

Methods for Soil Characterization

Unless otherwise specified, all chemicals referred to in this chapter, as well as in chapters 7 and 8, are "reagent" grade and conform to standards established by the American Chemical Society.

The following concentrated reagents are used.

Reagents:	Percent	Normality	Specific gravity
Acetic acid _____	99.5	18	
Hydrochloric acid-----	35-38	12	1.19
Nitric acid _____	70	16	1.42
Sulfuric acid _____	95-96	36	1.84
Ammonium hydroxide _____	28 (NH ₃)	15	.90

Dilutions are indicated by (1+2), (1+10), and other proportions. The first figure indicates the volume of concentrated reagent and the second the volume of water.

Several methods involve centrifugation processes that are specified in terms of time and relative centrifugal force (RCF), which is the ratio of the acceleration in the centrifuge to the acceleration of gravity, i. e., $RCF = 0.0000112 \times r \times (\text{r. p. m.})^2$ where r, p. m. is centrifuge speed in revolutions per minute and r is the radius in centimeters from the axis of the centrifuge to the bottom of the centrifuge vessel when in the rotating position.

Sampling, Soil Extracts, and Salinity Appraisal

(1) Soil Sample Collecting, Handling, and Subsampling

A round-nose trenching spade is a convenient tool for sampling surface soil. A soil tube is useful for small subsurface samples, whereas a barrel-type auger can be used when larger subsurface samples are required. Canvas bags are generally used as containers for soil samples, especially for samples of 100 to 200 pounds. For small samples, metal boxes or cardboard cartons can be used. Samples for salinity measurements require special handling, because at field-moisture content the salt in the soil is relatively mobile and moves with the soil water. It has been found that kraft paper nail bags are satisfactory for handling samples of saline soil, providing the bags are first waterproofed by soaking in a 5 or 10 percent solution of paraffin in gasoline or other wax solvent.

Soil should be air-dried before shipping or storing for any extended length of time. Air-dry soils that con-

tain deliquescent salts may accumulate enough moisture during a short shipping or storage period to decompose a canvas bag. A container impervious to water vapor should be used for such soils. Wax-treated bags, as mentioned above, or various types of water-proofed bags used for merchandising foodstuff or other hygroscopic material can be used. Samples in paper bags will withstand usual transportation handling if they are tightly packed in wooden boxes. To guard against accidental confusion of samples, it is desirable to place an identification tag inside the bag, in addition to using an external marking or tag.

The following recommendations will aid in determining the size of sample required:

Measurements to be made:	Soil required in grams
1. Electrical conductivity of the saturation extract, saturation percentage, and pH of soil paste _____	250
2. Soluble ion analysis (semimicro methods) for—	
Low salinity _____	500
High salinity _____	250
3. Exchangeable-cation analysis _____	15
4. Hydraulic conductivity (disturbed) _____	400
5. Gypsum and alkaline-earth carbonates _____	50

The total amount of soil to be obtained for the sample can be determined by adding up the amounts indicated for the individual tests to be made. If measurement 2 is to be made, then no extra soil will be required for measurement 1. Samples twice as large as those indicated above are desirable, if handling facilities permit.

Care must be taken to obtain representative subsamples of a granular material such as soil. Bulk samples at the Laboratory are air-dried before or after passing through a screen with 6-mm. square openings, are mixed, and are stored in galvanized iron containers. An attempt is made to maintain a level surface of soil in a container so that a minimum of segregation of particles or aggregates occurs from rolling. A subsample of the main sample is taken by means of several partial loadings of a hand scoop from different locations on the surface of the soil. The subsample is then screened to the desired size. For exchangeable-cation analysis and other determinations requiring samples of about 5 gm. or less, the soil is ground to pass a 0.5-mm. sieve. For a number of tests relating to moisture retention and moisture transmission, the soil is passed through a 2-mm. round-hole sieve with the aid of a rubber stopper. One purpose of such sieving is to remove rocks larger than 2 mm.; another is to reduce all aggregates to less than 2 mm. In the removal of rocks between 2 mm. and 6 mm., they may be

returned to the screened sample if desired. The entire subsample is then placed on a mixing cloth and pulled in such a way as to produce mixing. Some pulling operations will produce segregation instead of mixing, and special care must be exercised to obtain a well-mixed sample. The soil sample is then flattened until the pile is 2 to 4 cm. deep.

For moisture retentivity, hydraulic conductivity, and modulus of rupture tests, 2 to 6 subsamples, each having a fairly definite volume, are required. Use paper cups to hold the individual subsamples. Mark with a pencil line around the inside of the cup the height to which the cup is to be filled to give the correct amount of subsample. Then, using a thin teaspoon or a small scoop, lift small amounts of soil from the pile, placing each in successive cups and progressing around the pile until the cups are filled to the desired level. It is difficult with some soils, especially if they have been passed through a 2-mm. round-hole sieve, to take samples from the pile without allowing the larger particles to roll off the spoon or scoop. This rollback should be avoided because it makes the extracted subsample nonrepresentative. The rollback problem is practically absent from some soils, especially if all the sample has been passed through an 0.5-mm. sieve.

Three data forms, or work sheets, used at the Laboratory are shown herewith. The field data sheet should be at hand during sampling as an aid in recording pertinent information. The other two forms serve as work sheets for recording and calculating laboratory determinations.

(2) Saturated Soil Paste

Apparatus

Container of 250-ml. capacity or greater, such as a cup or moisture box.

Procedure

Prepare the saturated soil paste by adding distilled water to a sample of soil while stirring with a spatula. The soil-water mixture is consolidated from time to time during the stirring process by tapping the container on the workbench. At saturation the soil paste glistens as it reflects light, flows slightly when the container is tipped, and the paste slides freely and cleanly off the spatula for all soils but those with a high clay content. After mixing, the sample should be allowed to stand for an hour or more, and then the criteria for saturation should be rechecked. Free water should not collect on the soil surface nor should the paste stiffen markedly or lose its glistening appearance on standing. If the paste does stiffen or lose its glisten, remix with more water.

Because soils puddle most readily when worked at moisture contents near field capacity, sufficient water should be added immediately to bring the sample nearly to saturation. If the paste is too wet, additional dry soil may be added.

The amount of soil required depends on the measurements to be made, i. e., on the volume of extract desired. A 250-gm. sample is convenient to handle and provides sufficient extract for most purposes. Initially, the sample can be air-dry or at the field-moisture content, but the mixing process is generally easier if the soil is first air-dried and passed through a 2-mm. sieve.

If saturation pastes are to be made from a group of samples of uniform texture, considerable time can be saved by carefully determining the saturation percentage of a representative sample in the usual way. Subsequent samples can be brought to saturation by adding appropriate volumes of water to known weights of soil.

Special precautions must be taken with peat and muck soils and with soils of very fine and very coarse texture.

PEAT AND MUCK SOILS.—Dry peat and muck soils, especially if coarse or woody in texture, require an overnight wetting period to obtain a definite endpoint for the saturated paste. After the first wetting, pastes of these soils usually stiffen and lose the glisten on standing. Adding water and remixing then gives a mixture that usually retains the characteristics of a saturated paste.

FINE-TEXTURED SOILS.—To minimize puddling and thus obtain a more definite endpoint with fine-textured soils, the water should be added to the soils with a minimum of stirring, especially in the earlier stages of wetting.

COARSE-TEXTURED SOILS.—The saturated paste for coarse-textured soils can be prepared in the same manner as for fine-textured soils; however, a different moisture content is recommended for the salinity appraisal of such soils (Method 3b).

Method 27 gives procedures for determining the moisture content of saturated paste, i. e., the saturation percentage.

(3) Soil-Water Extracts

(3a) Saturation Extract

Apparatus

Richards or Buechner 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, Method 2, to the filter funnel with a filter paper in place and apply vacuum. Collect the extract in a bottle or test tube. Pyrex should not be used if boron is to be determined. If the initial filtrate is turbid, it can be refiltered through the soil or discarded. Vacuum extraction should be terminated when air begins to pass through the filter. If carbonate and bicarbonate determinations are to be made on the extract, a solution containing 1,000 p. p. m. of sodium hexametaphosphate should be added at the rate of one drop per 25 ml. of extract prior

Soil Acc. No. ____

Temporary No. ____

**UNITED STATES SALINITY LABORATORY
FIELD DATA FOR SOIL SAMPLES**

Sampled by _____ Mail address _____ Date _____

State _____ County _____ Nearest settlement _____

Site location $\frac{1}{4}$, _____ $\frac{1}{4}$, Sec. - ; T _____; R _____

Station or farm _____ District or valley _____

Directions for finding site: (Use reverse side for a sketch of roads showing nearest settlement and distance from local landmarks.)

References (Soil Survey Bul., other publications, or correspondence):

Profile description (color, texture, structure, horizons, hardpan, origin, parent material, water table, drainage, and soil series if known):

Topography _____ Surface slope _____ Percent topsoil erosion _____

Microrelief at the sampling site, furrow, ridge, etc. _____

Disturbance from land preparation, leveling, filling, etc. _____

Sample: Depth No. s a c k s Approx. total wt. (lb.) _____

Composite sample: Depth _____ No. h o l e s Sampling method and pattern _____

_____ Approx. total wt. (lb.) _____

Undisturbed structure sample: Depth _____ No. of replicates _____

Yrs. of cultivation _____ Yrs. of irrigation Source of water _____

Crop data (rotation, yield history, detailed description of plant condition at time of sampling):

Management practices:

(It is expected that not all the above blanks can be filled for every sample but the usefulness of laboratory determinations depends on the completeness and accuracy of the field data.)

Soil sample No. _____ . Description:		
Moisture in air-dry Soil	Saturation Percentage	
Can No. _____	From Water Added	By Drying
Air-dry Oven-dry	Can No. _____	Can No. _____
Gross		Wet Oven-dry
Tare _____	Air-dry soil, gm. _____	Gross _____
Net _____	Oven-dry soil, gm. _____	Tare _____
$\frac{OD}{AD}$ <input style="width: 50px; height: 20px;" type="text"/>	H ₂ O, ml. (_____ + _____)	Net _____
P_w (Oven-dry basis) <input style="width: 50px; height: 20px;" type="text"/>	P_w at sat. P_w at sat. (Oven-dry basis) <input style="width: 50px; height: 20px;" type="text"/>	basis) <input style="width: 50px; height: 20px;" type="text"/>
pH of Saturated Soil Paste	Electrical Conductivity	Alkaline-earth Carbonates
<input style="width: 50px; height: 20px;" type="text"/>	Saturation Extract	(Lime)
pH of Saturation Extract	T°C. _____	(Scale: low, medium, high)
<input style="width: 50px; height: 20px;" type="text"/>	k _____	Lime <input style="width: 50px; height: 20px;" type="text"/>
pH of Suspension	R _____	Boron _____ ml.
Soil _____	Millimhos/cm.	_____ %T. p.p.m. B
Water _____	at 25° C. <input style="width: 50px; height: 20px;" type="text"/>	B
		p.p.m. <input style="width: 50px; height: 20px;" type="text"/>
Calcium plus Magnesium (Versenate titration)	Sodium	Potassium
	Standard _____ meq./l.	Standard _____ meq./l.
	r = _____ * _____ - meq./l.	r = _____ * _____ meq./l.
Ca+Mg, sat. ext. meq./l. <input style="width: 50px; height: 20px;" type="text"/>	Na, sat. ext. meq./l. <input style="width: 50px; height: 20px;" type="text"/>	K, sat. ext. meq./l. <input style="width: 50px; height: 20px;" type="text"/>
Ca+Mg, dry soil meq./100 gm. <input style="width: 50px; height: 20px;" type="text"/>	Na, dry soil meq./100 gm. <input style="width: 50px; height: 20px;" type="text"/>	K, dry soil meq./100 gm. <input style="width: 50px; height: 20px;" type="text"/>

Soil Sample No. _____				
1	Centrifuge tube number			
2	Sample, air-dry weight	gm.		
3	Sample, oven-dry weight	gm.		
<u>CATION-EXCHANGE-CAPACITY</u>				
(Saturated with NaAc: Na by flame photometer)				
4	Extracting solution diluted to	ml.		
5	Dilution: Solution 4	dilution ratio		
6	Flame photometer standard	Na meq./l.		
7	Flame photometer reading			
8	Sodium, from graph	meq./l.		
9	Cation-exchange-capacity (OD basis)	meq./100 g.		
<u>EXCHANGEABLE SODIUM</u>				
10	Extracting solution (NH ₄ Ac) diluted to	ml.		
11	Dilution: Solution 10	dilution ratio		
12	Flame photometer standard	Na meq./l.		
13	Flame photometer reading			
14	Sodium, from graph	meq./l.		
15	Total sodium (OD basis)	meq./100 gm.		
16	Sodium in sat. extract (OD basis)	meq./100 gm.		
17	Exchangeable sodium (OD basis)	meq./100 gm.		
18	Exchangeable-sodium-percentage			
<u>EXCHANGEABLE POTASSIUM</u>				
19	Extracting solution (NH ₄ Ac) diluted to	ml.		
20	Dilution: Solution 19	dilution ratio		
21	Flame photometer standard	K meq./l.		
22	Flame photometer reading			
23	Potassium, from graph	meq. l.		
24	Total potassium (OD basis)	meq./100 gm.		
25	Potassium in sat. extract (OD basis)	meq./100 gm.		
26	Exchangeable potassium (OD basis)	meq./100 gm.		
27	Exchangeable-potassium-percentage			

to **stopping and storing**. This prevents the precipitation of calcium carbonate on standing.

For appraising soil salinity for most purposes, the extraction can be made a few minutes after preparing the saturated paste. If the soil contains gypsum, the conductivity of the saturation extract can increase as much as 1 or 2 mmhos/cm. upon standing. Therefore, if gypsum is present, allow the saturated paste to stand several hours before extracting the solution.

If the solution is to be analyzed for its chemical constituents, the saturated paste should stand 4 to 16 hours before extraction.

References

Richards (1949a), Reitemeier and Fireman (1944).

(3b) Twice-Saturation Extract for Coarse-Textured Soils (Tentative)

The following procedure gives a moisture content that is approximately 8 times the 15-atmosphere percentage instead of 4 times, which is a usual factor for the saturation percentage of finer textured soils. The conductivity of the "twice-saturation" extract, therefore, is doubled before using the standard saturation-extract scale for salinity evaluation.

Apparatus

Soil container of 10 to 12 cm. diam. (i. e., 1-lb. coffee can) with a loosely fitting basket formed from galvanized screen with openings approximately 6 mm. square.

Pipet, 2-ml. capacity. Other items are the same as for Method 3a.

Procedure

Place the wire basket in the can, fill the basket with soil to a depth of 2 or 3 cm. Level the soil and by use of a pipet add 2 ml. of water dropwise to noncontiguous spots on the soil surface, cover, and allow to stand for 15 min. Gently sift the dry soil through the wire basket and weigh the moist pellets of soil retained thereon. Calculate the moisture content of the pellets as follows:

$$P_w = (2 \times 100) / (\text{wet weight in grams} - 2)$$

Weigh 250 gm. of air-dry soil and add sufficient water to make the moisture content up to 4 times the value found in the pellets. Use a vacuum filter to obtain the soil extract. For salinity appraisal of coarse-textured soil from which this extract was obtained, determine the electrical conductivity of the extract at 25° C. Multiply this conductivity value by 2 before using the standard saturation-extract salinity scale for interpretation (chs. 2 and 4).

(3c) Soil-Water Extracts at 1:1 and 1:5

Apparatus

Filter funnels, fluted filter paper, and bottles for soil suspensions and filtrates.

Procedure

Place a soil sample of convenient size in a bottle, add the required amount of distilled water, stopper, and agitate in a mechanical shaker for 15 min. Allow the contents to stand at least an hour, agitate again for 5 min., and filter. If shaken by hand, invert and shake bottle vigorously for 30 sec. at least 4 times at 30-min. intervals before filtering.

