68	1.75	0.6	18.4	10.1	.07		3.8	5.3			
Depth (in.)	Extractable bases				6E2a Ext. acid- ity	5A3a Sum cat- ions	6G1d Al	Base saturation									
	6D2d Ca	6D2b Mg	6F2a Na	6G2a K				5C3 Sum cat- ions Pct.	Pct.								
1/2-5	4.3	1.5	0.1	0.5	12.8	19.2	0.4		33								
5-10	2.6	1.1	.1	.2	10.8	13.8	1.5		22								
10-19	1.8	1.6	.1	.2	7.8	11.5	.7		32								
19-34	1.4	2.4	.1	.2	7.8	11.9	1.0		34								
34-48	1.5	3.2	Tr.	.2	7.6	12.5	1.2		38								
48-60	1.1	3.0	Tr.	.2	6.9	13.0	1.2		33								

FIGURE 2.—Laboratory data sheet for Hayter silt loam, McCreary County, Ky.

Soil Type: Hayter silt loam
 Soil No.: S63Ky-74-6
 Location: McCreary County, Kentucky, North off Hwy. 759 about 2 miles east of U. S. Hwy. 27.
 Vegetation and land use: Hickory, persimmon, yellow poplar.
 Slope and land form: 50 percent.
 Drainage: Well drained.
 Parent Material: Colluvium from sandstone and shale.
 Sampled by and date: D. P. Franzmeier, E. J. Pedersen, C. R. Gass, L. Manhart, G. Chapman, October 15, 1963.
 Described by: J. H. Winsor, C. K. Losche.

Horizon and
 Beltsville
 Lab. No.

O1	1-1/2 to 0 inches. Hardwood leaf litter.
Ap1	0 to 1/2 inch. Very dark grayish brown (10YR 3/2) silt loam; moderate fine granular structure; very friable; 12 percent sandstone fragments (>3 in. diameter); many roots; pH 7.0.
Ap2 63776	1/2 to 5 inches. Brown (10YR 4/3) silt loam; weak medium granular structure; very friable; 12 percent sandstone fragments; many roots; pH 5.0.
B1 63777	5 to 10 inches. Brown (7.5YR 4/4) silt loam; weak to moderate fine subangular blocky structure; friable; 25 percent sandstone fragments; many roots; pH 5.0.
B21 63778	10 to 19 inches. Brown (7.5YR 4/4) silt clay loam/silt loam; moderate medium blocky structure; friable; 25 percent sandstone fragments; common roots; pH 5.0.
B22t 63779	19 to 34 inches. Brown to dark brown (7.5YR 4/4-3/2) silty clay loam; moderate medium blocky structure; friable; common clay films; 20 percent sandstone fragments; few roots; pH 5.0.
B23t 63780	34 to 48 inches. Brown (7.5YR 4/4) silty clay loam; moderate medium subangular blocky structure; friable to firm; 30 percent sandstone fragments; common clay films; few roots; pH 5.0.
B3t 63781	48 to 60 inches. Brown (7.5YR 5/4) silty clay loam; weak to moderate medium subangular blocky structure; friable to firm; common clay films; 35 percent sandstone fragments; few roots; pH 5.0.

Notes: Colors given are for moist soil. The B21 and B23t layers were sampled for the Bureau of Public Roads. Reaction was determined by Soiltext.

FIGURE 3.—Profile description of Hayter silt loam, McCreary County, Ky.

SAMPLE COLLECTION AND PREPARATION

FIELD SAMPLING

1A

Site selection

1A1

Select sample sites that represent the dominant use of the soil to be studied. Locate the site away from roads, fence rows, old farmsteads, and any other features that may have caused aberrant properties. Locate a duplicate profile at a site 1 to 20 miles from the first site and in a different mapping delineation. If a group of sites are sampled in transect to illustrate some genetic variable, duplication is less important since reliability of sampling can be checked within the transect set.

Soil sampling

1A2

Take samples from freshly dug pits and not from road cuts. Dig the pit wide enough to expose one face of a pedon and deep enough to expose part of the C horizon or the control section, whichever is deeper. Describe the pedon and any variations within the pedon. In laterally uniform pedons sample from a face about 50 cm wide. Each sample should be representative of the entire cross section of each horizon. 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.

If coarse fragments >20 mm are present, follow procedures outlined in 1A2a or 1A2b. Do not mix horizons if they are interfingered or discontinuous. If contrasting soil components are so small that they cannot be sampled separately, estimate the proportions of each component and record in the pedon description. Make arbitrary subhorizons if morphologically recognizable subhorizons are more than 25 cm thick in the upper part of the pedon or more than 50 cm in the lower part. Consider the requirements of the classification system in locating subhorizon boundaries.

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

Stony soils

1A2a

Volume estimates.—In each horizon or group of horizons estimate the volume percentages of the 20- to 75-mm ($\frac{3}{4}$ in. to 3 in.) and the 75- to 250-mm (3 to 10 in.) fractions. Record the percentages in the pedon description. Collect a 5- to 7-kg sample of the <75-mm fraction and store in an airtight plastic bag if field moisture content is to be

determined. Calculate the volume percentages of coarse fractions (3B2).

Weight estimates.—Estimate and record the volume percentage of the >75-mm fraction as outlined in volume estimates. Collect a 15- to 20-kg sample of the <75-mm fraction and weigh. Sieve out and weigh the 20- to 75-mm fraction. Record the weights of the 20- to 75-mm fraction and the <20-mm fraction. Collect a 5- to 7-kg sample from the <20-mm fraction and store in an airtight plastic bag for determination of field moisture content. Calculate the weight percentages of coarse fractions (3B1).

Marsh and swamp soils

1A2b

Remove the surface mat with a spade. Sample the lower depth with a post-hole digger. Transfer the sample immediately to a plastic bag, knead to remove air, and tie. Invert and place inside a second bag. Remove air from this bag and tie. Place both bags inside a third plastic bag and staple shut. Dig and bag rapidly to minimize exposure to air.

LABORATORY PREPARATION

1B

Standard (airdry) material

1B1

Spread the field samples on trays and airdry at 30 to 35° C. Thoroughly mix and roll the sample with a wooden rolling pin to break up clods. Separate, weigh, and discard the >2-mm fractions. Continue rolling and sieving until only coarse fragments that do not slake in water or sodium metaphosphate (3A1) remain on the sieves. Calculate the percentages of the various fractions as described in 3B. If carbon, total nitrogen, extractable iron, gypsum, or calcium carbonate are to be determined, and subsampling problems are evident, grind the <2-mm material between mullite disks to about 80 mesh; otherwise do not grind.

Square-hole 2-mm sieve*¹

1B1a

Pass sample through a square-hole 2-mm sieve.

Round-hole 2-mm sieve

1B1b

Pass the sample through a round-hole 2-mm sieve.

¹ Methods marked with an asterisk throughout this report are not being used at the present time. They are included because many of the published laboratory data have been determined by these methods.

Field-moist material**1B2**

Force the field-moist sample through a 2-mm screen by hand, using a large rubber stopper. Place the prepared samples in polyethylene bags and seal in quart fruit jars. Airdry part of the sample and grind to 80 mesh for carbon, extractable iron, gypsum, calcium carbonate and nitrogen determinations.

Carbonate-containing material**1B3***Procedure*

Prepare dialysis membrane sacks from 5½-inch cellulose casing (Visking Company), using large rubber bands to tie the bottoms. Place the sample (as much as 6 kg if very gravelly and highly calcareous) in a dialysis membrane

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 reservoir 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.

CONVENTIONS

SIZE-FRACTION BASE FOR REPORTING DATA

2A

Particles <2 mm

2A1

Unless otherwise specified, report all data on the basis of the <2-mm material.

Particles <specified size>2 mm

2A2

The maximum coarse-fragment size for the >2-mm base varies. The base usually includes fragments as large as 75 mm (3 inches) if present in the soil. The maximum size for fragments larger than 75 mm is decided during sampling. It is established either because of the difficulty in handling larger material or because, by definition, soil does not include material larger than 250 mm (10 inches) in diameter.

Record the particle size set as the maximum in parentheses in the column heading. 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 symbols are used or have been used for trace and zero quantities and for samples not tested.

- tr, Tr... Trace, either not measurable by quantitative procedure used or less than reportable amount.
- tr(s).... Trace, detected only by qualitative procedure more sensitive than quantitative procedure used.
- Analysis run but none detected.
- (s).... None detected by sensitive qualitative test.
- blank.... Analysis not run.
- nd..... Not determined, analysis not run.
- <..... Either none present or amount is less than reported amount, e.g., <0.1 is in fact <0.05 since 0.05-0.1 is reported as 0.1.

PARTICLE-SIZE ANALYSES

PARTICLES <2 mm (PIPET METHOD) 3A

Airdry samples 3A1

Apparatus

- Electrolytic beakers, 250-ml.
- Pasteur-Chamberlain filter candles, fineness "F."
- Nursing bottles, 8-oz, or centrifuge bottles, flat-bottomed, 250-ml.
- Shaker, horizontal, 120 oscillations per minute.
- Cylinders, 1,000-ml.
- Stirrer, motor-driven.
- Stirrer, hand.—Fasten a circular piece of perforated brass sheeting to one end of a brass rod; place a wide rubber band around the edge of the brass sheeting to prevent abrasion.
- Shaw pipet rack.
- Lowy pipets, 12-second filling time.
- Asbestos pipe-insulating cover.
- Shaker with ½-inch vertical and lateral movements and 500 oscillations per minute.
- Weighing bottles.
- Set of sieves, as follows:

Sieve opening (mm)	Specifications
1.0....	Perforated brass plate, round holes, No. 3 straight, 0.04-inch diameter holes, 240 holes per square inch.
0.5.....	Perforated brass plate, round holes, No. 00 staggered, 0.02-inch diameter holes, 714 holes per square inch.
0.25....	60-mesh, Bureau of Standards (Phosphor Bronze wire cloth).
0.177...	80-mesh, Bureau of Standards (Phosphor Bronze wire cloth).
0.105...	150-mesh, Bureau of Standards (Phosphor Bronze wire cloth).
0.074...	200-mesh, Phosphor Bronze wire cloth (added in 1966).
0.047...	300-mesh, Phosphor Bronze wire cloth,

Procedure

Removing organic matter.—Place about 10 g airdry soil containing no particles larger than 2 mm in an electrolytic Pyrex beaker. Add about 50 ml water and then add a few milliliters of H₂O₂. Cover the beaker with a watchglass. If a violent reaction occurs, repeat the cold H₂O₂ treatment periodically until no more frothing occurs. Heat the beaker to about 90° C on an electric hot plate. Add H₂O₂ in 5-ml quantities at 45-minute intervals until the organic matter is destroyed as determined visually. Continue heating for about 30 minutes to remove any excess H₂O₂.

Removing dissolved mineral matter.—After the H₂O₂ treatment, place the beaker in a rack and add about 150 ml 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, using finger as policeman. Place the sample overnight in an oven at 105° C, cool in a desiccator, and weigh to the nearest milligram. Transfer the sample to a nursing or centrifuge bottle for dispersion and record the oven-dry weight of the beaker. Use the calculated weight of the oven-dry H₂O₂-treated sample as the base weight for calculating percentages of the various fractions.

Removing cementing agents (optional).—Treat the sample with about 200 ml of 1N sodium acetate buffered at pH 5 to remove CaCO₃. When CO₂ 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 0.1N NaOH. Wash free of salts with a filter candle system before proceeding.

Dispersing the sample.—Add 10 ml sodium metaphosphate² dispersing agent and transfer

the sieve above the cylinder. Avoid using jets of water in washing the sample. Gently tap the sieve clamp with the side of the hand to facilitate washing. Continue washing until the volume in the cylinder is about 800 ml. Sand and some coarse silt remain on the sieve. 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, and dry at 105° to 110° C. Then place the evaporating dish in a desiccator until convenient to sieve and weigh the contents. Make the silt and clay suspension in the cylinder to 1 liter with demineralized water, cover with a watchglass, and set aside until the pipettings are to be made.

Pipetting.—First pipet the $<20\mu$ fraction at a 10-cm depth, varying sedimentation time according to temperature. Next, pipet $<2\mu$ fraction after a predetermined settling time (usually 6 to $6\frac{1}{2}$ hours), varying depth according to time and temperature. Use a Lowy 25-ml automatic pipet having a filling time of about 12 seconds. Before each pipetting, stir material in the sedimentation cylinder for 6 minutes with a motor-driven stirrer (8 minutes if suspension has stood for more than 16 hours). Remove stirrer, slip a length of pipe-insulating cover over sedimentation cylinder, and stir the suspension for 30 seconds with a hand stirrer, using an up-and-down motion. Note the time at completion of stirring. About 1 minute before sedimentation is complete, lower the tip of the pipet slowly into the suspension to the proper depth with a Shaw pipet rack. Fill the pipet and empty into a 60-ml weighing bottle having an outside cover. Rinse the pipet into the weighing bottle once. Use a vacuum to dry the pipet for the next sample. Dry the weighing bottle in an oven at 95° to 98° C and then dry further for about 4 hours at 105° C. The initial drying at a lower temperature prevents spattering. Cool the weighing bottle in a desiccator containing phosphorus pentoxide (P_2O_5) as a desiccant. Weigh.

Sieving and weighing the sand fractions.—Weigh the dry sands, including some coarse silt, and brush into a nest of sieves (include an 80-mesh sieve to obtain ISSS fraction I). Shake for 3 minutes on a shaker having $\frac{1}{2}$ -inch vertical and lateral movements and making 500 oscillations per minute. Record the weights of the individual sand fractions. If the sum of the weights of the size fractions is equal to the total weight, assume that there has been no error in weighing.

Calculations

B = weight correction for dispersing agent (g)

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

$$D = \frac{100}{\text{g of } H_2O\text{-treated oven-dry total sample}}$$

The $<20\mu$ fraction minus $<2\mu$ fraction equals ISSS fraction III. Subtract the sum of the percentages of sand and clay from 100 to get USDA silt.

Sand fractions:

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

References

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

Carbonate and noncarbonate clay 3A1a

Procedure

Check for calcium or magnesium carbonate in the clay fraction by adding a few drops of 6N HCl to the dried residue from the aliquot withdrawn during the regular pipet analysis. If carbonate is present, withdraw a second aliquot of clay. For a greater precision, use a 50-ml pipet to withdraw the second aliquot. Place the aliquot in a suitable reaction flask. Evaporate to dryness or near dryness and determine carbonate by an appropriate procedure (6E).

Calculations

Carbonate clay (pct. <2 mm)

$$C_c = \frac{\text{g } CO_2}{\text{g sample}} \times \frac{\text{ml in cylinder}}{\text{ml in pipet}} \times 227.4$$

Noncarbonate clay (pct. <2 mm)

$$N_c = \text{Total clay (pct. } <2 \text{ mm)} - C_c$$

Noncarbonate clay (pct. noncarbonate <2 mm)

$$= \frac{N_c}{1 - \frac{\text{Total carbonate}}{100}}$$

References

Shields and Meyer (1964).

Fine clay ($<0.2\mu$) 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 0.1 mg).

Procedure

Temperature	Minutes
20° C	39.2
25° C	34.9
30° C	31.2
35° C	28.1
40° C	25.5

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 seconds (avoid turbulence) and transfer to tared Erlenmeyer flask or weighing bottle. Dry at 110° C, cool in P₂O₅ desiccator and weigh.

Calculations

Proceed as in 3A1.

Water-dispersible clay

3A1c

Proceed as in 3A1 except eliminate peroxide and metaphosphate treatments. Shake sample overnight in distilled water.

Moist samples

3A2

For soils in which drying affects dispersion

volume percentage estimates can be used to calculate weight percentage estimates.

Weight estimates

3B1

By field or laboratory weighing

3B1a

Weigh and sieve the entire horizon sample through 75-mm and 20-mm screens. Weigh the >75-mm and 75- to 20-mm fractions. Calculate the <20-mm fraction by subtraction. Take a subsample of the <20-mm fraction for laboratory processing. Determine the air-dry weight of <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. Record these fractions as percentage of <20 mm. Also report these fractions as percentage of <75 mm and percentage of <250 mm (if applicable).