At a 1:1 soil-water ratio, it may be desirable to correct for hygroscopic moisture. Unless high precision is required, this is done by grouping the air-dried and screened soils roughly according to texture, and determining the percent moisture in 2 or 3 samples from each textural group. It is then possible to weigh out soil samples from the various groups and add sufficient water to bring the samples to approximately 100 percent moisture by weight. For example, an air-dry soil containing 3 percent moisture on an oven-dry basis can be brought to a 1:1 soil-water ratio by adding 97 ml. water to 103 gm. of air-dried soil.

At a soil-water ratio of 1:5 or greater, no allowance is ordinarily made for moisture in the air-dried sample.

(3d) Soil Extract in the Field-Moisture Range

A displacement method such as used by White and Ross (1937) does not require complicated apparatus; however, the pressure-membrane method described here can be used for a wider range of soil textures and a wider range of moisture contents.

Apparatus

Pressure-membrane cell with a cylinder 5 or 10 cm. high, tank of commercial water-pumped nitrogen, cans with watertight lids, plain transparent cellophane No. 600.

Procedure

Prior to use, the sheets of No. 600 cellophane are soaked in distilled water with daily changes of water in order to reduce the electrolyte content of the membrane. Electrical conductivity measurements on the water will indicate when the bulk of these impurities has been removed. Since washed and dried membranes may be somewhat brittle, they are stored wet until ready for use. They should be partially dried before mounting in the pressure-membrane apparatus.

The soil should be brought from the field at the moisture condition desired for the extraction and immediately packed in the pressure-membrane apparatus. If the soil has been air-dried, it may be passed through a 6-mm. screen and wetted to the desired water content with a fine spray of distilled water while tumbling in a mixing can or on a waterproofed mixing cloth. This wetted soil is stored in an airtight container, preferably in a constant-temperature room for 2 weeks and is mixed occasionally during this time. The pressure-membrane apparatus is then assembled, using No. 600

plain transparent cellophane for the membrane. The soil is firmly packed by hand on the membrane in the extraction chamber to a depth of 2 or 4 in., depending upon the height of cylinder available. The chamber is then closed and the extraction process started at 225 lb. per sq. in. (15 atm.) of nitrogen gas.

The extract should be collected in fractions of approximately equal volume. The first fraction is usually discarded to avoid contamination from the membrane. Electrical conductivity measurements can be made on subsequent fractions to determine the degree of uniformity of the extract. The extraction process may require 1 to 4 days.

References

Reitemeier (1946), Reitemeier and Richards (1944), Richards (1947), and White and Ross (1937).

(4) Electrical Conductivity of Solutions

(Pa) Standard Wheatstone Bridge

Remarks

Electrical conductivity is commonly used for indicating the total concentration of the ionized constituents of solutions. It is closely related to the sum of the cations (or anions) as determined chemically and usually correlates closely with the total dissolved solids. It is a rapid and reasonably precise determination that does not alter or consume any of the sample.

Apparatus

Wheatstone bridge, alternating current, suitable for conductivity measurements. This may be a 1,000-cycle a. c. bridge with telephone receivers, a 60-cycle a. c. bridge with an a. c. galvanometer, or one of the newer bridges employing a cathode ray tube as the null indicator.

Conductivity cell, either pipet or immersion type, with platinized platinum electrodes. The cell constant should be approximately 1.0 reciprocal centimeter. **New** cells should be cleaned with chromic-sulfuric acid cleaning solution, and the electrodes platinized before use. Subsequently, they should be cleaned and replatinized whenever the readings become erratic or when an inspection shows that any of the platinum black has flaked off. The platinizing solution contains platinum chloride, 1 gm., lead acetate, 0.012 gm., in 100 ml. water. To platinize, immerse the electrodes in the above solution and pass a current from a 1.5-volt dry battery through the cell. The current should be such that only a small quantity of gas is evolved, and the direction of current flow should be reversed occasionally.

A thermostat is required for precise measurements, but for many purposes it is satisfactory to measure the temperature of the solution and make appropriate temperature corrections.

Reagents

Potassium chloride solution, 0.01 N. Dissolve 0.7456 gm. of dry potassium chloride in water and make to 1 liter at 25° C. This is the standard reference solution and at 25° C. has an electrical conductivity of 1411.8×10^{-6} (0.0014118) mhos/cm.

Procedure

Fill the conductivity cell with the reagent, having known conductivity $\bar{E}C_{25}$. Most cells carry a mark indicating the level to which they should be filled or immersed. Follow the manufacturers' instructions in balancing the bridge. Read the cell resistance, R_{25} at 25° C. and calculate the cell constant (k), from the relation,

$$k = \bar{E}C_{25} \times R_{25}$$

The cell constant will change if the platinization fails, but it is determined mainly by the geometry of the cell, and so is substantially independent of temperature.

Rinse the cell with the solution to be measured. The adequacy of rinsing is indicated by the absence of resistance change with successive rinsings. If only a small amount of the sample is available, the cell may be rinsed with acetone and ventilated until it is dry. Record the resistance of the cell (R_t) and the temperature of the solution (t) at which the bridge is balanced. Keep the cell filled with distilled water when not in use.

Calculations

The electrical conductivity (EC_t) of the solution at the temperature of measurement (t) is calculated from the relation

$$EC_t = k/R_t$$

where

$$k = \bar{E}C_{25} \times R_{25}$$

For soil extracts and solutions, a temperature conversion factor (f_t), obtained from table 15, can be used for converting conductivity values to 25° C. Thus,

$$EC_{25} = EC_t \times f_t = k f_t / R_t$$

References

Campbell and others (1948), National Research Council International Critical Tables (1929).

(4b) Direct Indicating Bridge

Apparatus

Conductivity sets are available that have a bridge scale and cell design features suggested by the Laboratory especially for use with saturation extracts (fig. 26). This set is convenient to use and has sufficient accuracy for diagnostic purposes. The conductivity cell supplied with this bridge has a constant of 0.5 cm.^{-1} and a capacity of 2 to 3 ml. of solution. With this cell the

TABLE 15.—*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	2.277	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

bridge scale reads directly from 0.15 to 15 mmhos/cm. The bridge is operated by alternating current and makes use of a cathode ray tube null indicator. When the temperature of the solution is set on the temperature-compensating dial, the main dial, at balance, indicates electrical conductivity at 25° C.

The accuracy of calibration of the bridge scale should be checked with a saturated solution of calcium sulfate dihydrate. With the temperature-compensation dial correctly set, the bridge should read 2.2 mmhos/cm. with this solution.

Procedure

Obtain the saturation extract in accordance with Method 3a. Read the temperature of the extract. Rinse and fill the conductivity cell. Set the temperature com-

pensation dial. Close the contact switch on the cell briefly while balancing the bridge with the main dial. Read and record the electrical conductivity in millimhos per centimeter at 25° C.

If the bridge will not balance, the conductivity of the extract may be below 0.15 or above 15 mmhos/cm. If above, estimate conductivity by adding 9 parts of distilled water to 1 part of extract, by volume, and balancing the bridge with the diluted extract in the cell. The conductivity of the undiluted extract will be approximately 10 times the conductivity reading obtained on the diluted extract.

Alternatively, for concentrated extracts, a cell with a constant higher than 0.5 may be used. If, for example, the value of the cell constant is 5.0, then the scale reading of the bridge must be multiplied by 10.

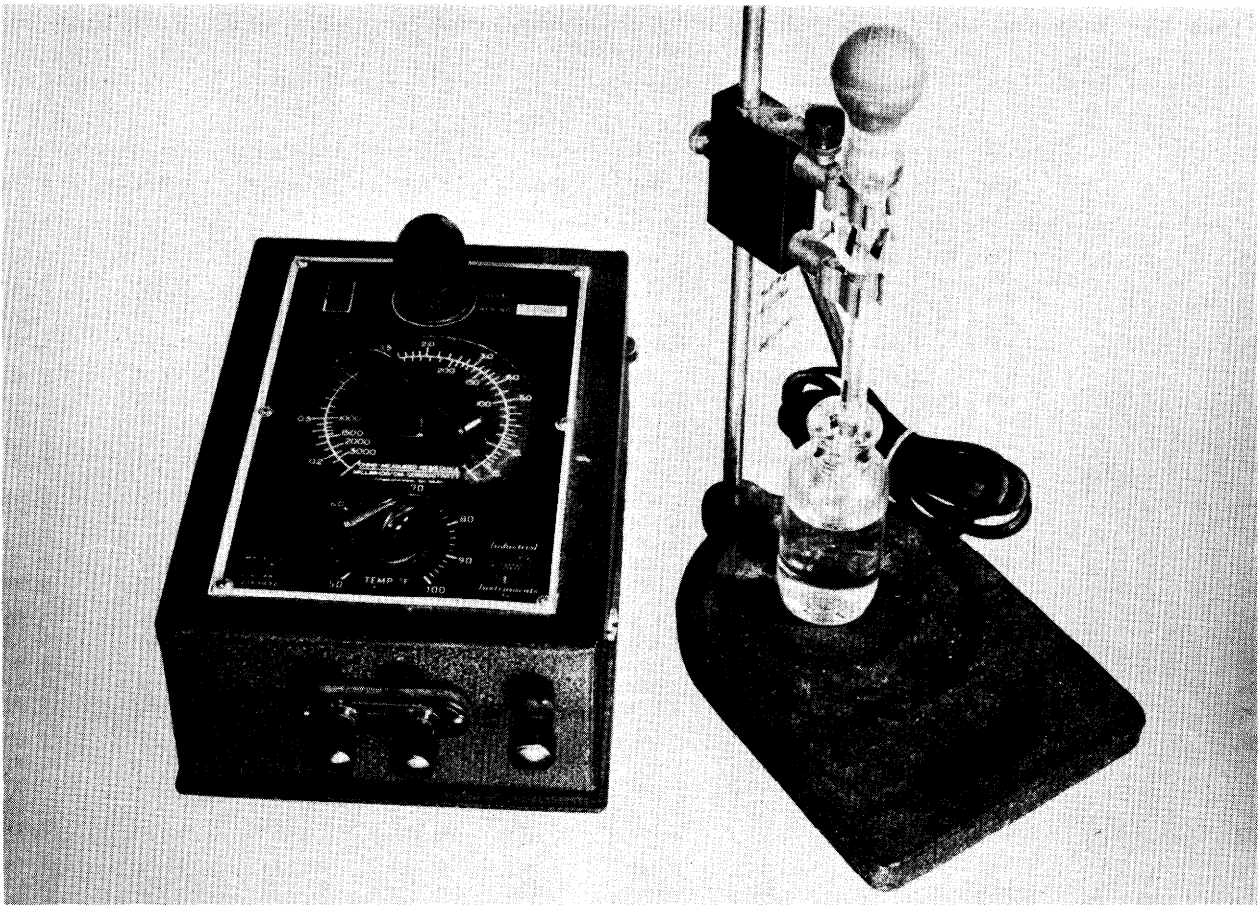


FIGURE 26.—Bridge and cell for measuring the conductivity of saturation extracts and irrigation waters.

(5) Resistance of Soil Paste and Percent Salt in Soil

Apparatus

Bureau of Soils electrode cup, alternating current Wheatstone bridge, and thermometer.

Procedure

Fill the electrode cup with saturated soil paste prepared in accordance with Method 2. Tap the soil cup on the workbench to remove air bubbles and strike off the soil paste level with the upper surface of the cup. Measure the resistance and the temperature of the soil paste in the cup. Use table 16 to convert the resistance reading to the temperature of 60° F. Then, by means of table 17, convert the paste resistance at 60° to approximate percent salt. Inasmuch as the saturation percentage varies with soil texture, it is necessary to estimate the textural class of the sample and to select the appropriate column in the table for making the conversion from resistance to percent salt.

References

Davis and Bryan (1910), Soil Survey Manual (1951).

(6) Freezing-Point Depression

(6a) *Freezing-Point Depression of Solutions*

Apparatus

Wheatstone bridge with approximately the following characteristics: 1,000 ohms equal arm ratio, 10,000-ohm decade balancing resistance adjustable to 1 ohm; galvanometer: type E, Leeds and Northrup DM-2430-c, or equivalent. Use a 2-volt lead cell for the bridge voltage supply. Thermistor: type 14B, Western Electric. Freezing bath: with either refrigerating coil or salt-ice mixture. Freezing tube: test tube 1.5 cm. inside diameter X 15 cm. long with rubber stopper. Air-jacket: test tube 2.9 cm. outside diameter X 20 cm. long. Use cork bushings cut by means of a grinding

TABLE 16.—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	a, 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, a67
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.....	a43	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.....	a93	1,786	2,679	3,572	4,465	5,358	6,251	7,114	a, 037
54.....	917	1, a34	2,751	3,668	4,585	5,502	6,419	7,336	a, 253
56.....	947	1, a94	2, a41	3,780	4,735	5,682	6,629	7,576	a, 523
58.....	974	1,948	2,922	3,896	4, a70	5, a44	6, ala	7,792	a, 766
60.....	1,000	2,000	3,000	4,000	5,000	6,000	7,000	a, 000	9,000
62.....	1,027	2,054	3,081	4,108	5,135	6,162	7,189	a, 216	9,243
64.....	1,054	2,108	3,162	4,216	5,270	6,324	7,378	a, 432	9,486
66.....	1,081	2,162	3,243	4,324	5,405	6,486	7,567	a, 648	9,729
68.....	1,110	2,220	3,330	4,440	5,550	6,660	7,770	a, 880	9,990
70.....	1,140	2,280	3,420	4,560	5,700	6, a40	7,980	9,120	10,260
72.....	1,170	2,340	3,510	4,680	5,850	7,020	a, 190	9,360	10,530
74.....	1,201	2,402	3,603	4,804	6,005	7,206	a, 407	9,608	lo, 809
76.....	1,230	2,460	3,690	4,920	6,150	7,380	a, 610	9, a40	11,070
78.....	1,261	2,522	3,783	5,044	6,305	7,566	a, a27	lo, 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	a, 154	9,513	lo, a72	12,231
86.....	1,393	2,786	4,179	5,572	6,965	a, 358	9,751	11,144	12,537
aa.....	1,427	2, a54	4,281	5,708	7,135	a, 562	9,989	11,416	12, a43
90.....	1,460	2,920	4,380	5, a40	7,300	a, 760	10,220	11,680	13,140
92.....	1,495	2,990	4,485	5,980	7,475	a, 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, a33	6,444	a, 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 a ohms would be equal to about 7 ohms. These separate values are added together thus,

2,000	1,734
500	434
60	52
a	7
<hr/>	

2,568 ohms at 50° = 2,227 ohms at 60°

machine to center and suspend the freezing tubes in the air-jackets. Mount the thermistor on a glass tube with plastic spacers so as to hold the thermosensitive bead at the center of a 5-ml. sample of the solution to be frozen. Plot a resistance-temperature calibration curve for the thermistor over the range from 1 to -5° C., using a standard thermometer or other source of reference temperature.

Procedure

Place 5-ml. samples of solutions in the freezing tubes and mount the tubes in the air-jacket in the freezing bath. An undercooling of approximately 2° C. has been found convenient for soil extracts and plant saps.

Place the thermistor in one of the samples when the sample has attained the bath temperature as indicated by the bridge resistance reading. Induce freezing by touching the solution with a metal probe cooled with solid carbon dioxide. Follow the course of the freezing by keeping the bridge approximately balanced until the minimum resistance (maximum temperature) is attained. With an undercooling of 2° C., a time of about 2 min. is required to attain the maximum observed freezing temperature. The minimum resistance value is recorded as the freezing resistance. The thermistor can then be transferred rapidly to the next sample so that ice crystals carried over in the process may initiate freezing. Include a tube of distilled

TABLE 17.—Bureau of Soils data for relating the resistance of soil paste at 60° F. to percentage of "mixed neutral salts" in soil (Davis and Bryan, 1910)

Resistance at 60° F. (ohms)	Salts in—			
	Sand	Loam	Clay loam	Clay
	Percent	Percent	Percent	Percent
	3.00	3.00
if.....	2.40	2.64	3.00
20.....	2.20	2.42	2.80	3.00
25.....	1.50	1.70	1.94	2.20
30.....	1.24	1.34	1.46	1.58
35.....	1.04	1.14	1.22	1.32
40.....	.86	.94	1.04	1.14
45.....	.75	.78	.88	.98
50.....	.67	.71	.77	.86
55.....	.60	.64	.69	.77
60.....	.55	.58	.63	.70
65.....	.51	.54	.57	.63
70.....	.48	.50	.53	.59
75.....	.45	.47	.50	.55
80.....	.42	.44	.47	.51
85.....	.39	.42	.44	.48
90.....	.37	.39	.41	.45
95.....	.35	.37	.39	.42
100.....	.33	.35	.37	.39
105.....	.31	.33	.35	.37
110.....	.30	.32	.33	.35
115.....	.28	.29	.31	.33
120.....	.27	.28	.29	.32
125.....	.25	.26	.28	.30
130.....	.24	.25	.26	.28
135.....	.23	.24	.25	.27
140.....	.22	.23	.24	.26
145.....	.21	.22	.23	.25
150.....	.21	.21	.22	.24
155.....	.20	.21	.21	.23
160.....	.20	.20	.21	.22
165.....	.19	.20	.20	.21
170.....	.19	.19	.20	.20

water with each batch of samples to provide a check on the resistance thermometer.

Calculations

By means of the standard curve constructed for the particular thermistor in use, convert the freezing resistance to degrees centigrade. Correct for undercooling, using the following relationship:

$$\Delta T = AT, (1 - 0.0125u)$$

where ΔT is the corrected freezing-point depression, AT , is the observed freezing-point depression, and u is the undercooling in degrees centigrade. A table of factors for correction for undercooling is given by Harris (1925). Calculate osmotic pressure from the equation :

$$OP = 12.06\Delta T - 0.021\Delta T^2$$

where OP is the osmotic pressure in atmospheres and AT is the freezing-point depression in degrees centigrade. Harris and Gortner (1914) present a table of osmotic pressures in atmospheres covering the range of 0 to 2.999° C. freezing-point depression.

References

Richards and Campbell (1948, 1949).

(6b) Freezing-Point Depression of Water in Soil Cores

Apparatus

Use the same resistance thermometer as in Method 6a, except the thermistor must be enclosed in a thin-walled metal tube sealed at the lower end and fastened at the upper end to the glass mounting tube. The calibration curve should be plotted for this thermistor after mounting in the protective metal jacket.

Soil sampling tube to deliver soil cores 1.7 cm. in diameter. Freezing tubes-glass test tube 2.0 cm. inside diameter (2.2 cm. outside diameter) X 17.0 cm. long with rubber stoppers. Soil core holders of rigid tubular material (hard rubber), 1.7 cm. inside diam. (1.9 cm. outside diameter) X 5.1 cm. long. Covers for soil core holders are disks of hard plastic material (Lucite), 1.9 cm. diam. X 3 mm. thick. One-half of the peripheral surface is turned to a smaller diameter (approximately 1.7 cm.) to give a snug fit in the ends of the soil core holders. A tapered hole large enough to accommodate the jacketed thermistor is drilled in one-half of the covers just described.

The Wheatstone bridge, galvanometer, freezing bath, and air-jacket tubes are as described in Method 6a. It is convenient to construct wooden racks to hold about 30 freezing tubes each.

Procedure

Soil cores are pushed from the sampling tube into the soil core holders and cut to length. A solid disk cover is placed on the bottom and a disk with a hole is placed on the top of the soil core holder. The disks are then pressed into position and are held there by the shoulder machined for that purpose. The cores are placed in the freezing tubes that are closed with rubber stoppers bearing the sample numbers. If the samples are to be stored for some time before freezing, both ends of the core holder may be dipped into melted paraffin to prevent moisture loss.

Prior to freezing the sample, a hole is drilled in the center of the soil core. The diameter of this hole should be slightly smaller than the thermistor jacket. The disturbance caused by insertion of the thermistor in an undercooled sample will then initiate freezing. The hole is drilled by hand with a twist drill mounted in a plastic rod having a free fit in the freezing tube.

The freezing tubes containing the samples to be frozen are centered and suspended in the air-jacket

tubes by means of a cork bushing. The thermistor is inserted into a soil core when the freezing bath is initially loaded so that the approach of the temperature of the cores to the bath temperature can be followed. The bath temperature should be held constant $\pm 0.1^\circ$ C. at approximately 1.5" below the expected freezing points for the batch of cores. When the samples attain the bath temperature, freezing of the first core is induced by a twist of the thermistor. Succeeding samples usually start to freeze at the time the thermistor is inserted into the sample. Frozen samples can be replaced in the bath with unfrozen samples, so that for a bath with capacity for 30 samples there is no waiting for undercooling of samples after the initial batch. An interval of about 1 hour is usually sufficient for samples at room temperature to come to bath temperature.

As with the solutions, the change in resistance (temperature) is followed by means of the galvanometer, and the minimum resistance (maximum temperature) recorded as the freezing resistance.

Calculations

The freezing resistance is converted to observed freezing-point depression in degrees centigrade by means of the calibration curve of the thermistor. No convenient method seems to be available at present for making an undercooling correction for water in soil. There is experimental indication that the undercooling correction is small for undercooling of 1.5" C. or less. Freezing-point depression is related to the sum of the tension (suction) and osmotic pressure of water in soil. Calculate the total soil-moisture stress (*SMS*) in atmospheres from the observed freezing-point depression (ΔT_0) of water in soil cores by the relation,

$$SMS = 12\Delta T_0$$

References

Ayers and Campbell (1951), Campbell (1952), Richards and Campbell (1949), and Schofield and Bothelho da Costa (1938).

Soluble Cations and Anions

(7) Calcium and Magnesium by Titration With Ethylenediaminetetraacetate (Versenate)

Reagents

A. Ammonium chloride-ammonium hydroxide buffer solution. Dissolve 47.5 gm. of ammonium chloride in 570 ml. of concentrated ammonium hydroxide and make to 1 liter.

B. Sodium hydroxide, approximately 4 N. Dissolve 160 gm. of sodium hydroxide in 1 liter of water.

C. Standard calcium chloride solution, 0.01 N. Dissolve 0.500 gm. of pure calcium carbonate (calcite crystals) in 10 ml. of approximately 3 N (1+3)

hydrochloric acid and dilute to a volume of exactly 1 liter.

D. Eriochrome black T indicator. Dissolve 0.5 gm. of Eriochrome black T (F 241) and 4.5 gm. of hydroxylamine hydrochloride in 100 ml. of 95 percent ethanol. This indicator is available under several different trade names.

E. Ammonium purpurate indicator. Thoroughly mix 0.5 gm. of ammonium purpurate with 100 gm. of powdered potassium sulfate.

F. Ethylenediaminetetraacetate (Versenate) solution, approximately 0.01 N. Dissolve 2.00 gm. of disodium dihydrogen ethylenediaminetetraacetate and 0.05 gm. of magnesium chloride hexahydrate in water and dilute to a volume of 1 liter. Standardize the solution against reagent C, using the titration procedures given below. The solution is standardized, using each of the indicators D and E, as the normality with E is 3 to 5 percent higher than with D.

Procedure

PRETREATMENT OF SOIL EXTRACTS.-Ammonium acetate and dispersed organic matter, when present in appreciable amounts, must be almost entirely removed from soil extracts prior to titration with Versenate. Evaporation of an aliquot of the soil extract to dryness followed by treatment with aqua regia (3 parts conc. hydrochloric acid + 1 part conc. nitric acid), and a second evaporation to dryness usually suffices for the removal of ammonium acetate and organic matter. Very dark colored soil extracts may require additional treatment with aqua regia. Dissolve the residue in a quantity of water equal to the original volume of the aliquot taken for treatment.

CALCIUM.-Pipet a 5- to 25-ml. aliquot containing not more than 0.1 meq. of calcium into a 3- or 4-inch diameter porcelain casserole. Dilute to a volume of approximately 25 ml. Add 0.25 ml. (5 drops) of reagent B and approximately 50 mg. of E. Titrate with F, using a 10-ml. microburet. The color change is from orange red to lavender or purple. When close to the end point, F should be added at the rate of about a drop every 5 to 10 seconds, as the color change is not instantaneous. A blank containing B, E, and a drop or two of F aids in distinguishing the end point. If the sample is overtitrated with F, it may be back-titrated with C.

CALCIUM PLUS MAGNESIUM.-Pipet a 5- to 25-ml. aliquot containing not more than 0.1 meq. of calcium plus magnesium into a 125-ml. Erlenmeyer flask. Dilute to a volume of approximately 25 ml. Add 0.5 ml. (10 drops) of reagent A and 3 or 4 drops of D. Titrate with F, using a 10-ml. microburet. The color change is from wine red to blue or green. No tinge of the wine-red color should remain at the end point.

Calculations

Milliequivalents per liter of Ca or Ca + Mg = (ml. of Versenate solution used X normality of Versenate solution as determined by appropriate indicator X 1,000) / (ml. in aliquot).

Remarks

Iron, aluminum, and manganese, when present in concentrations greater than 20 p. p. m., and copper, when present in concentrations greater than several tenths of a p. p. m., interfere with the performance of the Eriochrome black T indicator. Usually the concentrations of these metals in water and ammonium acetate extracts of soils of arid regions are insufficient to cause interference. If interference is encountered, it may be overcome as described by Cheng and Bray (1951).

References

Cheng and Bray (1951), Diehl and coworkers (1950).

(8) Calcium by Precipitation as Calcium Oxalate**Apparatus**

Centrifuge and 12-ml. conical tubes.

Reagents

(Keep reagents B, C, D, and E in Pyrex bottles.)

A. Methyl orange, 0.01 percent in water.

B. Hydrochloric acid, approximately 6 N (1+ 1).

C. Oxalic acid, approximately 0.2 N. Dissolve 12.6 gm. of oxalic acid dihydrate in water and make to 1 liter.

D. Ammonium hydroxide, approximately 7 N (1+1).

E. Ammonium hydroxide in ethanol and ether. Mix 20 ml. of conc. ammonium hydroxide with 980 ml. of a mixture of equal volumes of ethanol, ether, and water.

F. Perchloric acid, 4 N. Dilute 340 ml. of 70 percent perchloric acid or 430 ml. of 60 percent perchloric acid to 1 liter.

G. Nitro-ferroin indicator (5-nitro-1,10-phenanthroline ferrous sulfate solution, 0.001 M).

H. Ammonium hexanitrate cerate, 0.01 N in perchloric acid, 1 N. Dissolve 5.76 gm. of ammonium hexanitrate cerate in 250 ml. of 4 N perchloric acid and dilute to 1 liter. The reagent should be standardized in the following manner: Pipet 5 or 10 ml. of fresh standard 0.01 N sodium oxalate into a small beaker containing 5 ml. of reagent F, add 0.2 ml. of G, and titrate with the cerate solution to the pale-blue end point. Determine a blank titration correction on a similar sample minus the oxalate solution. The milliliters of oxalate used multiplied by 0.01 and divided by the corrected milliliters of cerate provide the normality of the cerate. Do not attempt to adjust the solution to exactly 0.01 N. Restandardize each time the reagent is used if more than 2 days have elapsed since the last standardization. Keep in a dark bottle away from light.

Procedure

Pipet an aliquot containing 0.005 to 0.08 meq. of calcium into a 12-ml. conical centrifuge tube, dilute or evaporate¹⁴ to 5 ml., and add 1 drop of reagent A, 2 drops of B, and 1 ml. of C. Heat to the boiling point in a water bath. While twirling the tube, add D drop wise until the solution just turns yellow. Replace in the bath, and, after 30 min., cool the tube in air or in water. If necessary, add more D to keep the solution just yellow.

Centrifuge at $RCF = 1,000$ for 10 min. Carefully decant the supernatant liquid into another 12-ml. conical centrifuge tube and save for the magnesium determination. Stir the precipitate and rinse the sides of the tube with a stream of 5 ml. of reagent E blown from a pipet. Centrifuge at $RCF = 1,000$ for 10 min. Decant and drain the tube by inversion on filter paper for 10 min. Wipe the mouth of the tube with a clean towel or lintless filter paper.

Blow into the tube 3 ml. of reagent F from a pipet. When the precipitate is dissolved, add 0.1 ml. of G. Titrate with H from a 10-ml. microburet to the pale-blue end point. If more than 5 ml. of H is required, transfer the sample to a small beaker and complete the titration. Determine the blank correction in the same manner; it is usually about 0.03 ml.

Calculations

Milliequivalents per liter of Ca = (corrected ml. of cerate solution X normality of cerate X 1,000) / (ml. in aliquot).

Reference

Reitemeier (1943).

(9) Magnesium by Precipitation as Magnesium Ammonium Phosphate**Apparatus**

Centrifuge, 12-ml. conical tubes, and photoelectric colorimeter.

Reagents

A. Ammonium chloride, 3 percent solution. Dissolve 3 gm. of ammonium chloride in water and dilute to 100 ml. Filter before use.

B. Ammonium dihydrogen phosphate, 5 percent solution. Dissolve 5 gm. of ammonium dihydrogen phosphate in water and dilute to 100 ml. Filter before use.

C. Phenolphthalein, 1 percent in 60 percent ethanol.

¹⁴ Evaporation operations carried on with centrifuge tubes in a water bath may be speeded up by the use of an air blower. For this, a bank of glass nozzle-tubes in an array to match positions in the centrifuge tube rack is supplied with air from a compressed air system. A stream of air is thus introduced into each drying tube.

D. Ammonium hydroxide, **conc.**

E. Ammonium hydroxide in ethanol and **ether**. Mix 20 ml. of **conc.** ammonium hydroxide with 980 ml. of a mixture of equal volumes of ethanol, ether, and **water**.

F. Magnesium sulfate solution, approximately 0.01 N, standardized. This is best prepared by dilution of a more concentrated solution of magnesium sulfate that has been standardized by gravimetric determination of magnesium (Method 78).

G. Sulfuric acid. Approximately 5 N (1-1-6).

H. Ammonium vanadate, 0.25 percent solution. Dissolve 2.5 gm. of ammonium vanadate in 500 ml. of boiling water, cool somewhat, and then add 60 ml. of reagent G. Cool to room temperature and dilute to 1 liter. Store in a brown bottle.

I. Ammonium molybdate, 5 percent solution. Dissolve 50 gm. of ammonium molybdate in 1 liter of water. Store in a brown bottle.

Procedure

To the 12-ml. conical centrifuge tube containing the calcium-free sample from Method 8, add 1 ml. each of reagents A and B and 1 drop of C. Heat to 90° C. in a water bath and then add D until permanently pink. After 15 min., add an additional 2 ml. of D. Stopper and let stand overnight.

Centrifuge at $RCF=1,000$ for 10 min., decant carefully, drain on filter paper for 10 min., and wipe the mouth of the tube with a clean towel or lintless filter paper. Wash the precipitate and sides of the tube with a stream of 5 ml. of reagent E from a pipet equipped with a rubber bulb or by a similar arrangement. Centrifuge at $RCF=1,000$ for 5 min., decant, drain for 5 min., and wipe the mouth of the tube. Repeat this washing procedure once.

Pipet 10 ml. of reagent G into the tube and twirl for a few seconds. After 5 min. wash the contents into a 100-ml. volumetric flask. Dilute to about 60 ml. and pipet 10 ml. each of H and I into the flask while twirling rapidly. Dilute to the mark and mix. After 10 min. measure the difference in light transmission of the sample and water, using optical cells and a 460-m μ filter.