If the fine earth adheres to the larger particles, wash the coarse material and apply the appropriate corrections.

From volume and weight estimates

3B1b

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

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

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

C_m has been reported on some data sheets. It is referred to as the coarse-fragment conversion

factor. This value multiplied by laboratory data converts laboratory results from a weight basis to a field volume basis. *C_m* 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.

$$>2 \text{ (pct. by vol)} = 100 (1 - C_m)$$

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

FABRIC-RELATED ANALYSES

BULK DENSITY

4A

Density is defined as mass per unit volume. Soil density differs from most density in that the mass of the liquid phase is excluded. Also, the volume over which the weight is determined includes interparticle space. Because of these irregularities, soil density has been called bulk density, D_b , to distinguish it from the more usual density that is based on intraparticle volume only. Furthermore, since the volume of a given mass of soil depends on its water content, subscripts are added to designate the moisture condition when the measurement was made. Thus D_{b_m} is the bulk density of a moist sample, D_{b_i} is the bulk density of a clod sample equilibrated at $\frac{1}{2}$ -bar tension, and D_{b_d} is the bulk density of a dry sample.

Saran-coated clods

4A1

Reagents

Methyl ethyl ketone.

Dow Saran F310.—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. Saran-solvent ratios of 1:4 to 1:8 are used, depending on the porosity of the soil to be coated.

To mix the plastic solution, fill a weighed 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 plastic with an air-powered or non-sparking electric stirrer under an exhaust hood.³

this piece remove 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. 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.

Hold the separated clod by a thread or fine wire and immerse it briefly in the plastic solution. For convenience, either of two concentrations of plastic solution is usually used—a 1:7 solution for the majority of soil samples or a 1:4 solution for clods that have large pores. Then suspend the immersed clod from a line to allow the coating to dry, usually 15 to 30 minutes.⁴ 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 apply additional coatings of plastic 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 moisture condition (e.g. $\frac{1}{2}$ -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 oven-dry clod in air and in water.

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 oven-dry weight of the clod.

Sometimes it is necessary to correct for weight and volume of the plastic coating. The coating has a density of about 1.3 g per cubic centimeter and it loses 10 to 20 percent of its airdry weight on oven-drying at 105° C. Thus, the amount of correction becomes smaller as bulk density of the soil approaches density of the coating and as moisture content of the soil approaches the weight loss of the coating.

Calculations

The example given is for a clod equilibrated at 1/2-bar tension.

$$Db_1 = \frac{wt\ clod_1 - wt\ >2\ mm - wt\ coat_{od}}{vol\ clod_1 - vol\ >2\ mm - vol\ coat}$$

$$Db_{od} = \frac{wt\ clod_{od} - wt\ >2\ mm - wt\ coat_{od}}{vol\ clod_{od} - vol\ >2\ mm - vol\ coat}$$

$$W_1 = \frac{wt\ clod_1 - wt\ clod_{od} - (wt\ coat_{od} - wt\ coat)}{wt\ clod_1 - wt\ >2\ mm - wt\ coat}$$

volume as described in 4A1a. Determine oven-dry weight and calculate bulk density as described in 4A1.

30-cm absorption (Db₃₀)

4A1c

After measuring airdry volume, remove a patch of the Saran coating from one side of each clod. Next place the clods on a sand tension table with the exposed side in contact with very fine sand that has been equilibrated to 30-cm water tension. Again weigh a few clods each day until they reach constant weight and assume that all the clods are at 30-cm water tension. Most clods reach equilibrium in 7 to 10 days. Remove the clods from the tension table and coat with Saran until waterproof. Measure volume of the clods and calculate bulk density as described in 4A1.

1/2-bar desorption I (Db₁)

4A1d

After measuring airdry volume, remove a patch

oven-dry weight and volume and calculate bulk density as described in 4A1.

Paraffin-coated clods*

4A2

Oven-dry (Db)

4A2a

~~Remove the clods coat with paraffin and~~

ing is contained 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 $\frac{1}{10}$ or $\frac{1}{8}$ bar than larger pieces or clods. The error resulting

Natural clods

4B1c

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

Cores*

4B1d

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

Pressure-membrane extraction (15 bars) 4B2

Apparatus

WATER-RETENTION DIFFERENCE

4C

Between 1/3-bar (or 30-cm) and 15-bar tension

4C1

$$WRD \text{ (g/cc)} = \frac{(W_1 - W_{15})(Dbt)Cm}{100}$$

where

WRD = Weight (g) of water retained in 1 cc of whole soil between 1/3-bar and 15-bar tension (can be converted to and usually reported as inches of water per inch of soil).

W₁ = Weight percentage of water retained

Nomographs for these calculations have been published. Enlarged copies are available from the soil survey laboratories.

References

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

MICROMORPHOLOGY

4E

Thin sections

4E1

Preparation

4E1a

Apparatus

Metallographic polisher with 8-inch cast-iron laps.

Diamond saw.

Electric oven.

Hotplate with temperature control or a petrographic slide warmer.

Polarizing microscope.

Mixing plastic solution.—There are many good resins on the market; one that has been used in our laboratories is Laminac resin 4110.⁵ The diluting solvent is monomeric styrene and the catalyst Lupersol DDW.⁶ Add one part Laminac resin 4110 to two parts monomeric styrene (by volume); add Lupersol DDW catalyst to constitute 5 percent (by volume) of the entire solution. The proportions of Laminac and styrene can be varied according to the viscosity needed to insure penetration. Best results are usually obtained with the thickest solution that will penetrate the material; the solution suggested works well with most soils.

Impregnation.—Place the sample in a porcelain vessel with sloping sides and cover with the plastic solution. Then place the dish in a vacuum desiccator and pump all the air from the sample. Do not mistake boiling of the solution under evacuated conditions for air bubbles escaping from the sample material. When impregnation is complete (usually about 5 min),

to overcook it. Place the sample obliquely on the balsam and slowly lower it until parallel with the glass slide. This presses out most of the air bubbles. Remove the slide and sample from the hot plate and lay on a table with the glass slide upward. Maintain slight pressure while the sample and slide are hot to reduce thickness of the balsam between the slide and the sample and to remove any remaining air bubbles. The chip must be perfectly flat and contact with the slide must be uniform or part of the section may be lost before it is ground to the desired thinness (usually about 0.030 mm).

Final grinding.—When the mounted sample is cool, begin the final grinding. Use coarse abrasive until the sample is relatively thin. Then use successively finer abrasives. Care and considerable practice are needed to develop the dexterity required to handle an almost finished section without overgrinding. Examine it frequently under a polarizing microscope during the final stages. If quartz is present in the sample, it can be used to judge thinness. If the sample is about 0.030 mm thick, the quartz interference colors are of the first order, i.e., white, gray, and pale yellow. It is often advisable to finish grinding on ground-glass plates, using the finest abrasive. Wash the section free of abrasive and dry thoroughly.

Plastic coating.—Before putting a protective cover glass over the finished soil section, coat it with very thin plastic. Flexible collodion serves this purpose very well. It is not necessary to coat sections of some minerals or rock materials, but coating soil sections keeps them from breaking apart when heated or when slight pressure is applied to the cover glass to remove any air bubbles. Further, if it becomes necessary to repair the slide for any reason, the thin section is less delicate and can be handled easily. The plastic coat serves also as a protective covering and, if desired, can eliminate use of a cover glass. Its refractive index is about the same as that of balsam. Apply the collodion with a soft-bristle art brush. Use a brush large enough to permit coating with one or two strokes. Too frequent brushings disturb the surface and cause it to appear wavy and out of focus under the microscope. Let the plastic dry.

Seating the cover glass.—Spread a small quantity of precooked balsam over the surface of the thin section and heat until it is liquid. Place a cover glass obliquely on one end of the section and lower it very slowly. If any air bubbles remain, squeeze them out by pressing lightly on the cover glass. Excess balsam can be removed with a razor blade when the section is cold.

Very dense soils and soils in which the clay fraction is 30 percent or more montmorillonite require special handling. It is necessary to use either a dry-grinding technique or a more pene-

trating impregnation procedure. To dry-grind, cut the sample (without using water) to the appropriate size. Sprinkle coarse abrasive (American Optical No. 190) on a ground-glass plate and commence grinding one face of the sample by hand. Best results are obtained by using a figure "8" or counterclockwise motion. Continue grinding, using successively finer abrasive, until the surface is highly polished. From this point follow the regular mounting technique.

Aroclor 5460, a thermoplastic chlorinated diphenyl resin (Monsanto), seems to give better impregnation of these dense materials. The procedure follows. 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), and Cady (1965).

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 due to 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 at first 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 needleshaped 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 a ball. 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 20 μ to 30 μ thick. Grains smaller than this are stacked up and cannot be seen as individuals, and pores much smaller than this cannot be seen clearly.

Identification of sand and silt grains in thin sections is 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 that 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 is important to the problem being studied, it is best to do this work on separated 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-

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 re-

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).

Moved-clay percentage

4E1c

Apparatus

Diamond tile saw.
Thin-section equipment.
Point-counting eyepiece.

Reagents

Aroclor 5460 (Monsanto).
Polyester resin.
Styrene.

Procedure

Impregnate an undisturbed field sample with Aroclor (4E1b). With a diamond saw cut the clods into pieces about 3 by 1 by 1 cm. Mount about 10 pieces side by side with polyester resin (use a little styrene) to form a block. Cut this assembly to form slices 3 by 1 by 1 cm. Slice all

the field sample, composite, and withdraw subsamples of 10 to 15 slices. Stack these slices, tape them together, and mount in plastic (polyester resin plus styrene). Cut a section through the stack parallel to the direction of stacking and along the longer of the two remaining axes. Mount one such section from each stack on a glass slide and prepare a thin section.

To estimate the moved-clay volume insert a point-counting eyepiece into the microscope and run a transect along each strip. Keep the transect length and the number of fields in a transect constant. Count the number of points that fall on moved clay. Divide this number by the total number of points to get an estimate of the proportion of moved clay. To convert these volume estimates to weight estimates, multiply by the ratio of the bulk density of the moved clay to the bulk density of the appropriate dry fabric. Assume that the moved clay has a bulk density of 2.00 g per cubic centimeter.

References

Grossman (1964).

Scanning electron microscopy

4E2

Electronically reproduced images of fabric surfaces can be obtained at magnifications ranging from 50 to 30,000 diameters. Depth of focus by this technique is large compared to that by light microscope. Stereoscopic pictures can be taken to give three-dimensional viewing.

Procedure

Take a sample of fabric up to 10 mm in diameter and 2 to 3 mm thick. Coat with a thin metallic layer and insert in the instrument. The image is displayed on a cathode ray tube.

PLASTICITY INDEX

4F

The plasticity index is the difference between the upper plastic limit and the liquid limit. Details are available in ASTM method D424.

References

American Society for Testing and Materials (1970).

Liquid limit

4F1

See ASTM method D423.

Upper plastic limit

4F2

See ASTM method D424.

ION-EXCHANGE ANALYSES

CATION-EXCHANGE CAPACITY

5A

Procedure

NH₄OAc, pH 7.0 (Büchner funnel)

5A1

Reagents

Ammonium acetate (NH₄OAc), 1N, pH 7.0.

—Mix 68 ml ammonium hydroxide (NH₄OH), specific gravity 0.90, and 57 ml 99.5-percent acetic acid (CH₃COOH) per liter of solution desired. Cool, dilute to volume with water, and adjust to pH 7.0 with CH₃COOH or NH₄OH.

—Optionally prepare from NH₄OAc reagent salt and adjust pH.

Ethanol (CH₃CH₂OH), 95-percent, U.S.P.

Nessler's reagent (optional).—Prepare according to Yuen and Pollard.

Procedure

Weigh 25 g⁷ airdry <2-mm soil into a 250-ml Erlenmeyer flask and add 35 to 50 ml NH₄OAc solution. Stopper, shake the flask for several minutes, and allow to stand overnight. Transfer contents of the flask to a Büchner funnel (Coors

Transfer the soil plus filter paper from method 5A1 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 1N NaOH. Connect the flask with the condenser and distill 200 ml into 50 ml 4-percent H₃BO₃ solution. Titrate the distillate to the first tinge of purple with 0.2N HCl, using 10 drops mixed indicator and 2 drops brom cresol green.

Calculations

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

Report on oven-dry basis.

References

Peech et al. (1947).

Displacement and distillation of adsorbed ammonia. semimicro Kjeldahl 5A1h

Calculations

$$CEC \text{ (meq/100 g)} = \frac{\text{ml H}_2\text{SO}_4}{\text{g sample}} \times N \text{ of acid} \\ \times \frac{\text{ml leachate}}{\text{ml aliquot}} \times 100$$

Report on oven-dry basis.

NaOAc, pH 8.2 5A2
Centrifuge method 5A2a

Reagents

Sodium acetate (NaOAc), 1N, pH 8.2.
Ethanol, 95-percent.
Ammonium acetate (NH₄OAc), 1N, pH 7.0.
—Add 57 ml concentrated acetic acid and 68 ml concentrated NH₄OH, specific gravity 0.90, to about 800 ml water. Cool and dilute to 1 liter and adjust to pH 7.0 by adding more NH₄OH or acetic acid.

Procedure

Weigh 5-g samples to an accuracy of 1 percent and place in centrifuge tubes. Add 33 ml NaOAc, stopper the tubes, and shake for 5 minutes. Remove stopper and centrifuge until the supernatant liquid is clear (usually 5 min). Decant the supernatant liquid as completely as possible and discard. Repeat four times, discarding the supernatant liquid each time. After the last saturation, wash the rubber stoppers and use absorbent paper to remove any acetate crystals remaining on lip of centrifuge tube. Add about 30 ml ethanol to each tube, stopper, shake for 5 minutes, remove stopper, and centrifuge until the supernatant liquid is clear. Decant and discard the supernatant liquid. Continue washing until the electrical conductivity of the supernatant liquid from the last washing is between 55 and 40 μ mho per centimeter. Optionally, decrease volume by about 5 ml each washing. Replace the absorbed sodium from the sample by extracting with three 30-ml portions of NH₄OAc solution. Dilute to 100 ml and determine the sodium concentration as described in 6P2a.

Calculations

$$CEC \text{ (meq/100 g)} = \frac{\text{meq/l Na from curve}}{\text{g sample}} \times \text{dilution} \times 10$$

Report on oven-dry basis.

References

Richards (1954).

Sum of cations 5A3
Acidity by BaCl₂-triethanolamine, pH 8.2;
bases by NH₄OAc, pH 7.0 5A3a

Procedure

Compute the cation-exchange capacity from the sum of the extractable bases (NH₄OAc extract, methods 6N2, 6O2, 6P2, 6Q2) and the extractable acidity obtained by titrating the

triethanolamine (TEA) extract (6H). This value for exchange capacity is not valid in the presence of significant quantities of soluble salts or calcium carbonate.

References

Peech et al. (1947).

Sum of bases plus Al 5A3b

Sum the extractable bases and the KCl-extractable aluminum.

KOAc, pH 7.0* 5A4

Procedure

Proceed as in 5A1 except substitute 1N KOAc, pH 7.0, for NH₄OAc. Determine potassium with flame photometer.

BaCl₂, pH 8.2 5A5

Apparatus

Leaching tubes.
Flame photometer.

Reagents

Buffer solution.—Barium chloride (BaCl₂), 0.5N, and triethanolamine (TEA), 0.2N. Adjust to pH 8.2 with HCl. Protect from CO₂ of the air by attaching a drying tube containing soda lime (sodium calcium hydrate) to the air intake.

Replacement solution.—Barium chloride (BaCl₂), 0.5N. Add 0.4 ml buffer solution per liter and mix. Protect from CO₂ with soda-lime tube.