Starting at the beginning of the Procedure above, prepare a photometer calibration curve on semilogarithmic graph paper, for 0, 0.5, 1, 2, 3, 4, and 5 ml. of reagent F. One ml. of 0.2 N oxalic acid should be added to each tube of standard before precipitating the magnesium. The amount of magnesium in the aliquot is obtained by simple interpolation on the curve.

Calculations

Milliequivalents per liter of Mg = (meq. of Mg found by interpolation \times 1,000) / (ml. in Ca aliquot \times 0.98). The factor of 0.98 corrects for magnesium lost in the washings from the calcium precipitate.

References

Kitson and Mellon (1944), Reitemeier (1943).

(10) Sodium

(10a) Sodium by Flame Photometer

Apparatus

Perkin-Elmer model 52 flame photometer with acetylene or propane burner.

Reagents

A. Ammonium acetate, approximately 1 N. To 700 or 800 ml. of water add 57 ml. of **conc.** acetic acid and then 68 ml. of **conc.** ammonium hydroxide. Dilute to a volume of 1 liter and adjust to pH 7.0 by the addition of more ammonium hydroxide or acetic acid.

B. Sodium chloride, 0.04 N. Dissolve 2.338 gm. of dry sodium chloride in water and dilute to exactly 1 liter.

C. Sodium chloride, 0.04 N in 1 N ammonium acetate. Dissolve 2.338 gm. of dry sodium chloride in reagent A. Dilute to exactly 1 liter with additional A.

D. Lithium chloride, 0.05 N. Dissolve 2.12 gm. of dry lithium chloride in water and dilute to exactly 1 liter.

Procedure

Using reagents B and D prepare a series of standard sodium chloride solutions, each containing the same concentration of lithium chloride. Prepare a similar series of standard sodium chloride solutions, using reagents C and D, and use A for dilution. Recommended concentrations of sodium chloride are 0, 0.2, 0.4, 0.6, 0.8, 1, 2, 3, and 4 meq./l. The optimum concentration of lithium chloride varies with individual flame photometers but is usually 5 to 10 meq./l. Standard solutions made up with water are employed for the analysis of waters and water extracts of soils; whereas, standard solutions made up in ammonium acetate solutions are used for the analysis of ammonium acetate extracts of soils. Calibrate the flame photometer for operation over the concentration range 0 to 1 meq./l. of sodium, using the first 6 standard solutions of the appropriate series. Use the first and the last 4 solutions of the appropriate series to calibrate the instrument for operation over the concentration range 0 to 4 meq./l. of sodium.

Pipet an aliquot of the solution to be analyzed, containing less than 0.2 meq. of sodium, into a 50-ml. volumetric flask. Add an amount of reagent D that, when diluted to a volume of 50 ml., will give a concentration of lithium chloride exactly equal to that in the standard sodium chloride solutions. Dilute to volume with water, or with A, if ammonium acetate extracts are being analyzed. Mix and determine the sodium concentration by use of the flame photometer and the appropriate calibration curve.

Calculations

Milliequivalents per liter of Na in water or extract = (meq./l. of Na as found by interpolation on calibration curve \times 50) / (ml. in aliquot).

(10b) Sodium by Precipitation as Sodium Uranyl Zinc Acetate

Apparatus

Centrifuge and 12-ml. conical tubes.

Reagents

A. Uranyl zinc acetate. Weigh 300 gm. of uranium acetate dihydrate, 900 gm. of zinc acetate dihydrate, and 10 mg. of sodium chloride into a large flask. Add 82 ml. of glacial acetic acid and 2,618 ml. of water. Stir or shake until the salts are dissolved, leaving only a small amount of sodium uranyl zinc acetate precipitate. Filter before use.

B. Acetic acid-ethanol. Mix 150 ml. of glacial acetic acid with 850 ml. of 95 percent ethanol. Shake with an excess of sodium uranyl zinc acetate crystals. Filter before use. Sodium uranyl zinc acetate crystals may be prepared as follows: Add 125 ml. of reagent A to 5 ml. of 2 percent sodium chloride solution, stir, and after 15 min. collect the precipitate in a porous-bottomed porcelain crucible. Wash several times with glacial acetic acid, then several times with ether, and finally dry in a desiccator.

C. Ether, anhydrous.

Procedure

Pipet an aliquot containing 0.003 to 0.07 meq. of sodium into a 12-ml. conical centrifuge tube. Evaporate on a water bath to 0.5 ml. Cool, add 8 ml. of reagent A, and mix by stirring with an aluminum wire bent into a loop. Let stand 1 hour. Centrifuge at $RCF = 1,000$ for 10 min. Decant and drain on filter paper for 10 min. Wipe the mouth of the tube with a clean towel or lintless filter paper. Suspend the precipitate and wash the sides of the tube, using 5 ml. of B blown from a pipet equipped with a rubber bulb. Centrifuge for 10 min., decant, and drain for 1 min. Wipe the mouth of the tube. Wash with 5 ml. of C, but centrifuge for only 5 min. Decant carefully without draining. Repeat washing and centrifuging once. Clean the outside of tube with chamois, dry for an hour or more at 60° C., cool in a desiccator, and weigh. Add 10 ml. of water, stir with the wire until the sodium precipitate is dissolved, centrifuge for 5 min., decant carefully, and drain for 5 min. on filter paper. Suspend the insoluble precipitate and wash the sides of the tube with 5 ml. of B blown from a pipet. Centrifuge for 5 min., and decant. Wash with 5 ml. of C, centrifuge for 5 min., clean tube with chamois, dry for an hour at 60°, cool in a desiccator, and weigh. The difference between the two weights is the weight of the sodium precipitate.

Calculations

Milliequivalents per liter of Na = (gm. of Na precipitate \times 650.2) / (ml. in aliquot).

Reference

Reitemeier (1943).

(11) Potassium

(11 a) Potassium by Flame Photometer

Apparatus

Perkin-Elmer model 52 flame photometer with acetylene or propane burner.

Reagents

A. Ammonium acetate, approximately 1 N. To 700 or 800 ml. of water add 57 ml. of conc. acetic acid and then 68 ml. of conc. ammonium hydroxide. Dilute to a volume of 1 liter and adjust to pH 7.0 by the addition of more ammonium hydroxide or acetic acid.

B. Potassium chloride, 0.02 N. Dissolve 1.491 gm. of dry potassium chloride in water and dilute to a volume of exactly 1 liter.

C. Potassium chloride, 0.02 N in 1 N ammonium acetate. Dissolve 1.491 gm. of dry potassium chloride in reagent A. Dilute to a volume of exactly 1 liter with additional A.

D. Lithium chloride, 0.05 N. Dissolve 2.12 gm. of dry lithium chloride in water and dilute to 1 liter.

Procedure

Using reagents B and D, prepare a series of standard potassium chloride solutions, each containing the same concentration of lithium chloride. Prepare a similar series of standard potassium solutions using reagents C and D, and use A for dilution. The concentrations of potassium chloride are 0, 0.1, 0.2, 0.3, 0.4, 0.5, 1, 1.5, and 2 meq./l. The optimum concentration of lithium chloride varies with individual flame photometers but is usually 5 to 10 meq./l. Standard solutions made up in water are employed for the analysis of waters and water extracts of soils; whereas, those made up in ammonium acetate solution are used for the analysis of ammonium acetate extracts of soils. Calibrate the flame photometer for operation over the concentration range 0 to 0.5 meq./l. of potassium, using the first 6 standard solutions of the appropriate series. Use the first and the last 4 solutions of the appropriate series to calibrate the instrument for operation over the concentration range 0 to 2 meq./l. of potassium.

Pipet an aliquot of the solution to be analyzed containing less than 0.1 meq. of potassium into a 50-ml. volumetric flask. Add an amount of reagent D which, when diluted to a volume of 50 ml., will give a concentration of lithium chloride exactly equal to that in the standard potassium chloride solutions. Dilute to volume with water or with A, if ammonium acetate extracts are being analyzed, mix, and determine the potassium concentration by use of the flame photometer and the appropriate calibration curve.

Calculations

Milliequivalents per liter of K in water or extract = (meg./l. of K as found by interpolation on calibration curve \times 50) / (ml. in aliquot).

(11 b) Potassium by Precipitation as Potassium Dipicrylamine**Apparatus**

Photoelectric colorimeter, centrifuge, and 12-ml. conical tubes.

Reagents

A. Lithium dipicrylamine solution. Dissolve 1.65 gm. of lithium carbonate in 250 ml. of water. Warm to 50° C. and then add 9 gm. of dipicrylamine. After the dipicrylamine has dissolved, filter and dilute 200 ml. of this solution to 1 liter. To the remaining portion of approximately 50 ml., add 0.25 gm. of potassium chloride. Separate and wash the resulting potassium dipicrylamine precipitate with a few milliliters of water by means of a centrifuge. Add the potassium salt to the warm solution of lithium dipicrylamine and shake for 30 min. Filter the solution before use.

B. Potassium chloride, 0.010 N. Dissolve 0.7456 gm. of dry potassium chloride in water and dilute to exactly 1 liter.

C. Phenolphthalein, 1 percent in 60 percent ethanol.

D. Sodium hydroxide, approximately 1 N. Dissolve 40 gm. of sodium hydroxide in water and dilute to 1 liter.

Procedure

Pipet an aliquot containing 0.005-0.035 meq. of potassium into a 12-ml. conical centrifuge tube. Add 1 drop of reagent C and then D until pink. Evaporate to dryness. This insures removal of ammonium. Cool and then add exactly 2 ml. of A. Grind the salt residue in the bottom of the tube by means of a glass rod and allow 1 hour for precipitation. Centrifuge the tube at $RCF = 1,000$ for 1 min. Remove a 0.2-ml. aliquot from the supernatant liquid by means of a blood pipet and dilute to a volume of 50 ml. Compare the light transmission in an optical cell through a 510- $m\mu$ filter with that of water in similar cell. Prepare a calibration curve for each set of samples by carrying a series of 0.5, 1, 1.5, 2, 2.5, 3, 3.5 ml. of B through the same operations. The amount of potassium in the sample is found by interpolation on this curve. When plotted on a linear scale the curve should be slightly S-shaped. The temperature at which the calibration curve is prepared should be within 2° C. of that at which the unknown determinations are made.

Calculations

Milliequivalents per liter of K = (meq. of K in aliquot as found by interpolation $\times 1,000$) / (ml. in aliquot).

Reference

Williams (1941).

(12) Carbonate and Bicarbonate by Titration With Acid**Reagents**

A. Phenolphthalein, 1 percent in 60 percent ethanol.
B. Methyl orange, 0.01 percent in water.
C. Sulfuric acid, approximately 0.010 N, standardized.

Procedure

Pipet an aliquot containing 0.005 to 0.04 meq. of chloride into a 15-ml. wide-mouthed porcelain crucible or a small porcelain casserole. Chloride is specified here because the same sample is subsequently used for the chloride determination in Method 13. Add 1 drop of reagent A. If the solution turns pink, add C from a 10-ml. microburet dropwise at 5-second intervals until the color just disappears. Designate this buret reading as y . Add 2 drops of B and titrate to the first orange color. Designate the new buret reading as z . Save the titrated sample for the chloride determination.

An indicator correction blank using boiled water should be determined and applied if it is not negligible. The lighting should be adequate for the recognition of the various colors. The use of comparison color standards at the correct end points is helpful.

Calculations

1. Milliequivalents per liter of $CO_3 = (2y \times \text{normality of } H_2SO_4 \times 1,000) / (\text{ml. in aliquot})$.
2. Milliequivalents per liter of $HCO_3 = (z - 2y) \times \text{normality of } H_2SO_4 \times 1,000 / (\text{ml. in aliquot})$.

Reference

Reitemeier (1943) .

(13) Chloride by Titration With Silver Nitrate**Reagents**

A. Potassium chromate, 5 percent solution. Dissolve 5 gm. of potassium chromate in 50 ml. of water and add 1 N silver nitrate dropwise until a slight permanent red precipitate is produced. Filter and dilute to 100 ml.

B. Silver nitrate, 0.005 N. Dissolve 0.8495 gm. of silver nitrate in water and dilute to exactly 1 liter. Keep in a brown bottle away from light.

Procedure

To the sample preserved from the carbonate-bicarbonate determination, add 4 drops of reagent A. While stirring, titrate under a bright light with B from a 10-ml. microburet to the first permanent reddish-brown color. The titration blank correction varies with the volume of the sample at the end point, and usually increases regularly from about 0.03 to 0.20 ml. as the volume increases from 2 to 12 ml.

Calculations

Milliequivalents per liter of Cl = (ml. of AgNO_3 - ml. of AgNO_3 for blank) $\times 0.005 \times 1,000 /$ (ml. in aliquot).

Reference

Reitemeier (1943).

(14) Sulfate**(14a) Sulfate by Precipitation as Barium Sulfate****Apparatus**

Centrifuge and 12-ml. conical tubes.

Reagents

- A. Methyl orange, 0.01 percent in water.
- B. Hydrochloric acid, approximately 1 N.
- C. Barium chloride, approximately 1 N. Dissolve 122 gm. of barium chloride dihydrate in water and dilute to 1 liter.
- D. Ethanol, 50 percent by volume.

Procedure

Pipet an aliquot containing 0.05 to 0.5 meq. of sulfate into a clean 12-ml. conical centrifuge tube of known weight. Dilute or evaporate to about 5 ml. Add 2 drops of reagent A, then B dropwise until pink, and then 1 ml. of B in excess. Heat to boiling in water bath. While twirling the tube add 1 ml. of C dropwise. Return to the hot water bath for 30 min. and then cool at least an hour in air.

Centrifuge at $RCF=1,000$ for 5 min. Carefully decant and let drain by inversion on filter paper for 10 min. Wipe the mouth of the tube with a clean towel or lintless filter paper.

Stir the precipitate and rinse the sides of the tube with a stream of 5 ml. of reagent D blown from a pipet. If necessary, loosen precipitate from bottom of tube by means of a wire bent in appropriate shape. Centrifuge for 5 min. and decant, but do not drain. Repeat this washing and decanting operation once. Wipe the outside of tube carefully with chamois and do not subsequently touch with fingers. Dry overnight in an oven at 105°C . Cool in a desiccator and weigh.

Calculations

Milliequivalents per liter of $\text{SO}_4 =$ (mg. of BaSO_4 precipitate $\times 8.568$) / (ml. in aliquot).

(Note. Care must be taken in the preparation or concentration of the unknowns so as not to precipitate foreign material which might be weighed as barium sulfate.)

(14b) Sulfate by Precipitation as Calcium Sulfate**Apparatus**

Wheatstone bridge, conductivity cell, centrifuge, and 50-ml. conical tubes.

Reagents

- A. Acetone.
- B. Calcium chloride, approximately 1 N. Dissolve 74 gm. of calcium chloride dihydrate in water and dilute to 1 liter.

Procedure

Pipet an aliquot containing 0.05 to 0.5 meq. of sulfate into a 50-ml. conical centrifuge tube. Dilute or concentrate to a volume of 20 ml. Add 1 ml. of reagent B and 20 ml. of A. Mix the contents of the tube and let stand until the precipitate flocculates. This usually requires 5 to 10 min. Centrifuge at $RCF=1,000$ for 3 min., decant the supernatant liquid, invert the tube, and drain on filter paper for 5 min. Disperse the precipitate and rinse the wall of the tube with a stream of 10 ml. of A blown from a pipet. Again centrifuge at $RCF=1,000$ for 3 min., decant the supernatant liquid, invert the tube, and drain on filter paper for 5 min. Add exactly 40 ml. of distilled water to the tube, stopper, and shake until the precipitate is completely dissolved. Measure the electrical conductivity of the solution, using Method 4b, and correct the conductivity reading to 25°C . Determine the concentration of CaSO_4 in the solution by reference to a graph showing the relationship between the concentration and the electrical conductivity of CaSO_4 solutions. This graph may be constructed by means of the following data from the International Critical Tables.

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

Calculations

Milliequivalents per liter of $\text{SO}_4 =$ (meq./l. of CaSO_4 from electrical conductivity reading) \times (ml. in aliquot / ml. of water used to dissolve precipitate).

Reference

Bower and Huss (1948).

(15) Nitrate by Phenoldisulfonic Acid**Apparatus**

Photoelectric colorimeter.

Reagents

A. Phenoldisulfonic acid. Dissolve 25 gm. of phenol in 150 ml. of conc. sulfuric acid, add 75 ml. of fuming sulfuric acid (13 to 15 percent **SO₃**), and heat at 100° C. for 2 hours.

B. Potassium nitrate, 0.010 N. Dissolve 1.011 gm. of dry potassium nitrate in water and dilute to exactly 1 liter.

C. Silver sulfate, 0.020 N. Dissolve 3.12 gm. silver sulfate in 1 liter of water.

D. Ammonium hydroxide, approximately 7 N (1+1).

E. Calcium oxide.

Procedure

First determine the concentration of chloride in an aliquot as directed under Method 13. Pipet another aliquot containing 0.004 to 0.04 meq. of nitrate into a 25-ml. volumetric flask. Add an amount of reagent C equivalent to the amount of chloride present. Dilute to volume and mix. Transfer most of the suspension to a 50-ml. centrifuge tube and separate the precipitate by centrifuging. After transferring the solution to another centrifuge tube, flocculate any suspended organic matter by adding about 0.1 gm. of E and clear by again centrifuging. Pipet a 10-ml. aliquot representing 2/5 of the sample into an 8-cm. evaporating dish. Evaporate the aliquot to dryness, cool, and dissolve the residue in 2 ml. of A. After 10 min., add 10 ml. of water and transfer to a 100-ml. volumetric flask. Make alkaline by the addition of D, dilute to volume, and mix. Measure the light transmission through a 460-m μ filter in an optical cell against that of water in a similar cell.

Prepare a calibration curve by pipeting 0, 0.2, 0.4, 0.8, 1.2, and 1.6 ml. portions of reagent B into evaporating dishes and treating as above omitting the additions of C and E, and the clarifying procedure.

Calculations

Milliequivalents per liter of $\text{NO}_3 = (\text{meq. of NO}_3 \text{ in aliquot as found by interpolation on NO}_3 \text{ curve}) \times 1,000 / (\text{ml. in aliquot})$.

(16) Silicate as Silicomolybdate**Apparatus**

Photoelectric colorimeter.

Reagents

A. Ammonium molybdate, 10 percent solution. Dissolve 10 gm. of ammonium molybdate in water and dilute to 100 ml.

B. Sulfuric acid, approximately 5 N (1 + 6).

C. Sodium silicate, 0.01 N. Dissolve 1.5 gm. of $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ in 1 liter of water. Determine the silicate (SiO_3) concentration of this solution, using a 100-ml. aliquot and Method 76a (ch. 8). Adjust the remaining solution to exactly 0.01 N by the addition of a calculated amount of water. Store in a plastic bottle.

Procedure

Pipet an aliquot containing 0.005 to 0.05 meq. of silicate into a 50-ml. volumetric flask. Dilute to a volume of 40 to 45 ml. with water. Add 2 ml. of reagent A and then 1 ml. of B. Dilute to 50 ml., mix, and after 15 min. measure the light transmission through a 420-m μ filter in an optical cell against water in a similar cell. Prepare a calibration curve by carrying a series of 0, 1, 2, 3, 4, and 5 ml. of C through the same operations.

Calculations

Milliequivalents per liter of $\text{SiO}_3 = (\text{meq. of SiO}_3 \text{ in aliquot as found by interpolation } \times 1,000) / (\text{ml. in aliquot})$.

Reference

Snell and Snell (1936).

(17) Boron

Determine boron as directed in Method 73b. If the solution is colored, transfer an aliquot to a platinum dish, make alkaline with NaOH, reagent A, and evaporate to dryness in an oven at 95° C. Ignite over an open flame until the residue fuses. Cool, add 5 ml. dilute HCl, reagent C, and complete as suggested in Method 73b under paragraph, Boron Concentration Too Low.

Exchangeable Cations**(18) Exchangeable Cations****Apparatus**

Centrifuge, 50-ml. round-bottom, narrow-neck centrifuge tubes, and reciprocating shaker.

Reagents

A. Ammonium acetate solution, 1.0 N. To 700 or 800 ml. of water add 57 ml. of conc. acetic acid and then 68 ml. of conc. ammonium hydroxide. Dilute to a volume of 1 liter and adjust to pH 7.0 by the addition of more ammonium hydroxide or acetic acid.

B. Nitric acid, conc.

C. Hydrochloric acid, conc.

D. Acetic acid, approximately 0.1 N.

Procedure

Ammonium acetate extractable cations : Samples for this determination should be approximately 4 gm. for

medium- and fine-textured soils and 6 gm. for coarse-textured soils. Weigh samples to an accuracy of 1 percent and correct for the air-dry moisture content. Place the sample in a centrifuge tube. Add 33 ml. of reagent A to the tube, stopper, and shake for 5 min. Remove the stopper and centrifuge at $RCF=1,000$ until the supernatant liquid is clear. This usually requires 5 min. Decant the supernatant liquid as completely as possible into a 100-ml. volumetric flask. Extract with A a total of 3 times by this procedure, decanting into the same flask. Dilute to volume, mix, and determine the amounts of the various extracted cations by flame photometric or chemical methods. Flame photometric analyses may be made directly upon aliquots of the extract. If chemical methods are to be employed for the determination of cations, pretreat the extract in the following manner: Transfer to a 250-ml. beaker and evaporate to dryness on a hot plate or steam bath. Wash down the walls of the beaker with a small quantity of water and again evaporate to dryness. Add 1 ml. of B and 3 ml. of C, evaporate, and dissolve the residue in 20 ml. of D. Filter through low-ash content filter paper into a 50-ml. volumetric flask, using water to wash the beaker and filter paper. Dilute to volume.

Soluble cations: Prepare a saturated soil paste as described in Method 2, using a 200- to 1,000-gm. sample of soil. The weight of soil will depend upon the number of cations to be determined, the analytical methods employed, and the salt content of the soil. Determine the saturation percentage by Method 27. Obtain the saturation extract as described under Method 3a and determine the soluble cation concentrations by flame photometric or chemical methods.

Calculations

Ammonium acetate extractable cations in $\text{meq./100 gm.} = (\text{cation conc. of extract in meq./l.} \times 10) / (\text{wt. of sample in gm.})$.

Soluble cations in $\text{meq./100 gm.} = (\text{cation conc. of saturation extract in meq./l.}) \times (\text{saturation percentage}) / 1,000$.

Exchangeable cations in $\text{meq./100 gm.} = (\text{extractable cations in meq./100 gm.}) - (\text{soluble cations in meq./100 gm.})$.

Reference

Bower and others (1952).

(19) Cation-Exchange-Capacity

Apparatus

Centrifuge, 50-ml. round-bottom, narrow-neck centrifuge tube, and reciprocating shaker.

Reagents

A. Sodium acetate solution, 1.0 N. Dissolve 136 gm. of sodium acetate trihydrate in water and dilute

to a volume of 1 liter. The pH value of the solution should be approximately 8.2.

B. Ethanol, 95 percent.

C. Ammonium acetate solution, 1.0 N. To 700 or 800 ml. of water add 57 ml. of conc. acetic acid and then 68 ml. of conc. ammonium hydroxide. Dilute to a volume of 1 liter and adjust to pH 7.0 by the addition of more ammonium hydroxide or acetic acid.

Procedure

Samples for this determination should be approximately 4 gm. for medium- and fine-textured soils and 6 gm. for coarse-textured soils. Weigh samples to an accuracy of 1 percent and correct for the air-dry moisture content. Place the sample in a centrifuge tube. Add 33 ml. of reagent A, stopper the tube, and shake for 5 min. Unstopper and centrifuge at $RCF=1,000$ until the supernatant liquid is clear. This usually requires 5 min. Decant the supernatant liquid as completely as possible and discard. Treat the sample in this manner with 33-ml. portions of A a total of 4 times, discarding the supernatant liquid each time. Add 33 ml. of B to the tube, stopper, shake for 5 min., unstopper, and centrifuge until the supernatant liquid is clear. Decant and discard the supernatant liquid. Wash the sample with 33-ml. portions of B a total of 3 times. The electrical conductivity of the supernatant liquid from the third washing should be less than 40 micromhos/cm. Replace the adsorbed sodium from the sample by extraction with three 33-ml. portions of C and determine the sodium concentration of the combined extracts after dilution to 100 ml. as described under Method 18.

Calculations

Cation-exchange-capacity in $\text{meq./100 gm.} = (\text{Na conc. of extract in meq./l.} \times 10) / (\text{wt. of sample in gm.})$.

Reference

Bower and others (1952).

(20) Exchangeable-Cation Percentages

(20a) Exchangeable - Cation Percentages by Direct Determination

Procedure

Determine the exchangeable-cation contents and the cation-exchange-capacity, using Methods 18 and 19.

Calculations

Exchangeable-cation percentage = $(\text{exchangeable-cation content in meq./100 gm.} \times 100) / (\text{cation-exchange-capacity in meq./100 gm.})$.

(20b) Estimation of Exchangeable-Sodium-Percentage and Exchangeable-Potassium-Percentage From Soluble Cations

Procedure

Prepare a saturation extract of the soil as described under Methods 2 and 3a. Determine the calcium plus magnesium, sodium, and potassium concentrations of the saturation extract, using Methods 7, 10, and 11, respectively.

Calculations

Exchangeable-sodium-percentage

$$\frac{100 (-0.0126 + 0.01475x)}{1 + (-0.0126 + 0.01475x)}$$

where x is equal to the sodium-adsorption-ratio.

Exchangeable-potassium-percentage

$$\frac{100 (0.0360 + 0.1051x)}{1 + (0.0360 + 0.1051x)}$$

where x is equal to the potassium-adsorption-ratio. The sodium-adsorption-ratio and the potassium-adsorption-ratio are calculated as follows:

Sodium-adsorption-ratio = $\frac{Na^+}{\sqrt{(Ca^{++} + Mg^{++})/2}}$
and

Potassium-adsorption-ratio = $\frac{K^+}{\sqrt{(Ca^{++} + Mg^{++})/2}}$
where Na^+ , K^+ , Ca^{++} , and Mg^{++} refer to the concentrations of designated cations expressed in milliequivalents per liter.

A nomogram, which relates soluble sodium and soluble calcium plus magnesium concentrations to the sodium-adsorption-ratio, is given in figure 27. Also included in the nomogram is a scale for estimating the corresponding exchangeable-sodium-percentage, based on the linear equation given in connection with figure 9 (ch. 2). To use this nomogram, lay a straightedge across the figure so that the line coincides with the sodium concentration on scale A and with the calcium plus magnesium concentration on scale B. The sodium-adsorption-ratio and the estimated exchangeable-sodium-percentage are then read on scales C and D, respectively.

Supplementary Measurements

(21) pH Determinations

(21a) pH Reading of Saturated Soil Paste

Apparatus

pH meter with glass electrode.

Procedure

Prepare a saturated soil paste with distilled water as directed in Method 2 and allow paste to stand at least

1 hour. Insert the electrodes into the paste and raise and lower repeatedly until a representative pH reading is obtained.

(21b) pH Reading of Soil Suspension

Procedure

Prepare a soil suspension, using distilled water, shake intermittently for an hour, and determine pH reading.

(21c) pH Reading of Waters, Solutions, Soil Extracts

Procedure

Determine pH reading by means of a glass electrode assembly with the solution in equilibrium with a known CO₂ atmosphere.

Remarks

Opinion varies as to the proper method for making pH readings. It is desirable to select a definite procedure and follow it closely, so that the readings will be consistent and have maximum diagnostic value. The method used should be described accurately so as to aid others in the interpretation of results.

The CO₂ status influences pH readings, and should be controlled or specified. Ordinarily, readings are made at the CO₂ pressure of the atmosphere. A special high-pH glass electrode should be used for pH values appreciably above 9.0.

(22) Gypsum

(22a) Gypsum by Precipitation With Acetone (Qualitative)

Reagent

Acetone.

Procedure

Weigh 10 to 20 gm. of air-dried soil into an 8-oz. bottle and add a measured volume of water sufficient to dissolve the gypsum present. Stopper the bottle and shake by hand 6 times at 15-min. intervals or agitate for 15 min. in a mechanical shaker. Filter the extract through paper of medium porosity. Place about 5 ml. of the extract in a test tube, add an approximately equal volume of acetone, and mix. The formation of a precipitate indicates the presence of gypsum in the soil.

Remarks

The soil should not be oven-dried, because heating promotes the conversion of CaSO₄·2H₂O to CaSO₄·0.5H₂O. The latter hydrate has a higher solubility in water for an indefinite period following its solution.

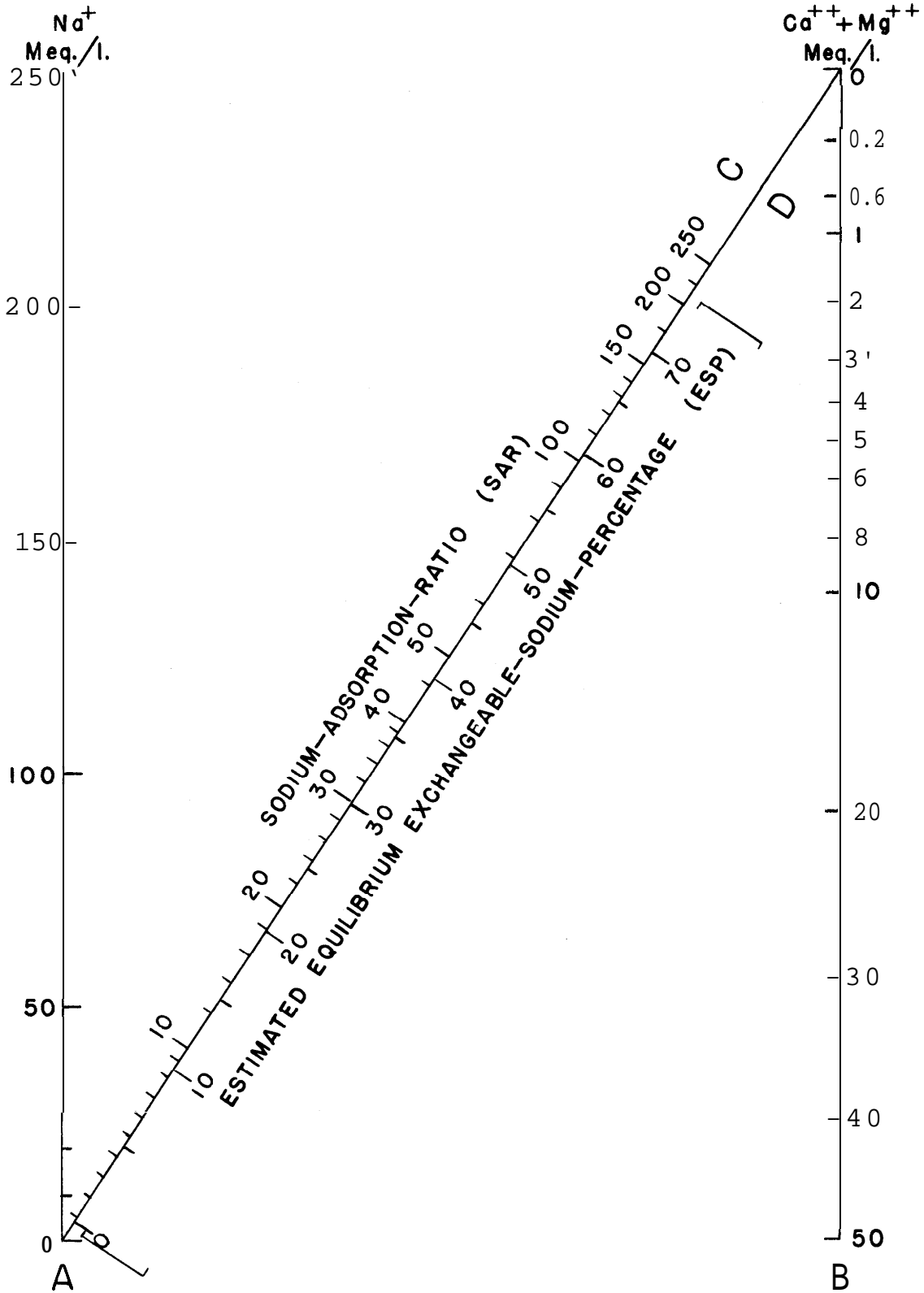


FIGURE 27.-Nomogram for determining the SAR value of a saturation extract and for estimating the corresponding *ESP* value of soil at equilibrium with the extract.