Magnesium nitrate (Mg(NO₃)₂), 1N.

Procedure

Transfer a 5-g sample to a leaching tube. For field-moist samples use a sample large enough to give an oven-dry weight of about 5 g. Leach with 50 ml BaCl₂-TEA solution, controlling the leaching rate to give at least 4 hours of soil-solution contact time. Follow with 100 ml BaCl₂ replacement solution, controlling the leaching rate so that the soil and BaCl₂ solutions are in contact for a total of 20 to 24 hours. Rinse walls of leaching tube with 15 to 20 ml H₂O, collecting this washing with leachates from BaCl₂ solutions. Extractable acidity can be determined by using this solution (6H1a).

Place leaching tube on a clean flask and wash with methanol until free of chloride ion. For many samples 100 ml methanol is enough, but more methanol may be needed for some soils, particularly those of heavy texture and containing large amounts of hydrous oxides. Leach with 100 ml 0.001N BaCl₂ to remove methanol.

Disconnect leaching tube and flask, rinse underside of leaching tube, place over a 250-ml volumetric flask, and leach with 100 ml 1N Mg(NO₃)₂ solution. Control leaching rate to give a soil-solution contact time of 16 hours or

more. Rinse walls of leaching tube with 15 to 20 ml H₂O; collect rinse in the Mg(NO₃)₂ leachate. Make to volume.

Barium by flame photometry **5A5a**

Make standards in 1N Mg(NO₃)₂. Determine barium by flame photometry at 489 mμ.

Calculations

$$CEC \text{ (meq/100 g)} = \frac{\text{meq/l Ba from curve}}{\text{g sample}} \times \text{dilution} \times 25$$

Report on oven-dry basis.

NH₄OAc, pH 7.0—leaching tube **56A**

Apparatus

Allihn leaching tubes or 50-ml plastic syringe barrels.

Reagents

Same as in 5A1.

Procedure

Prepare the Allihn tubes by placing either filter paper (Reagent Anal. No. 024 A H 2 cm fiber glass)

EXTRACTABLE BASES

5B

NH₄OAc. extraction

5B1

Procedure

Analyze the NH₄OAc leachate from method 5A1a for calcium, magnesium, sodium, and potassium (methods 6N2, 6O2, 6P2, 6Q2).

Uncorrected (extractable)

5B1a

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

Corrected (exchangeable)

5B1b

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 or for calcium in the presence of gypsum because these salts are soluble in NH₄OAc.

Uncorrected (extractable) 5B3a

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

Corrected (exchangeable) 5B3b

If a soil contains soluble salts, estimate their amounts from the saturation extract and correct as in 5B1b.

NH₄OAc, pH 7.0 (revised) 5B4

Analyze the NH₄OAc leachate from method 5A6 for Ca, Mg, Na, and K (methods 6N2, 6O2, 6P2, 6Q2).

Uncorrected (extractable) 5B4a

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

Corrected (exchangeable) 5B4b

If a soil contains soluble salts, estimate their amounts from the saturation extract and correct as in 5B1b.

BASE SATURATION 5C

NH₄OAc, pH 7.0 5C1

Divide sum of NH₄OAc-extracted bases by the exchange capacity determined by method 5A1.

NaOAc, pH 8.2 5C2

Divide sum of NH₄OAc-extracted bases by the exchange capacity determined by method 5A2a.

Sum of cations, TEA, pH 8.2 5C3

Divide sum of NH₄OAc-extracted bases by the sum of cations determined by method 5A3.

SODIUM SATURATION (EXCHANGEABLE-SODIUM PERCENTAGE) 5D

NaOAc, pH 8.2 5D1

Divide exchangeable sodium (meq/100 g) by the exchange capacity determined by method 5A2a.

NH₄OAc, pH 7.0 5D2

Divide exchangeable sodium (meq/100 g) by the exchange capacity determined by method 5A1.

SODIUM-ADSORPTION RATIO 5E

Calculate the sodium-adsorption ratio (SAR) by the following equation

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

where [Na⁺], [Ca⁺⁺], and [Mg⁺⁺] refer to the concentration of these cations expressed in milliequivalents per liter.

References

Richards (1954).

CALCIUM SATURATION (EXCHANGEABLE-CALCIUM PERCENTAGE) 5F

NH₄OAc, pH 7.0 5F1

Divide the NH₄OAc-extracted calcium by the exchange capacity determined by procedure 5A1 or 5A6.

CHEMICAL ANALYSES

ORGANIC CARBON

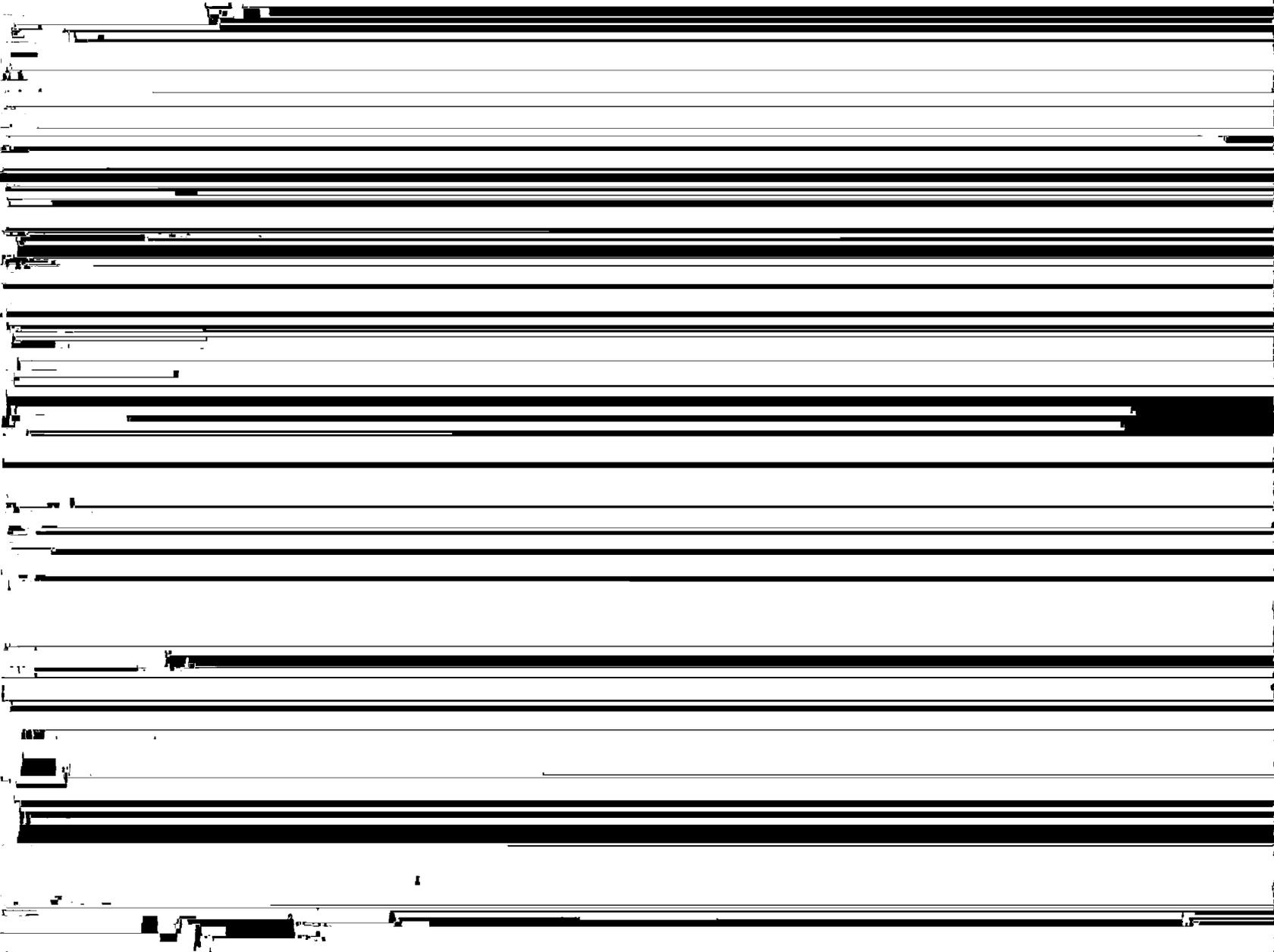
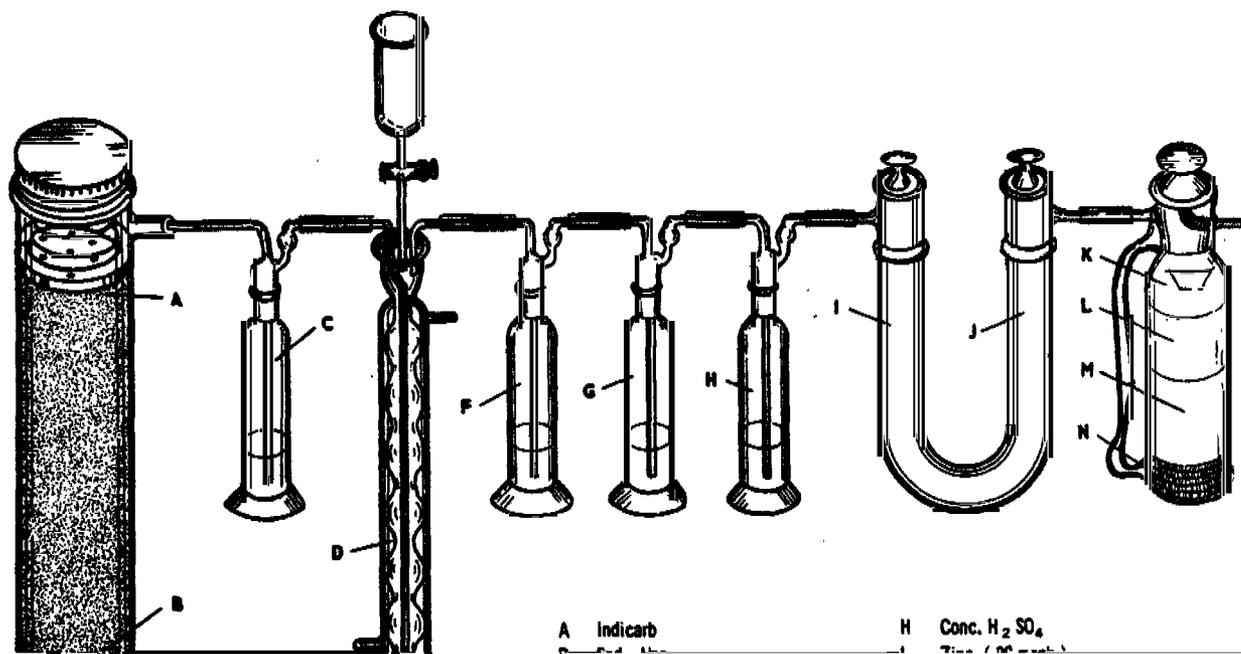
6A

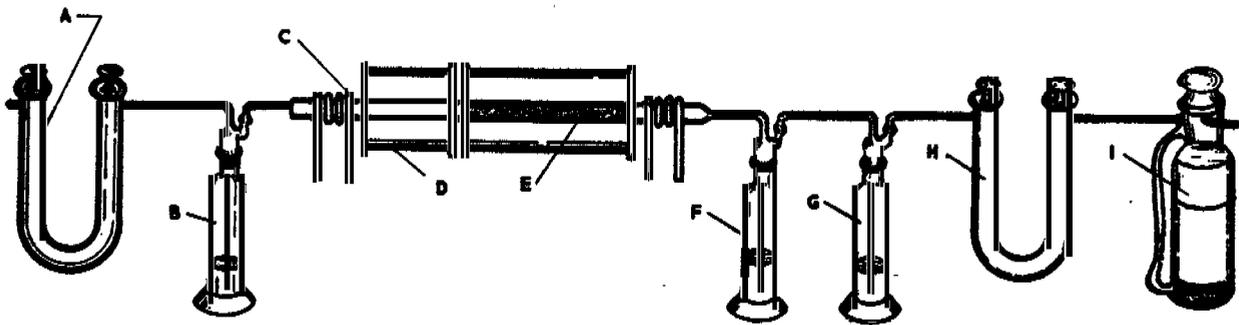
Calculations

Determine carbon for each horizon that may contain organic matter. Report as carbon percentage by weight of <2-mm material.

Organic carbon (pet.)

$$= \frac{\text{ml FeSO}_4 \text{ blank} - \text{ml FeSO}_4 \text{ sample}}{\text{g sample}} \times N \text{ of FeSO}_4 \times \frac{0.30}{0.77}$$

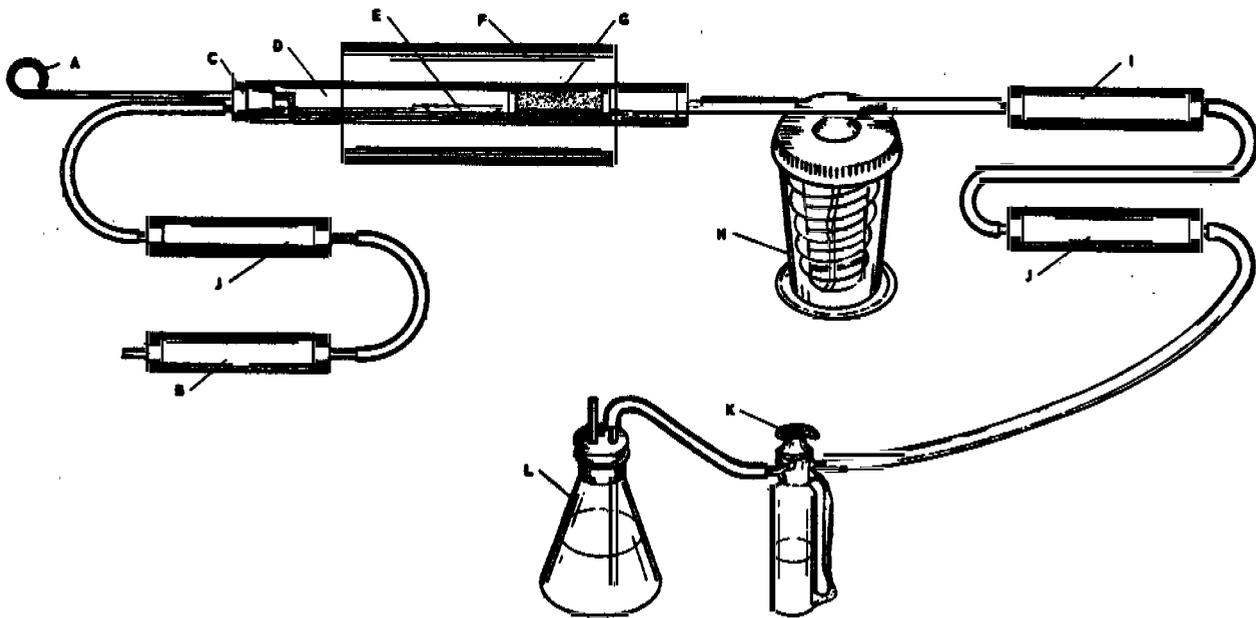




- A Schwitz absorption tube containing 8-20 mesh Caroxite
- B Gas washing bottle containing conc. H_2SO_4
- C Cooling coils
- D Combustion furnace
- E Platinum and asbestos catalyst
- F Gas washing bottle containing saturated Ag_2SO_4

- G Gas washing bottle containing conc. H_2SO_4
- H Schwitz absorption tube containing granular zinc in left arm and anhydrous magnesium perchlorate in right arm
- I Nesbit absorption bottle containing anhydrous magnesium perchlorate in top layers, 8-20 mesh Caroxite in middle layer, and glass wool in bottom layer

FIGURE 5.—Apparatus for organic carbon determination by dry combustion, carbon dioxide evolution I (6A2a).



- A Push rod
- B Drying tube containing indicarb
- C Rubber stopper
- D Aluminum combustion tube
- E Combustion boat
- F Combustion furnace

- G Cupric oxide wire
- H Milligen gas washing bottle containing concentrated H_2SO_4
- I Tube containing ZnO_2
- J Tube containing Anhydrous
- K Nesbit absorption bulb containing Anhydrous and indicarb
- L Bubble counter containing H_2SO_4

FIGURE 6.—Apparatus used for organic carbon determination by dry combustion, carbon dioxide evolution II (6A2b).

Procedure

Heat tube to approximately 950° C. Sweep with oxygen until weight of Nesbitt bulb is constant. Remove rubber stopper in the oxygen-inlet end of the tube and insert the boat containing 0.5 to 1.5 g soil. Reinsert the stopper and use the push rod to move the boat into the hot zone. Heat for 10 minutes, remove bulb, and record weight gain. Remove boat and repeat process with fresh sample, using the same Nesbitt bulb.

Calculations

Report on oven-dry basis as in 6A1a.

References

Robinson (1930) and Post.³

Peroxide digestion* 6A3

Gravimetric weight loss 6A3a

Reagents

Hydrogen peroxide (H₂O₂), 6-percent.

Procedure

Digest soil for several hours in a covered beaker with 6-percent H₂O₂. Remove soluble material by washing three to five times with a Pasteur-Chamberlain clay filter, "F" fineness. Dry the beaker and soil, and weigh.

Calculations

Organic matter (pct.)

$$= \frac{\text{g loss on heating} + \text{g dry matter in soln}}{\text{g sample}} \times 100$$

Note that organic matter differs from organic carbon (see 6A1a).

References

North-Central Regional Research Committee on soils (1955).

NITROGEN 6B

Kjeldahl digestion 6B1

Reagents

Concentrated sulfuric acid (H₂SO₄).

Salt mixture.—Potassium sulfate (K₂SO₄) or anhydrous sodium sulfate (Na₂SO₄), ferrous sulfate (FeSO₄), and copper sulfate (CuSO₄), ratio 10:1:½; or No. 1 Kel-Pak, 9.9 g K₂SO₄, 0.41 g mercuric oxide (HgO), 0.08 g CuSO₄.

Sodium hydroxide (NaOH), about 45-percent.—Add Na₂S 1:25 if mercury salt mixture used.

Antifoam mixture.—Equal parts n-octyl alcohol and mineral oil.

Mossy zinc or DeVardas alloy.

³ Post, G. J. A study of three methods for determination of organic carbon in Ohio soils of several great soil groups and the profile distribution of carbon-nitrogen ratios. M.Sc. thesis, The Ohio State University, 34 pp. 1956.

Procedure

Digest 10 g soil in an 800-ml Kjeldahl flask with 30 ml H₂SO₄ and about 10 g salt mixture. Continue digestion for 1 hour after mixture is colorless or nearly so.

References

Association of Official Agricultural Chemists (1945).

Ammonia distillation 6B1a

Reagents

Mixed indicator.—Methyl red, 0.125-percent, and methylene blue, 0.0825-percent, in 95-percent ethanol.

Methyl red (optional), 0.25-percent.

Brom cresol green, 0.1-percent aqueous solution.

Boric acid (H₃BO₃), 4-percent.

HCl, standardized, 0.1N or 0.05N.

Procedure

Cool digestion flask (6B1) and dilute contents with about 400 ml water. Add 2 to 3 g mossy zinc, 5 drops antifoam mixture, and 70 ml concentrated NaOH solution. Connect flask to condenser and distill ammonia into 25 or 50 ml H₃BO₃ solution. Titrate with standard HCl to purple end point, using 10 drops mixed indicator and 2 drops brom cresol green or 3 drops brom cresol green and 1 drop methyl red.

Calculations

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

Report on oven-dry basis.

Semimicro Kjeldahl 6B2

Apparatus

Aminco-Koegel semimicro rotary digestion rack and steam-distillation apparatus.

Reagents

Concentrated sulfuric acid (H₂SO₄).

H₂SO₄, 0.01N, standardized.

Sodium hydroxide (NaOH), 50-percent.

Boric acid (H₃BO₃), 2-percent.

Mixed indicator.—Mix 0.1 g methyl red and 0.1 g brom cresol green and dissolve in 250 ml ethanol.

Salt mixture.—Mix 790 g potassium sulfate (K₂SO₄), 100 g ferrous sulfate (FeSO₄), 100 g copper sulfate (CuSO₄), and 10 g selenium metal.

Procedure

Using an analytical balance, weigh on a cigarette paper either 0.500 or 1.000 g oven-dry soil that has been ground to about 0.2 mm. Roll soil in cigarette paper and drop into a 100-ml digestion-distillation flask. Add 2 g salt mixture, 1 ml water, and 5 ml concentrated H₂SO₄. Swirl vigorously and digest, rotating the flask

frequently until fumes are emitted. Continue digestion for at least 1 hour after mixture becomes white. Cool to room temperature and add 15 ml water. Shake until the contents of the flask are thoroughly mixed.

Ammonia distillation

6B2a

Procedure

Measure 10 ml 2-percent H_2BO_3 with an automatic pipet into a 125-ml flask and add 0.5 ml mixed indicator. Place this flask under delivery tube. Connect digestion-distillation flask containing soil digested according to method 6B2 to the distillation unit by the ground-glass connection. Start steam passing through the system and slowly add 15 ml 50-percent $NaOH$. Distill for 12 minutes, add 0.5 ml more mixed indicator, and titrate the absorbed ammonia with 0.01N H_2SO_4 .

Calculations

$$N \text{ (pct.)} = \frac{\text{ml } H_2SO_4}{\text{g sample}} \times N \text{ of } H_2SO_4 \times 1.4$$

Report on oven-dry basis.

IRON

6C

Dithionite extraction*

6C1

Reagents

Sodium dithionite powder ($Na_2S_2O_4$).
Hydrochloric acid (HCl), 10-percent.

Apparatus

8-oz Pyrex nursing bottles or 250-ml flat-bottomed centrifuge bottles.

Procedure

Place 4 g soil, ground to 80 mesh, in a nursing or centrifuge bottle. Add 4 g $Na_2S_2O_4$ and 75 ml water. Stopper and shake overnight or for 16 hours. Then adjust the pH to 3.5 to 4.0, if necessary, with 10-percent HCl. Let stand for no less than 1 hour, stirring four or five times. Transfer the suspension to a graduated cylinder, dilute to 200 ml with water, and mix. Centrifuge or filter a part of the suspension and transfer 50 ml of the clear solution to a 250-ml beaker.

References

Kilmer (1960).

Dichromate titration*

6C1a

Reagents

Mercuric chloride ($HgCl_2$), saturated aqueous solution.

Phosphoric acid (H_3PO_4), 85-percent.

Potassium dichromate ($K_2Cr_2O_7$), 0.100N, standard.

Barium diphenylaminesulfonate, 0.16-percent aqueous solution.

Procedure

Add 10 to 15 ml H_2O_2 (6C1) to the solution to destroy any excess reducing agent. Cover the beaker with a watchglass and warm on a hot plate until the reaction starts. Set the solution aside until the reaction subsides and then boil for 10 to 15 minutes. Add a slight excess of 1:1 NH_4OH and boil the solution for 15 to 20 minutes to insure complete removal of H_2O_2 . Dissolve $Fe(OH)_3$ by adding 1:1 HCl through the lip of the beaker. Usually 10 to 15 ml are enough. Heat the solution to 90° C and reduce by adding $SnCl_2$ by drops, stirring until the yellow color just disappears. Add three to four drops more. Cool the solution to room temperature and add 15 ml $HgCl_2$ solution all at once. A light silky precipitate of Hg_2Cl_2 forms if the proper amount of $SnCl_2$ has been added. Dilute the solution to 100 to 150 ml and add 5 ml H_3PO_4 . Add 10 drops of barium diphenylaminesulfonate and titrate the solution with standard $K_2Cr_2O_7$ to a violet-blue end point.

Calculations

$$Fe \text{ (pct.)} = \frac{\text{ml } K_2Cr_2O_7}{\text{g sample}} \times N \text{ of } K_2Cr_2O_7 \times \frac{\text{ml extract}}{\text{ml aliquot}} \times 5.58$$

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

Report on oven-dry basis.

EDTA titration*

6C1b

Reagents

Hydrogen peroxide (H_2O_2), 35-percent.

Ammonium persulfate ($(NH_4)_2S_2O_8$).

Salicylic acid, 1-percent in 95-percent ethanol.

EDTA, standardized as g iron per ml EDTA.

Prepare EDTA as described in 6N1a.

Iron standard, 0.500 g iron per liter.

Procedure

Pipet a 5- to 25-ml aliquot from the centrifuge tube of procedure 6C1 into a 250-ml beaker. Add 50 ml water to the beaker. Then add by drops 5 ml H_2O_2 and digest over low heat until bubbling from the decomposing H_2O_2 ceases. Remove immediately to avoid precipitation of Fe_2O_3 in samples high in iron. Caution: Add H_2O_2 slowly to prevent liberation of elemental sulfur from any remaining $Na_2S_2O_4$. Keep the volume in the beaker

oxidation of iron. Then add 1 ml indicator (1-percent salicylic acid) and titrate with 0.02N EDTA to a pale yellow or colorless end point.

Calculations

$$\text{Fe (pct.)} = \frac{\text{ml EDTA}}{\text{g sample}} \times V \times \frac{\text{ml extract}}{\text{ml aliquot}} \times 100$$

$$\text{Fe}_2\text{O}_3 \text{ (pct.)} = \text{pct. Fe} \times 1.43$$

where

V is the titer of EDTA in g Fe/ml EDTA

Report on oven-dry basis.

References

Cheng, Bray, and Kurtz (1953).

Dithionite-citrate extraction 6C2

Reagents

Sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$).
Sodium citrate.

Procedure

Weigh 1 to 4 g 80-mesh soil (approximately 0.2 g free-iron maximum) into an 8-oz nursing bottle. Add 2 g sodium dithionite and 20 to 25 g sodium citrate. Add water to 4 oz and shake overnight in a reciprocating shaker.

Orthophenanthroline colorimetry 6C2a

Apparatus

Seligson pipet, 0.1-ml.

Reagents

Orthophenanthroline, 0.25-percent.
Iron solution, 1,000 mg per liter, standard.
Sodium dithionite powder ($\text{Na}_2\text{S}_2\text{O}_4$).
Sodium citrate crystals.
Superfloc flocculating agent, 0.2-percent, in water.

Procedure

Add 5 drops Superfloc to the dithionite-treated soil suspension (6C2) and make to 8 oz. Shake vigorously for about 15 seconds and allow to settle. Pipet a 0.1-ml aliquot with a Seligson pipet into a 25-ml volumetric flask. Add water to about 10 ml. Using a small scoop, tap a pinch of dithionite and a pinch of sodium citrate into the flask. Add 0.5 ml 0.25-percent orthophenanthroline and make to volume. Shake and read in a colorimeter at 508 m μ after 1 hour. To prepare the standards, pipet 5-, 10-, 25-, 50-, 100-, 150-, and 200-ml aliquots of standard iron solution (1,000 mg/l) into 8-oz shaking bottles and make to 8 oz after adding reagents as in 6C2. Transfer 0.1-ml aliquots to 25-ml volumetrics and develop color by the above procedure.

Plot the standard curve as milligrams iron per 8-oz bottle against percentage transmission.

Calculations

$$\text{Fe (pct.)} = \frac{\text{mg Fe in bottle}}{\text{g sample}} \times 10^{-1}$$

$$\text{Fe}_2\text{O}_3 \text{ (pct.)} = \text{pct. Fe} \times 1.43$$

Report on oven-dry basis.

References

Holmgren (1967).

Atomic absorption 6C2b

Apparatus

Dilutor or Seligson pipet, 0.5 ml.
Perkin-Elmer Model 290 atomic absorption spectrophotometer.

Reagents

Iron solution 1,000 mg per liter, standard (optionally 2,000 mg/l).

Superfloc flocculating agent, 0.2 percent in water. Shake intermittently for several days to bring into solution.

Procedure

Bring the volume of the dithionite-treated soil suspension to 8 oz with water. Add 5 drops Superfloc, stopper, and shake vigorously for 15 seconds. Allow to settle for 1 hour. Or if a pinch of dry Superfloc is added, allow to settle 1 to 3 hours. Dilute the supernatant tenfold to fortyfold with the dilutor or the Seligson pipet.

Determine the flame absorption of the sample at 2,483 Å and compare with the absorption of standard solutions as prepared in 6C2a.

Calculations

$$\text{Fe (pct.)} = \frac{\text{mg Fe in bottle}}{\text{g sample}} \times \frac{\text{dilution}}{10}$$

$$\text{Fe}_2\text{O}_3 \text{ (pct.)} = \text{pct. Fe} \times 1.43$$

Report on oven-dry basis.

Dithionite-citrate-bicarbonate extraction 6C3

Reagents

Sodium bicarbonate (NaHCO_3), 1M.
Sodium citrate, 0.3M.
Sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$).
Sodium chloride (NaCl), saturated solution.
Acetone.

Procedure

Weigh 4 g soil (1 g clay) into a 100-ml centrifuge tube. Add 40 ml 0.3M Na-citrate and 5 ml 1M NaHCO_3 . Bring temperature to 80° C in water bath. Add 1 g solid $\text{Na}_2\text{S}_2\text{O}_4$, stir constantly for 1 minute and occasionally for 15 minutes. Add 10 ml NaCl solution and 10 ml acetone to promote flocculation. Mix, warm in water bath, and centrifuge 5 minutes at 1,600 to 2,200 rpm. Decant clear supernatant into 500-ml volumetric flask and make to volume.

References

Mehra and Jackson (1960).

Potassium thiocyanate colorimetry 6C3a

Apparatus

Colorimeter.

Reagents

Hydrochloric acid (HCl), 6*N*.
Potassium thiocyanate (KSCN), 20-percent.
Hydrogen peroxide (H₂O₂), 30-percent.

Procedure

Transfer suitable aliquot (0.5 to 3 ppm iron in final solution) to 50 ml-volumetric flask. Add water to 35 ml, 1 drop H₂O₂, 5 ml HCl, and 5 ml KSCN solution. Make to volume and read at 490 mμ in colorimeter.

Calculations

$$\text{Fe (pct.)} = \frac{\text{mg/Fe from curve}}{\text{g sample}} \times \frac{\text{ml extract}}{\text{ml aliquot}} \times 0.005$$

$$\text{Fe}_2\text{O}_3(\text{pct.}) = \text{Fe (pct.)} \times 1.43$$

Report on overdry basis.

References

Jackson (1958).

Pyrophosphate-dithionite extraction 6C4

Reagents

Pyrophosphate solution.—Dissolve 89.2 g Na₄P₂O₇·10H₂O in 800 to 900 ml water. Adjust the pH of this solution to 8.0 by adding hydrogen-saturated exchange resin. Decant or filter, wash the resin, and dilute the solution to 1,000 ml to make 0.2*M* Na₄P₂O₇.

Sodium dithionite (Na₂S₂O₄).

Digestion acid.—10 parts concentrated HNO₃, 4 parts concentrated H₂SO₄, and 4 parts concentrated HClO₄.

Procedure

Mix 80 ml pyrophosphate solution and 2.0 g solid sodium dithionite in a beaker and add this solution to 4 g soil in a centrifuge tube (pH 8.0 pyrophosphate solution and dithionite combined

References

Franzmeier, Hajek, and Simonson (1965).

Sodium pyrophosphate extraction 6C5

Reagents

Sodium pyrophosphate (Na₄P₂O₇), 0.1*M*.
Superfloc solution, 0.4 percent.

Procedure

Place 2 g soil into 250-ml centrifuge bottle (polypropylene). Add 200 ml 0.1*M* Na₄P₂O₇, cap, and shake overnight. Add 5 to 10 drops 0.4-percent Superfloc, shake, and centrifuge at 2,000 rpm (Int. No. II centrifuge). Transfer the supernatant liquid to a plastic or glass container and reserve for Fe and Al analyses.