(22b) Gypsum by Precipitation With Acetone (Quantitative)**Apparatus**

Centrifuge, 50-ml. conical centrifuge tubes, conductivity cell, and Wheatstone bridge.

Reagent

Acetone.

Procedure

Transfer a 20-ml. aliquot of the filtered extract obtained as described in Method 22a into a 50-ml. conical centrifuge tube. Add 20 ml. of acetone and mix. Let stand until the precipitate flocculates. This usually requires 5 to 10 min. Centrifuge at $RCF = 1,000$ for 3 min., decant the supernatant liquid, invert the tube, and drain on filter paper for 5 min. Disperse the precipitate and rinse the wall of the tube with a stream of 10 ml. of acetone blown from a pipet. Again, centrifuge for 3 min., decant the supernatant liquid, invert the tube, and drain on filter paper for 5 min. Add exactly 40 ml. of distilled water to the tube, stopper, and shake until the precipitate is completely dissolved. Measure the electrical conductivity of the solution, using Method 4b, and correct the conductivity reading to 25° C. Determine the concentration of gypsum in the solution by reference to a graph showing the relationship between the concentration and the electrical conductivity of gypsum solutions. This graph may be constructed by means of the following data from the International Critical Tables.

CaSO ₄ concentration (meq./l.):	Electrical conductivity at 25° C. Mmhos/cm.
1	0.121
2	.226
5	.500
10	.900
20	1.584
30.5	2.205

Calculations

Milliequivalents of CaSO₄ in aliquot = (meq./l. of CaSO₄ from conductivity reading) X (ml. of water used to dissolve precipitate) /1,000.

Milliequivalents of gypsum per 100 gm. of soil = $100 \times (\text{meq. of CaSO}_4 \text{ in aliquot}) / (\text{soil: water ratio} \times \text{ml. of soil-water extract used})$.

Remarks

Sodium and potassium sulfates when present in sufficiently high concentrations are also precipitated by acetone. The maximum concentrations of sodium sulfate and of potassium sulfate that may be tolerated are 50 and 10 meq./l., respectively.

At a 1: 5 soil-water ratio, water will dissolve approximately 15 meq. of gypsum per 100 gm. of soil. If it is found that the gypsum content of the soil ap-

proaches 15 meq./100 gm. by use of a 1: 5 soil-water extract, the determination should be repeated, using a more dilute extract.

Reference

Bower and Huss (1948).

(22c) Gypsum by Increase in Soluble Calcium Plus Magnesium Content Upon Dilution**Procedure**

Determine the saturation percentage and obtain a saturation extract of the soil using Methods 27 and 3a. Prepare another water extract of the soil, using a moisture content sufficient to dissolve the gypsum present as described under Method 22a. Determine the calcium plus magnesium concentrations of the two extracts by Method 7.

Calculations

Soluble Ca+Mg at the saturation percentage in meq./100 gm. = (Ca+ Mg conc. of saturation extract in meq./l.) X (saturation percentage) /1,000.

Soluble Ca+Mg at the high moisture percentage in meq./100 gm. = (Ca + Mg conc. of dilute extract in meq./l.) X (moisture percentage) /1,000.

Gypsum in meq./100 gm. of soil = (soluble Ca+Mg at the high moisture percentage in meq./100 gm.) - (soluble Ca+Mg at the saturation percentage in meq./100 gm.) .

(22d) Gypsum Requirement¹⁵**Reagent**

A. Approximately saturated gypsum solution of known calcium concentration. Place about 5 gm. of CaSO₄·2H₂O and 1 liter of water in a flask, stopper, and shake by hand several times during a period of 1 hr., or for 10 min. in a mechanical shaker. Filter and determine the calcium concentration of a 5-ml. aliquot of the solution by Method 7. The calcium concentration should be at least 28 meq./l.

Procedure

Weigh 5 gm. of air-dried soil into a 4-oz. bottle. Add 100 ml. of reagent A by means of a pipet. Stopper the bottle and shake by hand several times during a period of 30 min. or for 5 min. in a mechanical shaker. Filter part of the suspension and determine the calcium plus magnesium concentration of a suitable volume of the clear filtrate using Method 7.

¹⁵ SCHOONOVER, W. R. EXAMINATION OF SOILS FOR ALKALI. University of California Extension Service, Berkeley, California. 1952. (Mimeographed.)

Calculations

Gypsum requirement, meq./100 gm. = (Ca conc. of added gypsum solution in meq./l. - Ca + Mg conc. of filtrate in meq./l.) \times 2.

(23) Alkaline-Earth Carbonates (Lime)**(23a) Alkaline-Earth Carbonates by Effervescence With Acid****Reagent**

A. Hydrochloric acid, 3N (1 + 3).

Procedure

Place several grams of soil on a small watchglass. By means of a pipet add sufficient water to saturate the soil. This displaces most of the soil air so that its loss upon the addition of acid will not be confused with effervescence of lime. Add a few drops of reagent A to the soil and note any effervescence that occurs. The soil may be termed slightly, moderately, or highly calcareous in accordance with the degree of effervescence obtained.

(23b) Alkaline-Earth Carbonates by Gravimetric Loss of Carbon Dioxide**Reagent**

A. Hydrochloric acid, 3 N (1 + 3).

Procedure

Pipet 10 ml. of reagent A into a 50-ml. Erlenmeyer flask, stopper with a cork, and weigh. Transfer a 1- to 10-gm. sample of soil containing 0.1 to 0.3 gm. of calcium carbonate to the flask, a little at a time, so as to prevent excessive frothing. After effervescence has largely subsided, replace the stopper loosely and swirl the flask. Let stand with occasional swirling until the weight of the flask and contents does not change more than 2 or 3 mg. during a 30-min. period. The reaction is usually complete within 2 hours. Prior to weighing, displace any accumulated carbon dioxide gas in the flask with air. This is important and may be done by swirling with the stopper removed for 10 to 20 sec.

Calculations

Weight of CO₂ lost = (initial wt. of flask + acid + soil) - (final wt. of flask + acid + soil).

CaCO₃ equivalent in percent = (wt. of CO₂ lost \times 227.4) / wt. of soil sample.

Remarks

The accuracy of this method depends to a large extent upon the sensitivity of the balance used for weighing. Using a torsion-type balance capable of detecting weight differences of 2 to 3 mg., the relative error is about \pm 10 percent.

(23c) Alkaline-Earth Carbonates From Acid Neutralization**Reagents**

- A. Hydrochloric acid, 0.5 N, standardized.
- B. Sodium hydroxide, 0.25 N, standardized.
- C. Phenolphthalein, 1 percent in 60 percent ethanol.

Procedure

Place 5 to 25 gm. of soil in a 150-ml. beaker, add 50 ml. of reagent A by means of a pipet, cover with a watchglass, and boil gently for 5 min. Cool, filter, and wash all the acid from the soil with water. Determine the amount of unused acid by adding 2 drops of C and back-titrating with B.

Calculations

CaCO₃ equivalent in percent = (meq. HCl added - meq. NaOH used) \times S/weight of sample in gm.

Remarks

The calculation gives the CaCO₃ equivalent. This is the amount of CaCO₃ required to react with the acid. This value usually is somewhat high, because soil constituents other than lime may react with the acid.

(24) Organic Matter**Apparatus**

Erlenmeyer flasks, 500-ml., thermometer, 200° C.

Reagents

- A. Potassium dichromate, 1 N. Dissolve 49.04 gm. of potassium dichromate in water and dilute to 1 liter.
- B. Sulfuric acid, conc., containing silver sulfate. Dissolve 25 gm. silver sulfate in a liter of acid.
- C. Ferrous indicator (ortho-phenanthroline ferrous sulfate, 0.025 M). Dissolve 14.85 gm. o-phenanthroline monohydrate and 6.95 gm. ferrous sulfate in water and dilute to 1 liter.
- D. Ferrous sulfate, 0.5 N. Dissolve 140 gm. of FeSO₄·7H₂O in water, add 15 ml. of conc. sulfuric acid, cool, and dilute to 1 liter. Standardize this solution daily against 10 ml. of reagent A, as directed in the procedure below.

Procedure

Grind the soil to pass 0.5-mm. screen, avoiding contact with iron or steel. Transfer a weighed sample, not exceeding 1.0 gm. and containing from 10 to 25 mg. of organic carbon, to a 500-ml. Erlenmeyer flask. Add 10 ml. of reagent A followed by 20 ml. of B. Swirl the flask, insert thermometer, and heat gently so as to attain a temperature of 150° C. in a heating period of about 1 min. Keep contents of flask in motion in order

to prevent local overheating, which results in error caused by thermal decomposition of dichromate. After the 150° temperature is reached, place the flask on an asbestos pad, and allow to cool. Add 200 ml. of water and 4 or 5 drops of C. Titrate with D until the color changes from green to red.

Since some soils adsorb o-phenanthroline indicator, the titration may be improved by a prior filtration, using a rapid filter paper on a Buechner funnel. If more than 80 percent of the dichromate solution is reduced, the determination should be repeated with less soil.

Calculations

Organic carbon in percent = (meq. of $K_2Cr_2O_7$ added - meq. of $FeSO_4$ used) $\times 0.336$ / wt. of sample in gm.

Organic matter in percent = organic carbon in percent $\times 1.72$.

Remarks

This modification of Walkley's rapid method (1935, 1947) for the determination of organic carbon in soils has been found to give approximately 89 percent recovery of carbon, as compared to the dry-combustion method. The conversion factor 0.336 was obtained by dividing 0.003, the milliequivalent weight of carbon, by 89 and multiplying by 100 to convert to percent. Chloride interference is eliminated by the addition of the silver sulfate to the digesting acid as indicated. Nitrates up to 5 percent and carbonates up to 50 percent do not interfere.

References

Walkley (1935, 1947).

(25) Total and External Ethylene Glycol Retention

Apparatus

Vacuum pump, Central Scientific Company Hyvac or equivalent.

Vacuum desiccators, inside diameter 250 mm., with external sleeve or glass stopcock and porcelain plates.

Muffle furnace with automatic temperature control.

Aluminum moisture boxes, 2½ in. in diameter and ¾ in. high, with lids.

Reagents

A. Hydrogen peroxide, 10 percent solution.

B. Anhydrous calcium chloride, 8 or 12 mesh, technical.

C. Phosphorus pentoxide.

D. Ethylene glycol (Eastman). Redistill under reduced pressure, discarding the first and last 10 percent of the distillate.

Procedure

Soil preparation. Grind the soil sample to pass a 60-mesh sieve. The increase in surface area brought about by this degree of grinding is negligible. Treat approximately 10 gm. of the sieved soil with reagent A for the removal of organic matter (see Method 41). Transfer the treated soil to a 5- to 8-cm. diameter Buechner funnel fitted with filter paper and leach with several small portions of distilled water, using suction. Allow the soil to air-dry, then pass through a 60-mesh sieve.

Total ethylene glycol retention. Weigh 2.10 gm. of the 60-mesh soil into an aluminum moisture box. The tare weight of the box and its lid should be known. Spread the soil evenly over the bottom of the box. Place the box in vacuum desiccator over about 250 gm. of reagent C, apply vacuum by means of a Hyvac or equivalent pump, and dry the soil to constant weight. This usually requires 5 to 6 hours. Determine the weight of vacuum-dried soil. By means of a pipet, having a tip drawn to a fine point, distribute 1 ml. of D dropwise over the soil surface. Place the box in a second vacuum desiccator over 250 gm. of B and allow to stand overnight to obtain uniform wetting of the soil. Connect the desiccator to a Hyvac pump and evacuate at a temperature of $25 \pm 2^\circ$ C. until excess ethylene glycol is removed from the soil. Depending upon the temperature and vacuum conditions attained, this usually requires from 5 to 7 hours when 8 samples are present in a desiccator. In practice, the box is weighed after 5 hours in the vacuum desiccator and at intervals of 1 hour thereafter until the loss of weight per hour interval is less than 3 or 4 percent of the weight of ethylene glycol remaining on the soil. The next to the last weight taken is used to calculate ethylene glycol retained.

External ethylene glycol retention. Weigh exactly 2.10 gm. of the 60-mesh soil into an aluminum moisture box. Spread the soil evenly over the bottom of the box and heat at a temperature of $600 \pm 15^\circ$ C. for 2 hours in a muffle furnace having automatic temperature control. Remove the box, cover, cool in a desiccator containing reagent B, and weigh. Apply 1 ml. of D to the soil, let stand overnight, and remove the excess ethylene glycol by evaporation in vacuum as described previously for the determination of total ethylene glycol retention.

Calculations

Assuming that 3.1×10^{-4} gm. of ethylene glycol are required for the formation of a monolayer on 1 sq. m. of surface, as indicated by Dyal and Hendricks (1950), the formulas for calculation of total, external, and internal surface areas are as follows:

Total surface area, $m.^2/gm.$ = wt. of ethylene glycol retained by unheated soil, $gm./$ (wt. of vacuum-dried unheated soil, $gm. \times 0.00031$).

External surface area, $m.^2/gm.$ = wt. of ethylene glycol retained by heated soil, $gm./$ (wt. of vacuum-dried unheated soil, $gm. \times 0.00031$).

Internal surface area = (total surface area) - (external surface area).

Remarks

The Hyvac pump and desiccators are connected by means of tight-fitting vacuum rubber tubing. A glass tube filled with reagent B is inserted in the vacuum line to prevent undesirable vapors from entering the pump. The tube also permits the introduction of dry air into the desiccators to release the vacuum. High-vacuum stopcock lubricant should be used to seal the glass joints.

The adequacy of the vacuum system for removing excess ethylene glycol can be checked by determining the rate of evaporation of this liquid from a free surface. The average rate of evaporation over a 5-hour period from an aluminum moisture box of the size specified above and containing ethylene glycol should be at least 1 gm. per hour.

For greatest accuracy in the determination of internal surface area, removal of excess ethylene glycol from heated and unheated soil should be performed concurrently. Four unheated and the corresponding 4 heated samples are ordinarily placed together in a desiccator. The occasional inclusion of a standard sample having a known retention value serves as a useful control on procedure.

The anhydrous calcium chloride placed in the desiccator to absorb ethylene glycol should be renewed after each set of 8 determinations. The phosphorus pentoxide used for drying under vacuum may be used until it absorbs sufficient water to develop a syrupy consistency.

References

Bower and Gschwend (1952), Dyal and Hendricks (1950, 1952).

Soil Water

(26) Soil-Moisture Content

Procedure

Transfer a representative subsample of the soil to a tared can with lid. For accuracy, it is desirable where possible to use at least a 25-gm. sample. Weigh, dry to constant weight at 105° C., and weigh again.

Calculations

Moisture content in percent, $P_w = (\text{loss in weight on drying}) \times 100 / (\text{weight of the oven-dry soil})$.

(27) Saturation Percentage

(27a) Saturation Percentage From Oven-Drying

Procedure

Transfer a portion of the saturated soil paste, prepared according to Method 2, to a tared soil can with lid. Determine the moisture content by Method 26.