The supernatant liquid must be clear in reflected light. If fine colloids are visible, repeat the procedures. If fine colloids are still present, spin the suspension in a super centrifuge until the supernatant liquid is clear. Foam rubber can be used in the centrifuge cups as a cushion for the 250-ml flat-bottom plastic bottles.

References

Bascomb (1968).

Atomic absorption 6C5a

Apparatus

Atomic absorption spectrophotometer.

Reagents

Standard Fe solution, 0 to 50 ppm.

Procedure

Establish standard curve and match readings from extract to curve readings. Dilute where necessary.

Calculations

$$\text{Fe (pct.)} = \text{ppm Fe} \times \frac{\text{ml extract}}{\text{g soil}} \times \frac{1}{10,000} \times \text{dilution}$$

Report on overdry basis.

Ammonium oxalate extraction 6C6

Reagents

The supernatant liquid must be clear in reflected light. If fine colloids are visible repeat the procedure. If fine colloids are still present, spin the suspension in a supercentrifuge until the supernatant liquid is clear.

References

References

Jackson (1958).

CALCIUM CARBONATE

6E

HCl treatment

6F1

Gravimetric (weight loss)

6E1c

Apparatus

Figure 7.

- A Glass wool plugs
- B Anhydrous $Mg(ClO_4)_2$
- C Vial containing 6*N* HCl
- D Stopcock
- E Stopcock
- F 125-ml Erlenmeyer flask
- G Stopper
- H U tube
- I Calcium chloride tube (shortened)
- J Glass tube

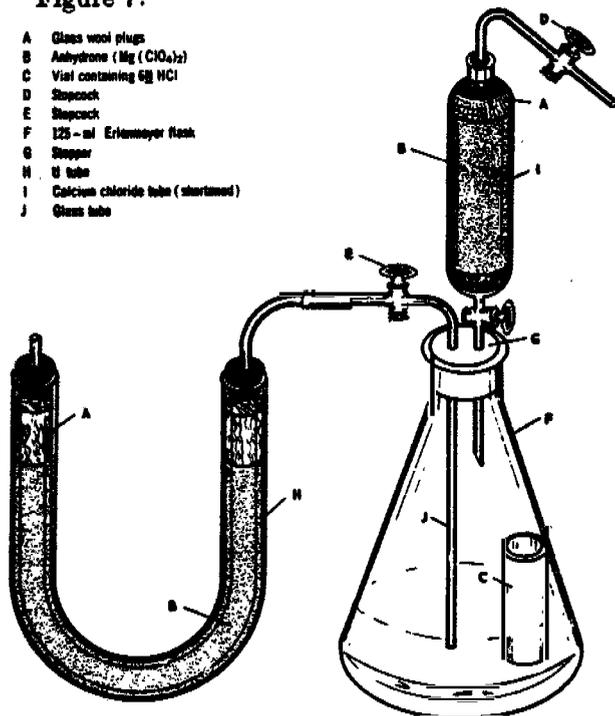


FIGURE 7.—Apparatus used for calcium carbonate determination by weight loss (6E1c).

stopcock D and then shake apparatus to upset the vial, allowing the acid to react with the carbonates. After 10 minutes, attach the rubber tube from the air-drying vessel to stopcock E. Open stopcock E and apply suction at stopcock D to give 5 to 10 bubbles per second at the base of tube J to sweep out CO_2 . Shake the flask after 10 minutes and again after 20 minutes. After 30-minute sweeping time, stop the suction and close stopcocks D and E. Return apparatus to the balance. Delay weighing for 1 hour to allow the heat generated by absorption of water by the anhydrous to be dissipated. Weigh apparatus with stopcock D open. Check the weight after 10 minutes.

Calculations

Carbonate as $CaCO_3$ (pct.)

$$\frac{\text{initial g flask} - \text{final g flask}}{\text{g sample}} \times 228$$

Report on oven-dry basis.

References

Erickson et al. (1947).

Gravimetric (weight gain)

6E1d

Proceed as in 6E1c except add additional trap containing CO_2 -absorbing Ascarite to end of gas train. Weigh Ascarite bulb before and after CO_2 evolution. Weight gain equals the CO_2 evolved from the sample. Better results are obtained if the Ascarite is size-graded so that CO_2 passes through the ~~coarser~~ material first. Indicarb can be used in

Warburg method 6E1f

Apparatus

Warburg manometer, mercury filled.

Warburg reaction vessel, 15-ml capacity, with vented stopper for sidearm.

Constant temperature bath.

Reagents

HCl, 1:1.

Na₂CO₃ solution for standard curve.—Dissolve 1.06 g Na₂CO₃ in water and make to 1 liter. Solution contains 1.06 mg Na₂CO₃ per ml or the equivalent of 1 mg CaCO₃ per ml. Obtain standard curve by measuring CO₂ pressure from 1, 2, 4, 6, 8, and 10 ml Na₂CO₃ solution.

Procedure

Weigh 100 mg sample of finely ground soil and transfer to Warburg reaction vessel. Be careful not to get any sample in center well. Pipet 1 ml water into vessel and mix well with sample. Pipet 1 ml 1:1 HCl into sidearm, insert greased stopper, and leave in vent-open position. Attach reaction vessel to manometer and fasten with rubber bands or spring supports. Place reaction vessel in constant temperature bath at 25° C for 5 to 10 minutes to bring flask contents to temperature of water bath.

Remove flask from bath, close stopper vent, and fasten with rubber bands or springs. Tilt flask to allow acid to flow from sidearm into reaction vessel, mix contents, and return vessel to water bath. Let stand for at least 30 minutes before reading manometer. Use the standard curve to convert the difference between the two manometer arm readings (mm), to milligrams CaCO₃.

Gently tap the manometer holder occasionally to prevent low readings caused by mercury adhering to manometer walls.

Sensitive qualitative method 6E2

Visual, gas bubbles 6E2a

Add few drops 6N H₂SO₄ to soil and observe under binocular microscope. Evolution of gas bubbles indicates the presence of CaCO₃.

H₂SO₄ treatment 6E3

Gravimetric (weight gain) 6E3a

Reagents

Sulfuric acid (H₂SO₄).

Dissolve 57 ml concentrated H₂SO₄ and 92 g of FeSO₄·7H₂O in 600 ml water, cool, and dilute to

the Nesbitt bulb, attach to the system, and adjust the carrier stream to a flow rate of 1 or 2 bubbles per second. Pour 25 ml of the acid solution into the funnel and let it enter the digestion flask E. Close the stopcock immediately. Apply heat slowly and bring contents of flask to a boil in about 4 minutes. Continue gentle boiling for exactly 3 minutes more for a total heating period of 7 minutes. Remove the flame, adjust the carrier stream to 6 or 8 bubbles per second, and continue aerating for 10 minutes. Disconnect the Nesbitt bulb and weigh.

Calculations

Carbonate as CaCO₃ (pet.)

$$\frac{\text{final g bulb} - \text{initial g bulb}}{\text{g sample}} \times 227$$

Report on oven-dry basis.

References

Allison (1960).

GYPSUM 6F

Water extract 6F1

Precipitation in acetone 6F1a

Reagents

Acetone (CH₃COCH₃).

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 minutes in a test-tube rack; run quantitatively all samples in which a precipitate forms, indicating 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 (Solu-bridge reading >0.90), repeat, using a 1:10 or greater dilution. Stopper the bottle and shake in a reciprocating mechanical shaker for 30 minutes. Filter the 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.

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 CaSO₄ 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 (Mhos/cm)
1	0.121
2	.226
5	.500
10	.900
20	1.584
30.5	2.205

Calculations

CaSO₄ in aliquot (meq)

$$= \text{meq/l CaSO}_4 \text{ from curve} \times \frac{\text{ml H}_2\text{O to dissolve ppt.}}{1,000}$$

CaSO₄ in soil (meq/100 g)

$$= \frac{\text{meq CaSO}_4 \text{ in aliquot}}{\text{soil:water ratio}} \times \frac{100}{\text{ml aliquot}}$$

CaSO₄ as gypsum (pct.)

$$= \text{meq/100 g CaSO}_4 \times 0.086$$

Report on oven-dry basis.

References

Richards (1954).

Indirect estimate

6F1b

Procedure

Add a weighed quantity of soil to enough water to dissolve all the gypsum by overnight shaking. Determine total sulfate in this extract by any appropriate procedure. Also determine Ca and SO₄ in a saturation extract by any appropriate procedure.

Calculations

$$\text{Gypsum} = (\text{SO}_4)_{\text{DE}} - (\text{SO}_4)_{\text{NO GYPSUM SE}}$$

but

$$\text{SO}_4_{\text{NO GYPSUM SE}} = (\text{SO}_4)_{\text{SE}} - (\text{SO}_4)_{\text{GYPSUM SE}}$$

∴

$$\text{Gypsum} = (\text{SO}_4)_{\text{DE}} + (\text{SO}_4)_{\text{GYPSUM SE}} - (\text{SO}_4)_{\text{SE}}$$

where

$$(\text{SO}_4)_{\text{DE}} = \text{SO}_4 \text{ in dilute water extract}$$

$$(\text{SO}_4)_{\text{SE}} = \text{SO}_4 \text{ in saturation extract}$$

$$(\text{SO}_4)_{\text{GYPSUM SE}} = 30 \text{ meq/l if SO}_4 \text{ and Ca are } \geq 30 \text{ meq/l}$$

$$= (\text{SO}_4)_{\text{SE}} \text{ if } (\text{Ca})_{\text{SE}} > (\text{SO}_4)_{\text{SE}}$$

$$= (\text{Ca})_{\text{SE}} \text{ if } (\text{Ca})_{\text{SE}} < (\text{SO}_4)_{\text{SE}}$$

All quantities expressed in meq/100 g.

$$\text{Gypsum (pct.)} = \text{meq/100 g} \times 0.0861$$

References

Lagerwerff, Akin, and Moses (1965).

ALUMINUM

6G

KCl extraction I (30 min)

6G1

Reagents

Potassium chloride (KCl), 1N.

Procedure

Weigh 10-g soil samples into 125-ml Erlenmeyer flasks. Add 50 ml 1N KCl to each flask, mix several times, and let stand for 30 minutes. Filter through 5.5-cm Whatman No. 42 filter paper in Büchner funnel, using suction as necessary. Leach each sample as rapidly as possible with about five 9-ml portions of KCl, using the first to help transfer the remaining soil in the Erlenmeyer flasks to the Büchner funnels. Transfer the extract to 100-ml volumetric flasks and dilute to volume with the extracting solution. Or use Allihn leaching tubes and bring to standard weight in tared suction flasks.

References

Lin and Coleman (1960) and Pratt and Bair (1961).

Aluminon colorimetry I, hot color development*

6G1a

Reagents

Thioglycolic acid (HSCH₂COOH).—Dilute 1 ml purified acid to 100 ml with water.

Aluminon reagent.—Dissolve in separate containers 0.75 g Aluminon (ammonium aurine tricarboxylate), 15 g gum acacia, and 200 g NH₄OAc crystals. To the NH₄OAc solution add 189 ml concentrated HCl, then the gum acacia, and finally the Aluminon. Mix, filter, and dilute to 1,500 ml with water. To get the gum acacia in suspension, add slowly to boiling water while stirring constantly.

Aluminum standard.—Add 2.24 g AlCl₃·6H₂O per liter of water. This solution should be nearly 250 ppm aluminum. Check concentration of an aliquot containing 10 ppm aluminum by analyzing for chloride.

Procedure

If samples contain less than 5 meq per 100 g aluminum, pipet a 1-ml aliquot of each extract into numbered and calibrated test tubes. If more aluminum is present, dilute before the aliquot is taken. Dilute to approximately 20 ml with distilled water. Add 2 ml dilute thioglycolic acid to each tube, stopper, and shake all the tubes. Pipet 10 ml Aluminon into each tube and dilute to exactly 50 ml. The pH should be between 3.7 and 4.0. Stopper and shake all tubes. Place tubes in a rack and heat in a boiling-water bath for 4 minutes. Cool in running water to room temperature. Transfer samples to reading tubes and measure light transmittance at 535 mμ and compare with a standard curve.

Calculations

$$\text{Al (meq/100 g)} = \frac{\text{mg/l Al from curve}}{\text{g sample}} \times \frac{\text{ml extract}}{\text{ml aliquot}} \times \frac{9}{5}$$

Report on oven-dry basis.

References

Chenery (1948) and Yoe and Hill (1927).

Aluminon colorimetry II,
HCl predigestion **6G1b**

Procedure

Proceed as in 6G1a but first add 3 ml *N* HCl to the aliquot and heat for 30 minutes at 80° to 90° C.

References

Hsu (1963).

Aluminon colorimetry III,
overnight color development **6G1c**

Proceed as in 6G1a except eliminate boiling-water bath, adjust pH to 4.0, and allow color to develop overnight before reading.

Fluoride titration **6G1d**

Reagents

Potassium fluoride (KF), 1*N*.—Titrate with NaOH to a phenolphthalein end point. This eliminates the need for a blank correction in the Al titration.

Sodium hydroxide (NaOH), 0.1*N*, standardized.
Sulfuric acid (H₂SO₄), 0.1*N*, standardized.
Phenolphthalein, 0.1 percent.

Procedure

Add 6 to 8 drops phenolphthalein to the leachate in the suction flask (6G1). Titrate with standard NaOH to a pink color that persists for 30 seconds or more. Correct for a KCl blank to obtain KCl-extractable acidity. Then add 10 ml KF, and titrate with standard H₂SO₄ until the pink color disappears. Set aside while other samples are titrated and then complete to a lasting colorless end point. If there is a considerable amount of Al, add a few more drops of phenolphthalein.

Calculations

$$\text{Acidity (meq/100 g)} = \frac{\text{ml NaOH}}{\text{g sample}} \times N \text{ of NaOH} \times 100$$

$$\text{Al (meq/100 g)} = \frac{\text{ml H}_2\text{SO}_4}{\text{g sample}} \times N \text{ of H}_2\text{SO}_4 \times 100$$

References

Yuan (1959).

Atomic absorption **6G1e**

Apparatus

Perkin-Elmer Model 290 atomic absorption spectrophotometer with nitrous oxide burner attachment.

Reagents

Standard Al solution, 0 to 5 meq per liter.

Procedure

Dilute sample to within range of standard curve. Compare absorbance with standard curve.

Calculations

$$\text{Al (meq/100 g)} = \frac{\text{meq/l from curve}}{\text{g soil}} \times \text{dilution} \times \frac{\text{ml extract}}{10}$$

KCl extraction II, overnight **6G2**

Weigh 10 g soil into 125-ml Erlenmeyer flask. Add 50 ml 1*N* KCl and let stand overnight. In the morning transfer to filter funnels and leach with an additional 50 ml KCl.

Aluminon colorimetry I **6G2a**

Follow procedure for aluminum analysis described in 6G1a.

NH₄OAc extraction **6G3**

Prepare soil as described in 5A1.

Aluminon colorimetry III **6G3a**

Follow procedure of 6G1c.

NaOAc extraction **6G4**

Prepare soil as described in 5A2.

Aluminon colorimetry III **6G4a**

Follow procedure of 6G1c.

Sodium pyrophosphate extraction **6G5**

Prepare extract as described in 6C5.

Atomic absorption **6G5a**

Apparatus

Atomic absorption spectrophotometer.

Reagents

Standard Al solution, 0 to 50 ppm or 0 to 160 ppm.

Procedure

Establish standard curve and match readings from extract to curve readings. Dilute where necessary.

Calculations

$$\text{Al (pct.)} = \text{ppm Al} \times \frac{\text{ml extract}}{\text{g soil}} \times \frac{1}{10,000} \times \text{dilution}$$

Report on oven-dry basis.

Ammonium oxalate extraction **6G6**

Prepare extract as described in 6C6.

Atomic absorption **6G6a**

Analyze extract as described in 6G5a.

Dithionite-citrate extraction 6G7

Prepare extract as described in 6C2.

Atomic absorption 6G7a

Analyze extract as described in 6G5a.

EXTRACTABLE ACIDITY⁹ 6H

BaCl₂-triethanolamine I 6H1

Reagents

Buffer solution.—Barium chloride, 0.5*N*, and triethanolamine, 0.2*N*. Add about 90 ml, 1*N* HCl per liter to adjust pH to 8.2. Protect the buffer solution from CO₂ 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 buffer solution per liter. Protect the replacement solution from CO₂ of the air by attaching a drying tube similar to that used for the buffer solution.

Procedure

Weigh 5 g soil into a 125-ml Erlenmeyer flask. Add 15 ml buffer solution and let stand for 30 minutes, swirling occasionally to mix. Use 35 ml buffer solution to transfer all the soil solution to a No. 4 Gooch crucible containing a moist Whatman No. 540 filter paper and filter into a 500-ml suction flask. The rate of filtration should be such that at least 30 minutes is needed to complete the filtering and leaching. Then leach the soil with 100 ml of the replacement solution, adding small amounts at a time. It may be necessary to use a larger amount of buffer solution to leach allophanic soils high in organic matter with extractable acidity of more than 35 meq per 100 g.

Back-titration with HCl. 6H1a

Reagents

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

Brom cresol green, 0.1-percent aqueous solution.

Mixed indicator.—Dissolve 1.250 g methyl red indicator and 0.825 g methylene blue in 1 liter 90-

Calculations

EA (meq/100 g)

$$\frac{\text{ml HCl blank} - \text{ml HCl sample}}{\text{g sample}} \times N \text{ of HCl} \times 100$$

Report on oven-dry basis.

References

Peech et al. (1947).

BaCl₂-triethanolamine II 6H2

Apparatus

Sulfur absorption tubes.

Whatman No. 41 filter paper or glass-fiber filter paper cut to fit sulfur absorption tubes.

Reagents

Buffer solution.—BaCl₂, 0.5*N*, and triethanolamine, 0.2*N* as in 6H1.

Mixed indicator.—Dissolve 1.250 g methyl red and 0.825 g methylene blue in 1 liter 90-percent ethanol.

Celite.

Procedure

Stopper bottom of sulfur absorption tubes with medicine-dropper bulbs and fit to a 300-ml suction flask with a rubber stopper. Place Whatman No. 41 filter paper in bottom of absorption tube, cover with 1/4 inch of acid-washed sand, and add exactly 25 ml buffer solution. Weigh 10 g soil and mix with teaspoonful of Celite. Add to the absorption tube by means of a funnel. After 30 minutes remove the medicine-dropper bulbs, wash bulbs out with a little water, and add washings to absorption tubes. Leach with 25 ml more buffer solution and then leach with 100 ml replacement solution in small increments. If necessary, use suction to facilitate leaching.

Back-titration with HCl 6H2a

Reagents

Same as in 6H1a.

Procedure

Titrate with standard HCl, using either 2 drops

acid to leachate and washings and back-titrate with standard alkali (NaOH). Titrate an equal volume of acid to the same end point for a blank.

Calculations

EA (meq/100 g)

$$\frac{\text{ml NaOH blank} - \text{ml NaOH sample}}{\text{g sample}} \times N \text{ of NaOH} \times 100$$

References

North-Central Regional Research Committee (1955).

CARBONATE 6I

Saturation extract 6II

Prepare saturation extract as directed in 8A1 or 8B1.

Acid titration 6IIa

Reagents

Sulfuric acid (H₂SO₄), 0.05N, standardized.
Phenolphthalein.

Procedure

Pipet an appropriate aliquot¹⁰ of saturation extract into a 250-ml Erlenmeyer flask or a porcelain crucible. Make volume to 50 ml (10 ml for porcelain crucible) with water. To the 50 ml in the Erlenmeyer flask, add a drop or two of phenolphthalein. If a pink color is produced, titrate with 0.05N H₂SO₄, adding a drop every 2 or 3 seconds until the pink color disappears. Use this solution to determine bicarbonate (6J1a).

Calculations

$$\text{Carbonate (meq/l)} = \frac{\text{ml H}_2\text{SO}_4}{\text{ml aliquot}} \times N \text{ of H}_2\text{SO}_4 \times 2,000$$

References

Association of Official Agricultural Chemists (1945) and Richards (1954).

BICARBONATE 6J

Saturation extract 6J1

Prepare saturation extract as directed in 8A1.

Acid titration 6J1a

Reagents

Sulfuric acid (H₂SO₄), 0.05N, standardized.
Methyl orange, 0.01-percent aqueous solution.

¹⁰ The electrical conductivity (EC×10³) of the saturation extract (8A1a) can be used to determine the aliquot to be used for carbonate, bicarbonate, and chloride determinations. Where EC×10³ is 1.0 or less, use a 10-ml aliquot; if 1.0 to 10.0, use a 5-ml aliquot; if more than 10.0, use a 2-ml aliquot.

Procedure

Use solution remaining from carbonate titration (6I1a). To the colorless solution from this titration or to the original solution if no color is produced with phenolphthalein, add 4 drops methyl orange and continue titration to the methyl orange end point without refilling the buret. Retain this solution for the chloride determination (6K1a). Make a blank correction for the methyl orange titration.

Calculations

Bicarbonate (meq/l)

$$\frac{\text{Total ml H}_2\text{SO}_4 - 2 \times \text{ml H}_2\text{SO}_4 \text{ (from 6I1a)}}{\text{ml aliquot}} \times N \text{ of H}_2\text{SO}_4 \times 1,000$$

References

Association of Official Agricultural Chemists (1945) and Richards (1954).

CHLORIDE 6K

Saturation extract 6K1

Prepare saturation extract as described in 8A1 or 8B1.

Mohr titration 6K1a

Reagents

Potassium chromate (K₂CrO₄) indicator.—Dissolve 5 g K₂CrO₄ in water and add a saturated solution of AgNO₃ until a permanent slight red precipitate is produced, filter, and dilute to 100 ml.

Silver nitrate (AgNO₃), 0.05N, standardized.

Sodium bicarbonate (NaHCO₃), saturated solution (optional).

Nitric acid (HNO₃), 0.1N (optional).

Procedure

To the solution from the bicarbonate titration (6J1a) add 6 drops K₂CrO₄ indicator and titrate with AgNO₃ to a reddish-orange end point. Make a correction with a blank of 50 ml water containing the indicators of both titrations. The laboratory at Riverside, Calif., modifies this procedure by adding saturated NaHCO₃ solution to a pink end point and neutralizing to a colorless end point with HNO₃ before adding the indicator.

Calculations

Chloride (meq/l)

$$\frac{\text{ml AgNO}_3 \text{ sample} - \text{ml AgNO}_3 \text{ blank}}{\text{ml aliquot}} \times N \text{ of AgNO}_3 \times 1,000$$

References

Association of Official Agricultural Chemists (1945).

Potentiometric titration***6K1b****Apparatus**

Silver Billet combination electrode, No. 39187.
Zeromatic pH meter (expanded scale).

Reagents

Standard silver nitrate (AgNO_3), 0.025*N*.

Buffer solutions.—Either potassium acid phthalate or trisodium citrate and citric acid.

To prepare phthalate buffer, weigh 37.5 g potassium acid phthalate and bring to a volume of 500 ml with water; 4 ml of this buffer added to a 46-ml solution brings the pH to about 4.

To prepare trisodium citrate buffer, weigh 43.8 g trisodium citrate and 43.3 g citric acid into 500-ml volumetric flask and bring to volume with water. Add a small amount of toluene to the solution for storage; 10 ml of this buffer added to a 40-ml solution brings pH to about 4.

Procedure

Standardize the pH meter by adjusting the needle to a convenient setting (about 0.8) on the expanded scale when the electrode is immersed in buffer solution (4 or 10 ml made to 50 ml) without chloride. To titrate the sample, pipet an aliquot containing as much as 2.0 meq chloride into a beaker and add 4 ml buffer. Make to 50 ml. Immerse the electrode and buret tip into the beaker and titrate with AgNO_3 to the end point previously established for the buffer without chloride.

Calculations

$$\text{Chloride (meq/l)} = \frac{\text{ml AgNO}_3}{\text{ml aliquot}} \times N \text{ of AgNO}_3 \times 1,000$$

SULFATE**6L****Saturation extract****6L1****Calculations**

$$\text{SO}_4 \text{ (meq/l)} = \frac{\text{mg BaSO}_4}{\text{ml aliquot}} \times 8.568$$

References

Richards (1954).

EDTA titration**6L1b****Apparatus**

Repipet, automatic dilutor, pipet range 0.1 to 1.0 ml.

Titration assembly including a 10-ml buret with magnetic stirrer.

Reagents

Thymol blue indicator, 0.04-percent.

Nitric acid (HNO_3), 0.4*N*.

Calcium nitrate ($\text{Ca}(\text{NO}_3)_2$), 0.05*N*. Dissolve 5.90 g $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ in 1 liter CO_2 -free water. EC is 5.15 ± 0.15 mmhos per cm at 25° C.

Acetone, reagent grade, boiling range 55.5 to 57.5° C.

Ethanol, 95-percent, reagent grade.

Hydrochloric acid (HCl), 0.01*N*.

EDTA solution, 0.02*N*.—Standardize against CaCl_2 .

Procedure

Pipet an aliquot containing 0.01 to 0.05 meq SO_4 from soil-water extracts and transfer to a 100-ml beaker. Bring volume to 7.5 ± 0.5 ml with water. Add 2 drops 0.04-percent thymol blue and 0.4*N* HNO_3 drop by drop until color changes from yellow to distinct red. Add 2 ml 0.05*N* $\text{Ca}(\text{NO}_3)_2$, 20 ml acetone, and stir. Allow 30 minutes for the precipitate to flocculate. Place a 9.0-cm Whatman No. 42 filter paper in a 5.0-cm fluted funnel and fit snugly with water. Wash the sides of filter paper

NH₄OAc extraction

Obtain extract by procedure 5B1.

Gravimetric, BaSO₄ precipitation

Proceed as in 6L1a. A greater quantity of acid will be needed to lower the pH. Otherwise the procedures are the same.

NITRATE

Saturation extract

Prepare saturation extract as described in 8A1 or 8B1.

Phenoldisulfonic acid colorimetry

6L2

anions in the extract. A quantitative measurement can be made if there is a positive indication of NO₂ (6M1a).

6L2a

Apparatus

Dropper pipet.
White spot plate.

Reagents

Diphenylamine in H₂SO₄.—Dissolve 0.05 g diphenylamine in 25 ml concentrated sulfuric acid. Store in polyethylene dropper bottle.

Procedure

6M

6M1

6M1a

Atomic absorption

6N1b

Apparatus

Perkin-Elmer Model 290 atomic absorption spectrophotometer.

Hollow cathode, multielement Ca, Mg tube.
Diluter.

Reagents

Standard Ca solution, 0-0.5 meq per liter.

for the precipitate and solution to 250-ml Erlenmeyer flasks. Dilute the solution to a total volume of about 100 ml. Place the sample on a magnetic stirrer, add 5 ml 10-percent NaOH, 2 drops Calcon indicator solution, and titrate with the standard EDTA solution to the blue color of a blank carried through the procedure. The pH of the solution should be about 12.5. The color change is from red to clear blue. Titrate until the color in the sample and in the blank are the same.

imately 4.6 by slowly adding *N* NH_4OH , stirring constantly. Let digest at about 80°C for 1 hour or until the supernatant liquid is clear. Collect the CaC_2O_4 precipitate on a compact asbestos pad in a Gooch crucible or in a Whatman No. 42 filter paper in filter funnel. Rinse the beaker four times with water or water saturated with CaC_2O_4 and pour the washings into the crucible. Wash the precipitate five more times with water saturated with CaC_2O_4 .

Remove the Gooch crucible from its holder, rinse the outside, and replace crucible in the beaker. If filter paper is used, pierce the paper and wash most of the precipitate into the beaker with 3.6*N* H_2SO_4 . Wash off excess H_2SO_4 with water and place filter paper on watchglass. Add 100 ml water and 7 ml concentrated H_2SO_4 . Heat to 90°C and stir until CaC_2O_4 is dissolved. Titrate with standard KMnO_4 solution to a pink color. Add filter paper to solution and titrate to a permanent pink color.

Calculations

Ca (meq/100 g)

$$= \frac{\text{ml } \text{KMnO}_4}{\text{g sample}} \times N \text{ of } \text{KMnO}_4 \times \frac{\text{ml extract}}{\text{ml aliquot}} \times 100$$

Report on oven-dry basis.

References

Peech et al. (1947).

Oxalate precipitation II, KMnO_4 titration (Fe, Al and Mn removed)* 6N2c

Proceed as in 6N2b but after muffle treatment and before oxalate precipitation, remove iron, aluminum, and manganese by the following procedure.

Reagents

Hydrochloric acid (HCl), 6*N*.

Ammonium hydroxide (NH_4OH), 2*N*.

Bromine water, saturated.

Ammonium chloride (NH_4Cl), 6*N*.

Concentrated nitric acid (HNO_3).

Procedure

Dissolve salts and oxides by adding 5 ml 6*N* HCl and heating on a hot plate until all salts and oxides are in solution. Add 75 to 100 ml water and heat the solution until it is nearly boiling. Immerse the pH electrodes into the hot solution and precipitate the hydroxides of iron, aluminum, and titanium by slowly adding 2*N* NH_4OH until the meter indicates a pH of 6.2 to 6.4. Add 2 more drops of NH_4OH to neutralize the acidifying effect of the 15 ml saturated bromine water, which is slowly added next to precipitate manganese hydroxide. Since bromine water lowers the pH of the solution, readjust it to 6.2 to 6.4 with 2*N* NH_4OH . Heat the solution with precipitate until it just begins to boil (1 or

2 min on a Bunsen burner) and remove from the heat.

Place on a hot plate at a temperature of 80° to 90°C for 1 hour. Filter when the breaker has cooled enough to handle easily. Use an 11-cm Whatman No. 42 filter paper or its equivalent. Collect the filtrate in a beaker of the same size as those used for precipitating calcium. Wash and police the beaker containing the precipitate with hot 2-percent NH_4Cl . Wash the precipitate on the filter with the same solution. Five washings are usually enough. To the filtrate add 10 ml concentrated HNO_3 and evaporate to dryness; add 5.0 ml 6*N* HCl , take to dryness, and use high heat to dehydrate silica. Proceed with the calcium precipitation (6N2b).

References

Washington (1930) and Fieldes et al. (1951).

Oxalate precipitation, cerate titration* 6N2d

Proceed as in 6N2b except substitute the following for the permanganate titration.

Reagents

Ammonium hexanitrate cerate ($(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6$) in molar perchloric acid (HClO_4), 0.1*N*.—Add 85 ml 70- to 72-percent perchloric acid to 500 ml water. Dissolve 56 g ammonium hexanitrate cerate in the acid solution and dilute to 1 liter.

Ammonium hexanitrate cerate in molar perchloric acid, 0.05*N*.—Follow the directions for the preparation of the 0.1*N* solution but use only 28 g cerate.

Perchloric acid (HClO_4), 2*N*.—Add 170 ml 70- to 72-percent perchloric acid to 500 ml water and dilute to 1 liter.

Nitro-ferroin indicator solution.—Dilute a solution of nitro-orthophenanthroline ferrous sulfate with water to a convenient working strength. Two to four drops of the solution should give a sharp color change at the end point.

Standardize the cerate solutions against accurately weighed quantities of primary standard-grade sodium oxalate. Convenient weights of sodium oxalate are 0.10 to 0.11 g for the 0.05*N* solution and 0.10 to 0.18 g for the 0.1*N* cerate solution. Dissolve the sodium oxalate in 100 to 150 ml 2*N* perchloric acid and titrate as directed in the following procedure.