Calculations

Saturation percentage (SP) = (loss in weight on drying) $\times 100 /$ (weight of the oven-dry soil).

(27b) Saturation Percentage From Volume of Water Added

Remarks

When the air-dry moisture content of the sample is known, as it usually is when exchangeable-cation analyses are made, the saturation percentage can be determined as follows:

Procedure

Transfer a known weight of air-dry soil to a mixing cup. Add distilled water from a buret or graduated cylinder with stirring until the soil is saturated as described in Method 2. Record the volume of water added.

Calculations

Weight of oven-dry soil = (weight of air-dry soil) $\times 100 / (100 + \text{air-dry moisture percentage})$.

Total water = (water added) + (water in air-dry soil) = (weight of water added) + (weight of air-dry soil) - (weight of oven-dry soil).

$SP = 100 \times (\text{total weight of water}) / (\text{weight of oven-dry soil})$.

(27c) Saturation Percentage From the Weight of a Known Volume of Paste

Remarks

By this method, the saturation percentage is calculated from the weight of a known volume of saturated soil paste. It is assumed that the soil particles have a density of 2.65 gm./cm.³, and that the liquid phase has a density of 1.00 gm./cm.³.

Apparatus

Balance, accurate to 0.1 gm.
A cup of known volume. This measurement can be combined with the soil-paste resistance measurement

using the same loading of the Bureau of Soils electrode cup.

Procedure

Determine the volume and weight of the cup. Fill the cup with saturated soil paste, jarring it during filling to exclude air, and strike off level with the top. Weigh and subtract the cup weight to get the net weight of the paste.

Calculations

$$SP = \frac{100 (2.65V - W)}{2.65(W - V)}$$

where SP = saturation percentage ; V = volume of saturated soil paste, in cm^3 , = a constant; and W = net weight of $V \text{ cm}^3$ of saturated soil paste, gm. Calculations are simplified by the use of a table or graph relating values of W and SP for a given value of V .

Reference

Wilcox (1951).

(28) Infiltration Rate

Remarks

Infiltration rate (infiltration capacity) is the rate of water entry into the soil where water covers the surface at a shallow depth and downward flow into and through the soil is nondivergent. The latter condition is satisfied by rainfall or if the ponded area is infinitely large. For practical purposes, the subsidence rate of the free-water surface in a large basin is taken as a measure of the infiltration rate. The effect of divergent flow increases as the ponded area decreases. If small basins or cylinders are used, it is difficult to determine the true infiltration rate. For soils in which permeability increases with depth, errors from flow divergence may be negligible; but, if the permeability decreases with depth, the effect of flow divergence may be considerable. Flow divergence that occurs with small plots or cylinders may be minimized by ponding water in a guard ring or border area around the plot or cylinder.

If infiltration measurements are made under conditions where divergent flow may not be negligible, the water-intake rate should be reported as infiltration velocity and accompanied by a description of the measuring method.

Under some conditions the evaporation rate may not be negligible and must be taken into account in infiltration measurements. In small basins or where cylinders are used, evaporation may be minimized by covering the water surface with a film of oil.

There is no single method best suited to all field conditions. Experience and judgment are required in obtaining and evaluating infiltration measurements. (See discussion in chapter 2.)

(28a) Basin

Apparatus

Gage for measuring water elevation, and watch.

Procedure

Pond water on an area of soil enclosed by dikes or ridges. Measure the rate of subsidence of the water surface with a staff gage (a linear scale standing in the water), hook gage, or water-stage recorder.

The infiltration rate will depend on the time and depth of water that has entered the soil.

Calculations

A curve showing the depth of water that has entered the soil as a function of time can be plotted from the water elevation -and time readings or taken from a water-stage recorder. Average or instantaneous values of the infiltration rate can be taken from this curve, depending on the purpose of the measurement. Express infiltration rate in centimeters per hour or in inches per hour.

(28b) Cylinder

Apparatus

Cylinders 11 to 14 in. in diameter and 16 in. long. The cylinders can be rolled from 16-gage sheet iron. Butt-weld and grind the weld smooth. Reinforce the upper end with $\frac{1}{8}$ -in. by 1-in. iron strip, welded to cylinder. Galvanize cylinders after fabrication. For ease in transportation, cylinders can be made with different diameters so that they will fit, one within another.

Circular driving cap and hammer. Torch-cut the driving cap from $\frac{1}{2}$ -in. steel plate and screw in a $\frac{1}{2}$ -in. central rod to serve as the hammer guide. The hammer can consist of a 50- to 80-lb. block of iron. This should have a central pipe to slide on the guide rod of the cap. Attach crosshandles to the pipe.

Hook gage or staff gage, watch, thin metal tamp, splash guard of rubber sheet or burlap, field source of water.

Procedure

Drive the cylinders into the soil to a depth of 6 or 8 in. Alternatively, the cylinders can be jacked into the soil, if a heavy tractor or truck is available. Care should be exercised to keep the sides of the cylinder vertical and to avoid disturbance of the soil column within the cylinder. Tamp soil into the space between the soil column and the cylinder. If this space is greater than $\frac{1}{8}$ in., the cylinder should be reset. Cover the soil with a splash guard and apply 4 to 6 in. of water. Record the elevation of the water surface and the time at convenient intervals. A staff gage is often satisfac-

tory, but a hook gage should be used if the subsidence rate is low. Several adjacent cylinders are usually installed. These need not be carefully leveled if a mark is placed on each cylinder for locating the base of the hook gage.

While the wetting front is in the cylinder, the water-subsidence rate corresponds to the infiltration rate. When the wetting front passes below the cylinder, more or less divergence of flow will occur and the subsidence rate then should be designated as intake rate or infiltration velocity. Divergent flow is minimized by installing cylinders in plots or within larger diameter rings in which the soil is kept flooded.

Where desired, water-entry rates into subsurface soil layers can be measured by excavating to the desired depth before setting the cylinders.

Calculations

Express infiltration rate and infiltration velocity in centimeters per hour or in inches per hour, using values averaged over time intervals appropriate to the purposes of the measurement.

(29) 1/10-Atmosphere Percentage

Apparatus

Pressure-plate apparatus. Retainer rings approximately 1 cm. high and 6 cm. in diameter to hold at least 25-gm. samples. Balance, drying oven, and moisture boxes.

Remarks

Install the pressure plates to be used for the test in a pressure cooker, fill the cooker with water, fasten the lid on the cooker, and measure the rate of outflow of water from the ceramic plates at a pressure of 15 lb. in.⁻². This rate should be about 1 cc. per cm.² per hr. per atm. pressure difference or greater for satisfactory operation of the porous plates. Next check the pressure plates for entry value as follows: release the air pressure, empty excess water from the cooker pot and the plates, close the cooker pot, and apply a pressure of 1/2 atm. or other appropriate value. After a few minutes, the outflow of water from the plate outlets will cease and there should be no bubbling of air from these outlets, thus indicating that the entry values for the plates are above the value of the pressure applied to the pressure cooker. At the conclusion of the entry-value test, submerge the pressure cooker in water while the pressure is on or make other equivalent tests to make sure that there are no air leaks at the cooker gasket or attendant connections. Air leaks from the cooker cause troubles with air-pressure control and may also cause serious errors in retentivity determinations through direct loss of water vapor from the soil samples.

Procedure

Prepare duplicate 25-gm. samples that have been passed through a 2-mm. round-hole sieve, using the subsampling procedure outlined in Method 1. Place the sample retainer rings on the porous plate. In order to avoid particle-size segregation, dump *all* of the soil sample from each container into a ring and level. Allow the samples to stand at least 16 hours with an excess of water on the plate. Close the pressure cooker and apply a pressure of 100 cm. of water. Samples 1 cm. high can be removed any time after 48 hours from initiating the extraction or when readings on a buret indicate that outflow has ceased from all of the samples on each plate. Some soils will approach equilibrium in 18 to 20 hours. Before releasing the air pressure in the pressure cooker, put a pinch clamp on the outflow tube for each plate. This prevents backflow of water to the samples after the pressure is released. To avoid changes in the moisture content of the samples, transfer the samples quickly to moisture boxes. Determine the moisture content by drying to constant weight at 105° C. Express the moisture content as percent, dry-weight basis.

References

Richards and Weaver (1944), and Richards (1949b).

(30) 1/3-Atmosphere Percentage

Apparatus

Same as in Method 29.

Procedure

Same as in Method 29, except that the extraction pressure is 345 cm. of water.

(31) 15-Atmosphere Percentage

Apparatus

Pressure-membrane apparatus with sausage-casing membrane. Rubber soil-retaining rings 1 cm. high and approximately 6 cm. in diameter that hold about 25 gm. of soil. Balance, drying oven, and moisture boxes.

Procedure

Prepare duplicate 25-gm. samples that have been passed through a 2-mm. round-hole sieve, using the subsampling procedure outlined in Method 1. Moisten the cellulose membrane, install in the apparatus, and trim the edge by running a knife around the brass cylinder. Place the soil-retaining rings on the membrane. In order to avoid particle-size segregation, dump *all* of the soil sample from each sample container into one

ring. Pouring out part of the sample and leaving part in the container will give a nonrepresentative sample. Level the sample in the ring, cover with a square of waxed paper, and allow the samples to stand at least 16 hours with an excess of water on the membrane. Remove excess water from the membrane with a pipet or rubber syringe, close the pressure-membrane apparatus and admit air to the soil chamber at a pressure of 15 atm. (220 lbs. in.⁻²).

After a few hours, there is a marked decrease in the rate of water outflow from the soil, the outflow rate then being limited mainly by the low capillary conductivity of the soil rather than the low membrane permeability. At this time, the soil samples have sufficient rigidity to resist plastic flow and compaction and so a 4 lb. in.⁻² pressure differential may be applied to the rubber diaphragm at the top of the soil chamber. This diaphragm action holds the sample firmly in contact with the membrane and considerably hastens moisture extraction for fine-textured soils that shrink appreciably. The diaphragm is unnecessary for medium- and coarse-textured soils.

Remove the samples any time after 48 hours from the commencement of the extraction or when the readings on an outflow buret indicate equilibrium has been attained. Most soils will approach hydraulic equilibrium with the membrane in 18 to 20 hours, but some soils may require a considerably longer time. In order to avoid changes in the moisture content, transfer the samples to moisture boxes as soon as possible after releasing the extraction pressure. Determine the moisture content by drying to constant weight at 105° C. Express moisture content as percent, dry-weight basis.

Remarks

Care must be taken to keep soil away from the lower gasket of the cell. Otherwise, gasket flow that occurs when the cell is closed may press sand particles into the membrane and cause leaks.

References

Richards (1947), Richards and Weaver (1944).

(32) Moisture-Retention Curve

Each sample, with disturbed or undisturbed structure, is contained in a retainer consisting of a brass cylinder, a plastic lid, and a porous ceramic bottom plate, all held together with rubber bands. Various moisture equilibria and weighings are thus made possible with a minimum of disturbance to the sample.

Apparatus

Pressure-plate apparatus, pressure-membrane apparatus, balance, drying oven, large straight-edged carving knife, and aluminum moisture boxes with lids, 3½ in. diameter by 2 in. high.

Soil sampling tube with retainer cylinders cut from brass tubing 2¼ in. outside diameter by 19-gage wall. The core retainer cylinders are 3 cm. high, and while in the sampling tube have guard rings 1 cm. high at each end. (See drawing of apparatus in the Appendix.)

Plastic and ceramic disks serve as lids and bottoms for the core retainer. The lids are cut from ⅛ in. transparent plastic sheet and are 2¼ in. in diameter. The ceramic disks are 2¼ in. in diameter by ⅜ in. thick, with a peripheral groove to attach two hooks formed from twisted wire at points on the disk 180° apart. The porous ceramic body should be like that used for tensiometer cups. The entry value should be greater than 1 atm., and the hydraulic conductivity should be equal to or greater than 8×10^{-4} cm./hr.

A layer of cheesecloth and sieved soil make capillary contact between the retainers and the control membranes. The cheesecloth should be treated with a bactericide such as Dovicide No. 4. The fraction of a loam soil that passes a 60-mesh screen makes a good capillary contact medium.

A complete core-retainer set consists of a moisture box with lid, a brass cylinder, a plastic lid, two strong rubber bands, and a ceramic plate. All of the parts in a retainer set should bear the same identifying number. The tare weight of each retainer set with the ceramic disk *saturated with water* should be determined and recorded.

Procedure

Take the cores with the sampling tube when the soil is moist. Remove the 1-cm. guard rings from either end of the 3-cm. retainer cylinder. Roughly trim the cores in the field and transport to the laboratory in the aluminum moisture boxes. Trim the cores accurately in the laboratory with the carving knife. Fasten the plastic lids and ceramic plates to the brass cylinders by stretching the rubber bands across the lids and attaching the bands to the hooks at the opposite edges of the ceramic plates.

Place the core retainers on a porous brick with a free water surface 1 or 2 mm. below the surface of the brick. After 24 hours, wipe the excess water from the retainers, place each in its moisture box, and weigh. Replace the retainers on the brick with the water surface set for 10 cm. After 24 hours, weigh again. These two weighings will not represent equilibrium values, but high precision is usually not required at 0 and 10 cm. of suction. Prepare the pressure-plate apparatus as indicated in Method 29. Spread approximately a 3-mm. layer of screened loam soil on the pressure plate and cover with a single layer of treated cheesecloth. Moisten the soil and cloth with water and set the retainers firmly in contact. Close the pressure cooker and adjust the pressure for the next suction value.

Follow the approach to hydraulic equilibrium at each pressure by connecting the outflow tube from each plate or membrane to the lower end of a buret and recording the buret readings occasionally. When

equilibrium is attained, clamp off the outflow tubes and release the air pressure in the cooker or membrane cell. Lift the core retainers from the membrane, brush off any adhering soil, place each in its numbered moisture box, and weigh. For a retention curve, weighings can be made at tensions of 0, 10, 30, 100, and 345 cm. of water and 1, 3, and 15 atm. Other suction values can be used, depending on the information desired.

The porous-ceramic retainers used at the Laboratory have an entry value of 1 atm. and do not change appreciably in moisture content at suction values up to 3 atm. Therefore, the gross tare for a core-retainer set is the same for all weighings at suction values up to and including 3 atm. At the 15-atmosphere equilibrium, the ceramic retainer is removed before the weighing, and a correspondingly different tare weight is used. Determine the weight of the soil core when oven-dried at 105° C.

Calculations

Determine the volume of the core retainer and calculate the bulk density of the soil in the core. From the gross weights at each suction, the tare weights, and the known weight of soil, calculate both the mass of water and the volume of water (numerically the same when c. g. s. units are used) in the core at each suction value. From the foregoing data, calculate the grams of water per 100 gm. of dry soil and the cubic centimeters of water per 100 cm.³ of soil at each suction value. The latter may be taken as the depth percentage, i. e., the depth of free water per 100 units of depth of soil. Plot these values on linear coordinates with moisture retention as the dependent variable, and suction or soil-moisture tension as the independent variable.

References

Richards (1947, 1948, 1949b, 1952).

(33) Field-Moisture Range

Remarks

Plants can grow in soil over a range of moisture contents referred to as the available range. The practical upper boundary for this range, sometimes referred to as field capacity, is characteristic of the field situation, and the best method for its determination is based on field sampling. The determination should be made after the soil has been wetted and the rate of downward drainage has decreased, but before appreciable moisture is lost from the profile by evaporation and root extraction. This determination loses significance or requires special interpretation if drainage is restricted or if a water table is close to the soil surface.

Apparatus

Soil tube or soil auger, watertight moisture boxes, balance, and drying oven.

Procedure

One to 3 days after the soil profile is thoroughly wetted with rain or irrigation water, take samples by horizons, by textural layers, or at 1-foot-depth intervals throughout the wetted zone. Determine the moisture content of the samples by drying to constant weight at 105° C. Express the results as moisture percentage, dry-weight basis, or as depth percentage if the bulk density can be determined. The available range for the soil at any given depth is then found by subtracting the 15-atm. percentage from the field determination of the upper limit of available water. The available range can be expressed either as a dry-weight percentage or as a depth percentage.