Procedure

Dissolve the filtered and washed (use water) calcium oxalate in 100 to 200 ml 2*N* perchloric acid. If a paper filter has been used, macerate it before titration. Add 2 to 4 drops of nitro-ferroin indicator solution and titrate with 0.05*N* or 0.1*N* cerate solution, depending upon the amount of oxalate present. The solution changes from red to colorless at the end point.

Calculations

$$\text{Ca (meq/100 g)} = \frac{\text{ml cerate}}{\text{g sample}} \times N \text{ of cerate} \times \frac{\text{ml extract}}{\text{ml aliquot}} \times 100$$

Report on oven-dry basis.

Atomic absorption 6N2e

Proceed as in 6N1b except use sample from NH_4OAc extract.

Calculations

Ca (meq/100 gm)

$$= \frac{\text{Ca meq/l from curve}}{\text{g sample}} \times \text{dilution} \times \frac{\text{ml extract}}{10}$$

Report on oven-dry basis.

NH_4Cl -ethanol extraction (calcareous soils) 6N3

Apparatus

Figure 8.

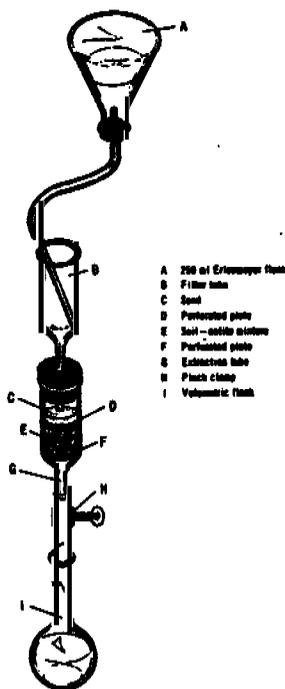


FIGURE 8.—Apparatus for ammonium chloride-ethanol extraction for calcium (6N3).

Reagents

Ammonium chloride (NH_4Cl), 1*N*, in 60-percent ethanol.—To make 9 liters of extraction solution, dissolve 482 g NH_4Cl in 2,835 ml water and add 5,985 ml 95-percent ethanol. Adjust pH to 8.5 with 140 to 145 ml NH_4OH .

Celite.

Procedure

Fill extraction tube with water, set tube upright in holder, and let most of the water drain out. Close screw clamp and place filter paper on plate with a stirring rod. Let remainder of

the water drain out of tube. The filter paper provides enough tension to keep the bottom part of the tube filled with water. Place tube on the rack and add about 1½ teaspoons washed sand. Place an extra perforated plate (inverted) on top of the sand and cover the plate with more sand. Place heaping teaspoon of Celite on the sand and pour about 20 ml extraction solution into the tube. Pour remainder of 400 ml extraction solution into a 500-ml Erlenmeyer flask. Add soil sample slowly and then stir with a rod to mix soil and Celite. Allow sample to settle and then place filter paper on top of the soil column. Put upper tube in place, stopper, and let stand overnight.

In the morning, place a 500-ml volumetric flask under the delivery tip and open screw clamp on lower extraction tube slowly. When level of liquid is a few milliliters above the soil, invert the 500-ml Erlenmeyer flask containing remainder of extraction solution (delivery tube in place), place glass tip in the upper tube, and open the pinch clamp. Use the screw clamp on lower tube to adjust flow rate through soil column. When all the extraction solution has passed through the soil column, remove volumetric flask, make to volume with water, and mix.

EDTA titration

6N3a

Pipet a 50-ml aliquot for determination of Ca and Mg into a 100-ml beaker and evaporate to dryness. Add 10-ml concentrated HNO_3 and 1 or 2 ml concentrated HCl. Cover with watchglass, place on hot plate, and heat until no more brown fumes are evolved. Remove cover glass, rinse into beaker, and evaporate solution to dryness. Take up residue with 3 ml *N* HNO_3 . Quantitatively transfer solution with ethanol to a 50-ml conical centrifuge tube and proceed with determination of Ca according to 6N2a.

References

Tucker (1954).

KCl-triethanolamine extraction*

6N4

Prepare extract as in procedure 5B2.

Oxalate-permanganate titration

6N4a

Proceed as in 6N2b.

EDTA titration

6N4b

Reagents

Sodium hydroxide (NaOH), 4*N*.

EDTA 0.02*N*.—Dissolve 3.723 g disodium dihydrogen ethylenediamine tetraacetate in water and dilute to 1 liter. Standardize the solution against standard CaCl_2 prepared in the TEA buffer solution.

Ammonium purpurate (murexide) indicator.—Thoroughly mix 0.5 g ammonium purpurate with 100 g powdered potassium sulfate.

Eriochrome Black T (Erio T) indicator. Dissolve 0.5 g Erio T in 100 ml of triethanolamine.

Procedure

Pipet a 5-ml aliquot of extract from procedure 5B3 into a 100-ml beaker. Add 20 ml water, 5 drops 4N NaOH, and 50 mg murexide. Titrate

Atomic absorption

601b

Apparatus

Perkin Elmer Model 290 atomic absorption

usually requires 0.3 to 0.8 ml EDTA to get the proper ice-blue color. Correct for a blank carried through this procedure and use the corrected titration to calculate the magnesium in the sample.

Calculations

Mg (meq/100 g)

$$= \frac{\text{ml EDTA}}{\text{g sample}} \times N \text{ of EDTA} \times \frac{\text{ml extract}}{\text{ml aliquot}} \times 100$$

Report on oven-dry basis.

References

Barrows and Simpson (1962).

Phosphate titration

602b

Reagents

Sodium hydroxide (NaOH), 0.1N, standardized.—Protect from CO₂ of the air with a soda-lime trap.

Sulfuric acid (H₂SO₄), 0.1N.

Ammonium hydroxide (NH₄OH), concentrated.

end point, prepare a color standard by pipetting 5 ml potassium dihydrogen phosphate (2-percent solution) into a 250-ml Erlenmeyer flask, adding 65 ml water, 5 drops brom cresol green, and a macerated filter paper.

Calculations

$$\text{Mg (meq/100 g)} = \frac{\text{ml NaOH blank} - \text{ml NaOH sample}}{\text{g sample}}$$

$$\times N \text{ of NaOH} \times \frac{\text{ml extract}}{\text{ml aliquot}} \times 100$$

Report on oven-dry basis.

References

Peech et al. (1947).

Gravimetric, magnesium pyrophosphate

602c

Reagents

Diammonium hydrogen phosphate ((NH₄)₂HPO₄) 10-percent solution

Atomic absorption

602d

Procedure

Proceed as in 601b except use samples from NH₄OAc extract.

Calculation

Mg (meq/100 g) = Mg from curve (meq/l)

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

Flame photometry

6P1a

Apparatus

Beckman Model DU spectrophotometer with flame attachment.

Reagents

Standard sodium solutions, 0.0 to 2.0 meq per liter.

Concentrated hydrochloric acid (HCl).

Hydrochloric acid (HCl), 6N.

~~Hydrochloric acid (HCl), 6N.~~

References

Fieldes et al. (1951).

Atomic absorption **6P2b**

Proceed as in 6P1b except use standards prepared in NH₄OAc.

Calculations

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

Report on oven-dry basis.

POTASSIUM **6Q**

Saturation extract **6Q1**

Prepare saturation extract as described in 8A1 or 8B1.

Flame photometry **6Q1a**

Apparatus

Beckman Model DU spectrophotometer with flame attachment.

Reagents

Standard potassium chloride (KCl) solutions ranging from 0.0 to 1.0 meq per liter.

Procedure

Proceed as in 6P1a. Determine flame luminosity of potassium at 768 mμ and compare with that of the standard solutions.

Calculations

$$\text{K (meq/l)} = \text{meq/l K from curve} \times \text{dilution}$$

References

Fieldes et al. (1951).

Atomic absorption **6Q1b**

Apparatus

Atomic absorption spectrophotometer with Osram lamp attachment. Hydrogen flame with 2-inch standard burner.

Reagents

Calculations

$$\text{K (meq/100 g)} = \frac{\text{meq/l K from curve}}{\text{g sample}} \times \text{dilution} \times \frac{\text{ml extract}}{10}$$

Report on oven-dry basis.

Atomic absorption **6Q2b**

Proceed as in 6P1b except use standards prepared in NH₄OAc.

Calculations

$$\text{K (meq/100 g)} = \frac{\text{meq/l K from curve}}{\text{g sample}} \times \text{dilution} \times \frac{\text{ml extract}}{10}$$

Report on oven-dry basis.

SULFUR **6R**

NaHCO₃ extract, pH 8.5 **6R1**

Reagents

Sodium bicarbonate (NaHCO₃), 0.5M. Adjust to pH 8.5 with NaOH.

Procedure

Add 40 ml NaHCO₃ solution to 10 g soil in 125-ml Erlenmeyer flask. Shake for 60 minutes and filter through Whatman No. 42 filter paper. Sulfur can be determined on a 2.0-ml aliquot of this filtrate by using methylene blue colorimetry.

Methylene blue colorimetry **6R1a**

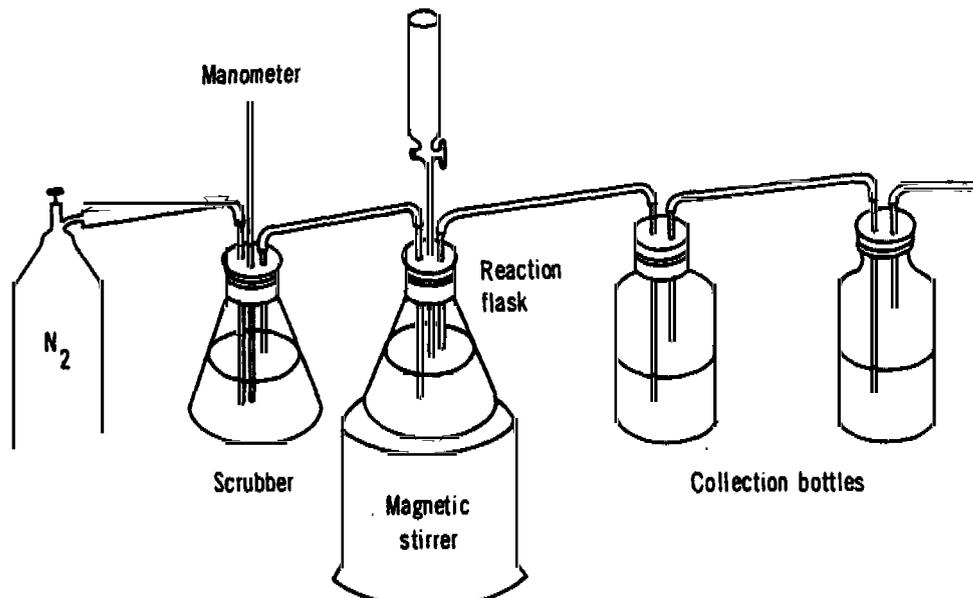
References

Kilmer and Neary (1960).

HCl release (sulfide) **6R2**

Apparatus

Nitrogen tank (water pumped). Scrubber—250-ml Erlenmeyer flask equipped



Procedure

Weigh 2.000 g oven-dry soil, ground to approximately 100 mesh, into a 300-ml Erlenmeyer flask, add 30 ml 60-percent HClO₄, and boil until the soil is white. Continue boiling 20 minutes longer to insure complete extraction. Soils high in organic matter should be pretreated with HNO₃ and HCl to destroy the readily oxidized organic matter.

Molybdovanadophosphoric acid colorimetry 6S1a

Apparatus

Spectrophotometer.

Reagents

After 10 minutes, the color is fully developed on most samples and can be read at 460 m μ . Prepare a standard curve covering the range 0 to 5 ppm phosphorus in 50 ml solution. Plot on semilog paper.

Calculations

$$\text{Total P (pct.)} = \frac{\text{ppm P from curve}}{400} \times \frac{250}{\text{ml aliquot}}$$

Comments

The color developed is molybdovanadophosphoric acid and is very stable, lasting 2 weeks or more.

clay suspension (100 mg clay in a 4-ml volume) in a 50-ml centrifuge tube. Mix with the suspension, remove 1-ml aliquot, and place it on a glass slide. Add approximately ½ ml 10-percent glycerol in ethanol to the tube. Mix and transfer a 1-ml aliquot to a glass slide or use a Mg-clay slide for both Mg and Mg-glycerol solvated slides. Record a diffraction pattern for the Mg-saturated clay film. After solvating the clay film with 10-percent glycerol solution, record a second X-ray pattern.

References

Rex (1967).

Thin film on glass, sodium metaphosphate pretreatment 7A2c

Shake soil overnight in sodium metaphosphate solution (3A2). Centrifuge to separate the clay or siphon off the clay. Pipet about 50 mg clay to a glass slide (47 by 26 mm). Concentrate the clay suspension if necessary. Scan the clay film at room temperature, again after glycerol solvation (7A2a), and finally after heating to 500° C. The clay film is Na⁺ saturated. The sodium metaphosphate peaks do not interfere with peaks of the more common clay minerals in this quick check method.

Thin film on tile, solution pretreatment 7A2d

Apparatus

Ceramic tile (porous precipitate drying plate, sawed into 27- by 46- by 7-mm blocks).

Procedure

Prepare clay suspensions as in 7A2a except dry the suspensions on ceramic tile blocks. Clay suspensions dry in a few seconds on tile, preventing particle-size segregation. Partly immerse the Mg-saturated clay films in a 10-percent glycerol solution. The porous tile rapidly transfers the glycerol to the clay film. Blot off excess glycerol before recording the X-ray pattern.

Thin film on tile, resin pretreatment 7A2e

Prepare clay suspensions as in 7A2b. Dry on ceramic tile blocks as in 7A2d. Solvate with glycerol as in 7A2d.

Thin film on tile, sodium metaphosphate pretreatment 7A2f

Prepare the sample as in 7A2c. Pipet the clay onto ceramic tile blocks as in 7A2d. Follow method 7A2c for the other treatments. Or solvate with glycerol as in 7A2d.

Powder mount, diffractometer recording 7A2g

Distinguishing dioctahedral and trioctahedral minerals requires random orientation of the sample. There is no completely satisfactory method for preparing a random mount, but several techniques are used.

Pack the sample in a box mount against a glass slide. When the box is full, tape the back of

the box. Invert the box and remove the slide to expose the sample to X-rays. For more random packing, sprinkle the dry sample (ground to <100 mesh) on double stick tape fixed on a glass slide or on a thin film of Vaseline on a glass slide. Scan the sample by X-ray and measure the reflections with a geiger, proportional, or other counter.

Quick checks for whole samples, particularly for nonlayered minerals, can be made with a modified powder mount. Form the sample into a thick slurry, apply to a glass slide, and let dry. This is for convenience rather than random orientation.

Powder mount, camera recording 7A2h

Photographic plates are still the best means of identifying minerals. Mount the sample in the center of a circular X-ray camera. Record the X-ray reflections on photographic film placed in a cylindrical film holder inside the camera. All diffraction peaks are recorded simultaneously.

Differential thermal analysis 7A3

Differential thermal analysis is a measurement of the difference in heat absorbed by or evolved from a sample and a thermally inert material as the two are heated at a constant rate. Thermocouples are arranged in wells in a metal block with one junction in a well for the unknown and one junction in another well in an inert material of similar composition. This assembly is placed in a well-insulated furnace. If a reaction occurs, a difference in temperature is registered on a strip-chart recorder or photographically. The magnitude of the difference depends on the nature 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.

The common endothermic reactions studied or recorded are loss of structural water in gibbsite, goethite, and kaolin and loss of carbon dioxide in carbonates. Change of state or rearrangement of crystal lattices can be either exothermic or endothermic. Oxidation reactions such as burning of carbon and oxidation of ferrous iron are exothermic.

Loss of water of constitution can be measured quantitatively by calibrating areas of peaks of known mixtures of standard minerals. This is done commonly to determine the percentage of kaolin and gibbsite in soils. The standard curves are prepared by running the known mixtures under the same conditions as the unknowns. Kaolin has an endotherm at 500° to 600° C and gibbsite at 310° C. Each worker should prepare his own standard curves.

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 hours 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 ex-

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.

touched by the 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

silt. Such material may resemble muscovite but it is cloudy, shows no definite extinction, and has very low birefringence.

Allophane occurs in many soils derived from

Electron microscopy**7B2**

Electron microscopy gives information on particle size and general morphology of clay-size particles. Evidence of clay formation or weathering can also be seen. Positive identification of halloysite often depends on observation of rolled structures under the electron microscope.

Procedure

Place a drop of dilute clay suspension on a 200-mesh copper grid. After drying, insert this grid in the microscope.

TOTAL ANALYSIS**7C****Chemical****7C1**

The procedures follow the standard procedures for rock analysis set forth by Hillebrand and Lundell (1929) and modified by Robinson (1930) and by Shapiro and Brannock (1956).

X-ray emission spectrography**7C2**

of standards of similar composition prepared in a similar manner, (2) fusing both samples and standards in borax or lithium borate to eliminate particle-size effects and to reduce matrix effects, and (3) making matrix corrections by calculating the absorption-enhancement coefficient of the sample for the particular radiation being measured.

References

Vanden Heuvel (1965).

SURFACE AREA**7D****Glycerol retention****7D1****Apparatus**

Weighing cans.

Reagents

Glycerol, 2-percent.

Procedure

Ovendry a clay sample (about 0.2 g) at 110° C

MISCELLANEOUS

SATURATED PASTE, MIXED

8A *References*

Apparatus

Container of 500-ml capacity with lid such as a pint polyethylene refrigerator dish.

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 400-g sample is convenient to handle and provides enough extract for most purposes. Mixing is generally easier if the soil is first airdried and passed through a 2-mm sieve.

Special precautions must be taken for peat and muck soils and soils of very fine texture. Dry peat and muck soils, especially if coarse-textured or woody, require overnight wetting to get a definite 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 at 110° C to determine moisture at saturation.

Richards (1954).

Saturation extract

8A1

Apparatus

Richards or Büchner funnels.

Filter rack or flask.

Filter paper.

Vacuum pump.

Extract containers such as test tubes or 1-oz bottles.

Procedure

Transfer the saturated soil paste to a filter funnel with a filter paper in place and apply vacuum until air begins to pass through the filter. Collect the extract in a bottle or test tube. If carbonate and bicarbonate are to be determined on the extract, add 1 drop of 1,000 ppm sodium hexametaphosphate solution for each 25 ml of extract to prevent precipitation of calcium carbonate on standing.

References

Richards (1954).

Conductivity of saturation extract

8A1a

Apparatus

Conductivity bridge.

Conductivity cell.

Procedure

Determine temperature of the saturation extract obtained by methods 8A1 or 8B1. Draw the extract into the cell and read the meter. Correct for temperature and cell constant using table 1 (table 15, Richards 1954) and report as electrical conductivity, mmhos per centimeter at 25° C. If the instrument fails to balance, dilute the extract 1:9 with distilled water and redetermine. The conductivity of the diluted extract is approximately one-tenth the conductivity of the saturation extract.

References

Richards (1954).

TABLE 1.—Temperature factors (f_t) for correcting resistance and conductivity data on soil extracts to the standard temperature of 25° C.

$$EC_{25} = EC_t \times f_t; EC_{25} = (k/R_t) \times f_t; R_{25} = R_t/f_t$$

° C.	° F.	f_t	° C.	° F.	f_t	° C.	° F.	f_t
3.0	37.4	1.709	22.0	71.6	1.064	29.0	84.2	0.925
4.0	39.2	1.660	22.2	72.0	1.060	29.2	84.6	.921
5.0	41.0	1.613	22.4	72.3	1.055	29.4	84.9	.918
6.0	42.8	1.569	22.6	72.7	1.051	29.6	85.3	.914
7.0	44.6	1.528	22.8	73.0	1.047	29.8	85.6	.911
8.0	46.4	1.488	23.0	73.4	1.043	30.0	86.0	.907
9.0	48.2	1.448	23.2	73.8	1.038	30.2	86.4	.904
10.0	50.0	1.411	23.4	74.1	1.034	30.4	86.7	.901
11.0	51.8	1.375	23.6	74.5	1.029	30.6	87.1	.897
12.0	53.6	1.341	23.8	74.8	1.025	30.8	87.4	.894
13.0	55.4	1.309	24.0	75.2	1.020	31.0	87.8	.890
14.0	57.2	1.277	24.2	75.6	1.016	31.2	88.2	.887
15.0	59.0	1.247	24.4	75.9	1.012	31.4	88.5	.884
16.0	60.8	1.218	24.6	76.3	1.008	31.6	88.9	.880
17.0	62.6	1.189	24.8	76.6	1.004	31.8	89.2	.877
18.0	64.4	1.163	25.0	77.0	1.000	32.0	89.6	.873
18.2	64.8	1.157	25.2	77.4	.996	32.2	90.0	.870
18.4	65.1	1.152	25.4	77.7	.992	32.4	90.3	.867
18.6	65.5	1.147	25.6	78.1	.988	32.6	90.7	.864
18.8	65.8	1.142	25.8	78.5	.983	32.8	91.0	.861
19.0	66.2	1.136	26.0	78.8	.979	33.0	91.4	.858
19.2	66.6	1.131	26.2	79.2	.975	34.0	93.2	.843
19.4	66.9	1.127	26.4	79.5	.971	35.0	95.0	.829
19.6	67.3	1.122	26.6	79.9	.967	36.0	96.8	.815
19.8	67.6	1.117	26.8	80.2	.964	37.0	98.6	.801
20.0	68.0	1.112	27.0	80.6	.960	38.0	100.2	.788
20.2	68.4	1.107	27.2	81.0	.956	39.0	102.2	.775
20.4	68.7	1.102	27.4	81.3	.953	40.0	104.0	.763
20.6	69.1	1.097	27.6	81.7	.950	41.0	105.8	.750
20.8	69.4	1.092	27.8	82.0	.947	42.0	107.6	.739
21.0	69.8	1.087	28.0	82.4	.943	43.0	109.4	.727
21.2	70.2	1.082	28.2	82.8	.940	44.0	111.2	.716
21.4	70.5	1.078	28.4	83.1	.936	45.0	113.0	.705
21.6	70.9	1.073	28.6	83.5	.932	46.0	114.8	.694
21.8	71.2	1.068	28.8	83.8	.929	47.0	116.6	.683

Conductivity of saturation extract
(quick test)

8A1b

Procedure

Rinse the soil cup with water, dry, and fill with soil paste (8A). Jar cup to remove air bubbles, strike off excess paste so the cup is level full, and connect cup to the bridge. Record resistance (ohms) and temperature of soil paste (°F).

Apparatus

Extractor, miniature Richards-type (fig. 10).
Conductivity cell, micropipet.
Filter paper, glass fiber, 3.0 cm.
Vacuum pump.

Calculations

Convert resistance of the soil paste in ohms to percentage of soluble salt by using the tables and

Procedure

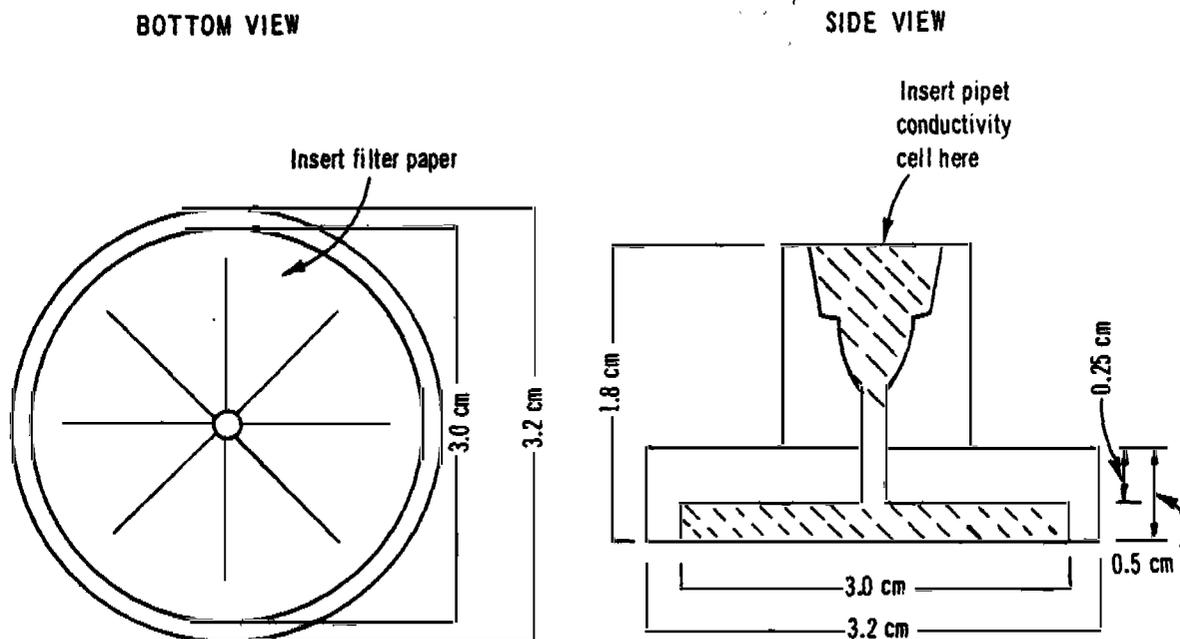


FIGURE 10.—Miniature Richards-type extractor made of polymethyl methacrylate (Lucite).

Procedure

Weigh 250 g air-dry soil into cups made from Whatman No. 52 (15-cm) filter paper and place them on a sand table wetted at 5-cm tension with water. The sand table used consists of two nested plastic dishpans. The outer pan holds distilled water, which is kept at a constant level by a Mariotte bottle. The inner pan, containing medium to fine (35 to 80 mesh) pure quartz sand, rests on rubber stoppers and is suspended in the distilled water. Its perforated bottom is covered with a fine cloth-mesh screen that permits water to move upward by capillarity through the sand to the table surface. The sand on the table surface is then smoothed and covered with an absorbent paper towel. Lightweight porous firebricks can be used in place of the sand table.

Keep the samples on the sand table 16 to 18 hours, remove them, and weigh. Water adsorption drops rapidly after an initial wetting of 2 hours and the rate becomes very slow after 6 to 9 hours. Moisture moves toward the top and center of the sample, which is wetted last, insuring retention of soluble salts in the soil. Calculate moisture at saturation from the wet- and dry-soil weights, correcting for the wet and dry filter paper weights. Add air-dry moisture percentage to moisture at saturation and report on oven-dry basis. After the wet weighing, transfer the sample to a pint polyethylene refrigerator dish, mix briefly with a spatula, and determine the pH. Keep a lid on the dish whenever possible to reduce evaporation.

References

Longenecker and Lyerly (1964).

Saturation extract

8B1

Proceed as in 8A1, using the saturated paste obtained by method 8B.

Conductivity of saturation extract

8B1a

Proceed as in method 8A1a except use saturation extract obtained by method 8B1.

REACTION (pH)

8C

Soil suspensions

8C1

Water dilution

8C1a

Procedure

For 1:1 dilution add an equal weight of water to 20 or 30 g soil in a 50-ml beaker or paper cup. Stir at regular intervals for about an hour. Measure pH of the soil suspension with a glass electrode, stirring well just before immersing the electrodes in the suspension. For other dilutions vary the amount of soil, keeping the volume of water constant.

Saturated paste

8C1b

Procedure

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

KCl

8C1c

Procedure

Proceed as in method 8C1a except use *N* KCl instead of water.

NaF

8C1d

Reagents

Sodium fluoride (NaF), 1N (saturated). Add 1,000 ml water to 50 g NaF in a 1-liter 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 0.01N 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 per liter, try another source of NaF.

Apparatus

pH meter.
Stopwatch or watch with minute sweep hand.

Procedure

RATIOS AND ESTIMATES

8D

To total clay

8D1

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

To noncarbonate clay

8D2

Divide cation-exchange capacity, extractable iron, or 15-bar water retention by the noncarbonate clay percentage determined by subtracting the carbonate clay from total clay.

Calcium to magnesium (extractable)

8D3

Divide extractable calcium by extractable magnesium.

Estimated clay percentage

8D4

For most soils clay percentage can be approxi-

TABLE 2.—Bureau of Soils data for reducing soil paste resistance readings to values at 60° F. (Whitney and Means, 1897)¹

° F.	Ohms								
	1,000	2,000	3,000	4,000	5,000	6,000	7,000	8,000	9,000
40.....	735	1,470	2,205	2,940	3,675	4,410	5,145	5,880	6,615
42.....	763	1,526	2,289	3,052	3,815	4,578	5,341	6,104	6,867
44.....	788	1,576	2,364	3,152	3,940	4,728	5,516	6,304	7,092
46.....	814	1,628	2,442	3,256	4,070	4,884	5,698	6,512	7,326
48.....	843	1,686	2,529	3,372	4,215	5,058	5,901	6,744	7,587
50.....	867	1,734	2,601	3,468	4,335	5,202	6,069	6,936	7,803
52.....	893	1,786	2,679	3,572	4,465	5,358	6,251	7,114	8,037
54.....	917	1,834	2,751	3,668	4,585	5,502	6,419	7,336	8,253
56.....	947	1,894	2,841	3,780	4,735	5,682	6,629	7,576	8,523
58.....	974	1,948	2,922	3,896	4,870	5,844	6,818	7,792	8,766
60.....	1,000	2,000	3,000	4,000	5,000	6,000	7,000	8,000	9,000
62.....	1,027	2,054	3,081	4,108	5,135	6,162	7,189	8,216	9,243
64.....	1,054	2,108	3,162	4,216	5,270	6,324	7,378	8,432	9,486
66.....	1,081	2,162	3,243	4,324	5,405	6,486	7,567	8,648	9,729
68.....	1,110	2,220	3,330	4,440	5,550	6,660	7,770	8,880	9,990
70.....	1,140	2,280	3,420	4,560	5,700	6,840	7,980	9,120	10,260
72.....	1,170	2,340	3,510	4,680	5,850	7,020	8,190	9,360	10,530
74.....	1,201	2,402	3,603	4,804	6,005	7,206	8,407	9,608	10,809
76.....	1,230	2,460	3,690	4,920	6,150	7,390	8,610	9,840	11,070
78.....	1,261	2,522	3,783	5,044	6,305	7,566	8,827	10,088	11,349
80.....	1,294	2,598	3,882	5,176	6,470	7,764	9,058	10,352	11,646
82.....	1,327	2,654	3,981	5,308	6,635	7,962	9,289	10,616	11,943
84.....	1,359	2,718	4,077	5,436	6,795	8,154	9,513	10,872	12,231
86.....	1,393	2,786	4,179	5,572	6,965	8,358	9,751	11,144	12,537
88.....	1,427	2,854	4,281	5,708	7,135	8,562	9,989	11,416	12,843
90.....	1,460	2,920	4,380	5,840	7,300	8,760	10,220	11,680	13,140
92.....	1,495	2,990	4,485	5,980	7,475	8,970	10,465	11,960	13,455
94.....	1,532	3,064	4,596	6,128	7,660	9,192	10,724	12,256	13,788
96.....	1,570	3,140	4,710	6,280	7,850	9,420	10,990	12,560	14,130
98.....	1,611	3,222	4,833	6,444	8,055	9,666	11,277	12,888	14,499

¹ Example: Suppose the observed resistance is 2,568 ohms at 50° F. In the table at that temperature, we find that 2,000 ohms is equal to 1,734 ohms at 60° F., 5,000 ohms is equal to 4,335 ohms at 60° F., hence 500 ohms would be equal to 434 ohms. Similarly, 60 ohms would be one-hundredth of 6,000 ohms in the table and therefore equal to approximately 52 ohms at 60° F., while 8 ohms would be equal to about 7 ohms. These separate values are added together thus,

2,000	1,734
500	434
60	52
8	7

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