(34) Hydraulic Conductivity

(34a) Hydraulic Conductivity of Soil Cores

Thin-walled cylinders or cans may be pressed into the soil in the field to obtain samples of soil of substantially undisturbed structure. More often, soil cores are obtained in metal sleeves that fit into a sampling tube, and, after the samples have been taken, the sleeves serve as the core retainers. Power-driven machines are available for taking undisturbed cores of 4- and 6-in. diameter. Such cores are encased in the field for transportation and subsequent water-flow measurements. Various casing methods have been used, such as painting the core with wax or plastic cement before and after wrapping in cloth.

Procedure

In the laboratory, the cores are mounted vertically and supported on a porous outflow surface such as sand or filter paper and metal screen. A shallow depth of water is usually maintained over the soil surface by a siphon tube from a constant-level reservoir. Flow tests should be conducted with water of the same quality as that which occurs in the field. If discharge rates are low, care must be taken to avoid errors arising from evaporation of the percolate. If possible, flow tests should be conducted at or near constant temperature.

Where desirable, especially for long cores, manometers can be attached at various points along the core. These should be installed at transition zones between horizons or at textural discontinuities.

Calculations

Water flow takes place in accordance with the equation:

$$\frac{Q}{t} = k A \frac{\Delta H}{\Delta L}$$

where Q is the volume of water passing through the core in time (t), A is the area of the core, and k is the average hydraulic conductivity in the soil interval

(ΔL), over which there is a hydraulic head difference of ΔH . Solving for k gives $k=Q\Delta L/At\Delta H$. Hydraulic conductivity (k) will be in centimeters per hour if t is expressed in hours, Q in cm^3 , A in cm^2 , and ΔH and ΔL are both in the same units.

References

Bower and Peterson (1950), Kelley and coworkers (1948), Marsh and Swarner (1949), and Richards (1952).

(34b) Hydraulic Conductivity of Disturbed Soil

Apparatus

Soil containers are made from 20-gage seamless brass tubing, 3 in. outside diameter, and 4 in. in length. The bottoms of the containers are machined from 20-gage brass sheet and are soldered into a recess or counterbore in the cylinders. The central outflow tubes are 2 in. long, are cut from $\frac{1}{2}$ in. outside diameter by 20-gage brass tubing, and are attached with solder.

Supports for soil and filter paper consist of circles of 20-mesh or coarser bronze screen cut so as to fit loosely on the inside of the soil container.

Packing block is made from a heavy wooden block approximately 4 by 4 by 8 in. A hole is made in the block to accommodate the outflow tube of the soil container, and guide rods are mounted in the block to keep the cylinder vertical and to insure square impacts. One rod is cut 2.5 cm. above the cylinder so that a finger placed over this rod gives a convenient index of height for the packing process.

Sharkskin filter paper, rack for supporting a number of soil containers, constant-level water supply, siphon tubes to connect soil containers to water supply, graduated cylinders, 2-mm. round-hole sieve, soil grinder, and mixing cloth.

Procedure

Air-dry the soil and pass it through a 2-mm. round-hole screen. A power grinder may be used for hard soils, but the grinding process must be standardized, with the plates set to reduce only the larger particles. Obtain representative 200-gm. subsamples in accordance with Method 1. Dump the entire subsample in one motion into the soil container that has been fitted with a screen and filter paper. This method of transferring the soil is used to prevent particle-size segregation. The cylinder containing the soil is dropped 20 times through a distance of 2.5 cm. onto the packing block. Place a filter paper on the soil surface and introduce water into the container with a minimum of soil disturbance. Record the time of application of water and, if possible, the time of the initial outflow. Collect the percolate in a suitable receptacle and measure the volume at convenient time intervals. Tests ordinarily are run until the volume of water that has passed

through the soil corresponds to approximately 12 cm. of depth of water on the soil surface. Calculate hydraulic conductivity and plot against accumulated equivalent depth of percolate. With soils having extremely low percolation rates, an attempt should be made to obtain at least one flow measurement, and time rather than depth of water is used to determine when to discontinue tests on such soils.

Remarks

While, according to theory, neither the diameter nor the height of the soil column to be tested needs to be within prescribed limits, it has been found that with many soils satisfactory results are not obtained unless the height is less than the diameter of the soil column. This is particularly important if the soil swells appreciably on wetting. Experience indicates that the cylinder should have at least a 7.5-cm. diameter for a 5-cm. depth of soil.

Hydraulic-conductivity measurements should be made in the temperature range from 65° to 75° F. (18° to 24° C.) . For the most part, the effect of temperature on hydraulic conductivity in this range is small compared with effects arising from such factors as quality of the water and the base status and salinity of the soil. The standard temperature for laboratory determination of hydraulic conductivity is usually taken as 68° F. (20° C.) . Corrections for viscosity effects on measurements at temperatures other than 68° F. can readily be calculated, but it has been observed that temperature has other and not always predictable effects upon the hydraulic conductivity of soils in addition to those arising from viscosity.

The hydraulic gradient is usually set in the range from 1 to 4, although values as high as 10 do not seem to affect the results significantly.

In general the water that will be used on the soil in the field should be used for the laboratory determinations, because small changes in water quality can produce large changes in rate of moisture movement.

Measurements are usually made in triplicate. The samples are discarded and the test repeated if the range of values is greater than 50 percent of the mean hydraulic-conductivity value. Between soils or treatments, average differences in conductivity of less than 15 or 20 percent are not considered significant.

Calculations

Water flow takes place in accordance with the equation :

$$\frac{Q}{t} = kA \frac{\Delta H}{\Delta L}$$

where Q is the volume of water passing through the material in time (t) ; A is the area of the soil column, and k is the average hydraulic conductivity in the soil interval (ΔL) over which there is a hydraulic-head difference (ΔH) . Solving for hydraulic conductivity:

$$k = \frac{Q\Delta L}{tA\Delta H}$$

It should be noted that AH must be measured from the surface of water standing on the soil to the elevation at which water will stand during the flow test in a riser or manometer connected at the bottom of the soil column. For experimental setups sometimes used, this elevation may be quite different from the elevation of the bottom of the soil column. The length of the soil column ΔL should be measured during or after water flow and not when the soil is dry.

References

Christiansen (1947), Fireman (1944), and Richards (1952).

(34c) Hydraulic Conductivity From Piezometer Measurements

Equipment

The piezometer pipe may be of any convenient diameter. The length will be governed by the depth at which measurements are to be made. The wall thickness should be as thin as practical to minimize soil disturbance during installation. Thin-walled electrical conduit, 1 to 2 in. inside diameter, has been found suitable for hydraulic-conductivity measurements at depths up to 10 ft. Other pieces of equipment needed for this measurement are: a screw-type soil auger having a free-fit inside the piezometer pipe; a hammer, such as is used for soil tubes or for steel fence posts, may be used for driving the pipe; a pump, such as a hand-operated pitcher pump, with a flexible hose attached to the inlet is needed to remove water and sediment from the pipe and the soil cavity; an electrical sounder is convenient for measuring the depth to the water surface within the pipe (see Method 35a); an ordinary watch is satisfactory for measuring time, except, if the rate of rise is rapid, two stop watches may be required to obtain a continuous rate-of-rise record; a soil-tube jack or other tube puller is useful in recovering the piezometer pipe.

Remarks

Hydraulic-conductivity measurements by this method are limited to soils below a water table. An auger hole is cased with a length of pipe and a cylindrical cavity is formed at the lower end of the pipe. Ground water flows into the cavity when water is pumped from the pipe, and the rate at which the water level rises in the pipe is a measure of hydraulic conductivity. Although the development of the equation is based upon an idealized condition of homogenous isotropic soil, this method may be used for determining the hydraulic conductivity of nonuniform soils and of individual soil layers. Information regarding water-table level and nature and position of subsoil layers should be available prior to installation of piezometer pipes to assist in determining proper placement of pipes and construction of cavities. For most purposes, the extremities

of the cavity should not be closer than one cavity length from either the top or bottom of the particular soil layer for which the determination is made.

This method is applicable only where a cavity of known shape can be maintained throughout the test. In many fine-textured soils, cavities will stand without support, but in sands and other noncohesive materials a supporting porous structure may be required.

Procedure

Remove grass sod or debris from the soil surface and install the pipe to any desired depth by alternately augering and driving. Auger to a depth of 6 to 12 in. below the end of the pipe from within the pipe, then drive or push the pipe to the bottom of the drilled hole. This is done to minimize soil disturbance as the pipe is driven. When the pipe has been installed to the desired depth, auger out a cavity below the pipe. Cavity lengths of 4 to 8 in. have been found convenient, with pipes 1 and 2 in. in diameter. The length of cavity can be accurately controlled by use of a screw clamp on the auger handle. The cavity should be formed with a minimum of disturbance to the surrounding soil.

Remove seepage water and sediment from the cavity by pumping several times. Measure the depth to the water in the pipe after allowing enough time for the water to rise in the pipe to the equilibrium level. In highly permeable soils the equilibrium level may be attained in a few minutes; in some fine-textured soils several days may be required. Pump the water from the pipe and measure the rate at which ground water rises in the pipe. The rate of rise should be measured as soon as practicable after pumping, since, it is assumed, in the development of the theory, that the draw-down of the water table is negligible. Rate of rise may be measured at any point between the water-table level and the lower end of the pipe, but measurements near the equilibrium level should be avoided. The rise increment should be selected to give convenient and measurable time intervals.

If hydraulic-conductivity determinations are desired at several depths, measurements can be made with the same pipe by successively augering to a greater depth following each determination.

Calculations

Hydraulic conductivity k calculated by use of the equation given by Kirkham (1946) as follows:

$$k = \frac{2.30 \pi R^2}{A (t_2 - t_1)} \log_{10} (h_1/h_2)$$

where k is hydraulic conductivity; R is the radius of tube; A is a geometrical factor (the A-function), which may be read from figure 28; h_1 is the distance from the water table to the water level in pipe at time t_1 ; h_2 is the distance from the water table to the water level in pipe at time t_2 ; $t_2 - t_1$ is the time interval for water to rise from h_1 to h_2 . Hydraulic conductivity (k) will be

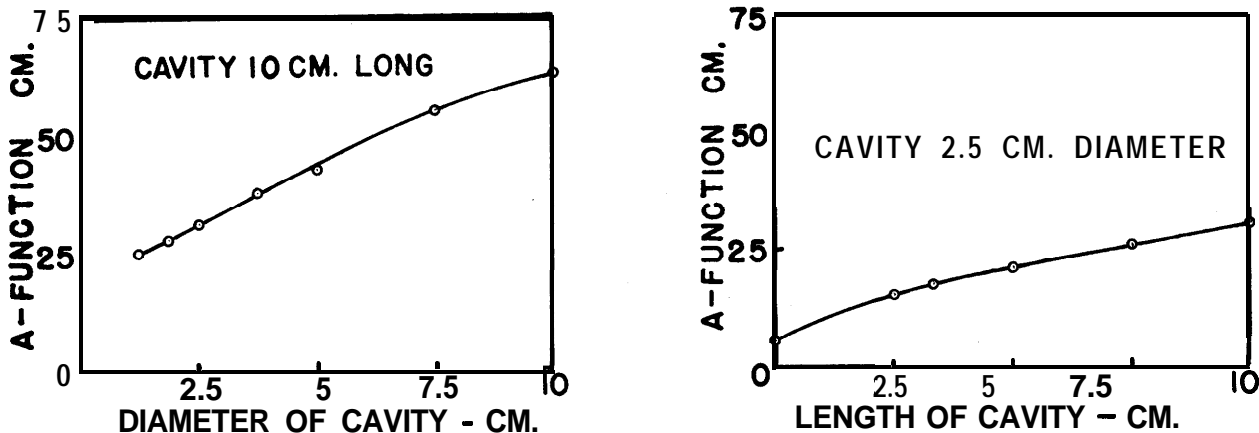


FIGURE 28.-Relation of the A-function to the length and diameter of the cavity for the piezometer method of measuring hydraulic conductivity. (Redrawn from Luthin and Kirkham, 1949.)

in centimeters per hour if R and A are in centimeters; h_1 and h_2 are both in the same units, and $(t_2 - t_1)$ in hours. However, any consistent system of units may be used. For values of the A-function not shown in the illustrations see Luthin and Kirkham (1949).

The hydraulic conductivity can also be calculated from an approximate equation that eliminates the use of logarithms. The constant inflow-rate equation of Kirkham (1946), slightly modified, is as follows:

$$k = \frac{\pi R^2}{A (t_2 - t_1)} \frac{\Delta h}{h_{av}}$$

where Δh is the increment of rise of the water level in the pipe in time $t_2 - t_1$; h_{av} is the average head, i. e., $h_{av} = (h_1 + h_2)/2$; and the other terms are as previously defined.

This approximate equation is sufficiently accurate for the usual values of Δh and h_{av} and may be used to simplify calculations. The error introduced by using this equation is small if the ratio $\Delta h/h_1$ is small, but increases as the ratio increases. The error in k is less than 4 percent for ratios of $\Delta h/h_1 < 0.5$ and less than 10 percent for ratios as large as 0.7.

References

Johnson, Frevert, and Evans (1952), Kirkham (1946), Luthin and Kirkham (1949), and Reeve and Kirkham (1951).

(34d) Hydraulic Conductivity From Auger-Hole Measurements

Equipment

Soil auger; any convenient size may be used, but it should permit making a hole below the water table with a minimum of soil disturbance. Water-level sounder; an electrical sounder mounted on a frame or tripod is convenient for measuring depth to water in an auger hole. In large-diameter auger holes, water levels can

be measured with a rule or tape. A rule attached to a float provides a convenient means for measuring the rate of rise of water in an auger hole.

A hand-operated pitcher pump with a flexible hose attached to the inlet may be used to pump water and sediment from the auger hole. In addition, a stop watch is needed for time measurements.

Remarks

This method is limited to measurements in the soil profile below a water table and is applicable only where a cavity of known shape can be maintained throughout the test.

Procedure

Drill an auger hole to the desired depth below a water table with as little disturbance to the soil as possible. Insert the pump intake hose to the bottom of the auger hole and empty the cavity several times. This is done to remove suspended sediment and to reopen soil pores in the wall that may have been altered by the auger. Measure the depth to water in the hole when equilibrium with the surrounding ground water is attained. In highly permeable soils the equilibrium level may be reached in a few minutes; whereas, in some clays several days may be required. Pump the water from the hole and measure the rate at which the water rises in the hole while the water level is near the bottom of the auger hole or at the time that the auger hole is half full. The rate of rise should be determined as soon as practical after the water level is pumped down, since it is assumed in the development of the theory that the drawdown of the water table is negligible. A small rise increment should be used since the A-function varies as the hole fills up. The formula given below involves this assumption.

Calculations

Hydraulic conductivity is calculated by use of the

equation given by Van Bavel and Kirkham (1949) as follows :

$$k = \frac{\pi a^2}{A (d-h)} \frac{\Delta h}{\Delta t}$$

where k is the hydraulic conductivity; a is the radius of the auger hole; A is the A-function, a geometrical factor which may be read from figure 29, for the case where the auger hole is empty and where it is half full; d is the depth of auger hole below the water table; h is the depth of water in the auger hole; Δh is the

increment of rise of the water level in 'the hole in the time interval Δt .

Hydraulic conductivity (k) will be in centimeters per hour if a , d , h , and A are in centimeters and t is in hours. However, any consistent system of units may be used.

In selecting values of the A-function (fig. 29), information on the depth (s) to an impermeable layer below the bottom of the auger hole is required. When an impermeable layer occurs at a depth in the range from $s=0$ to $s=d$, the A-function should be selected

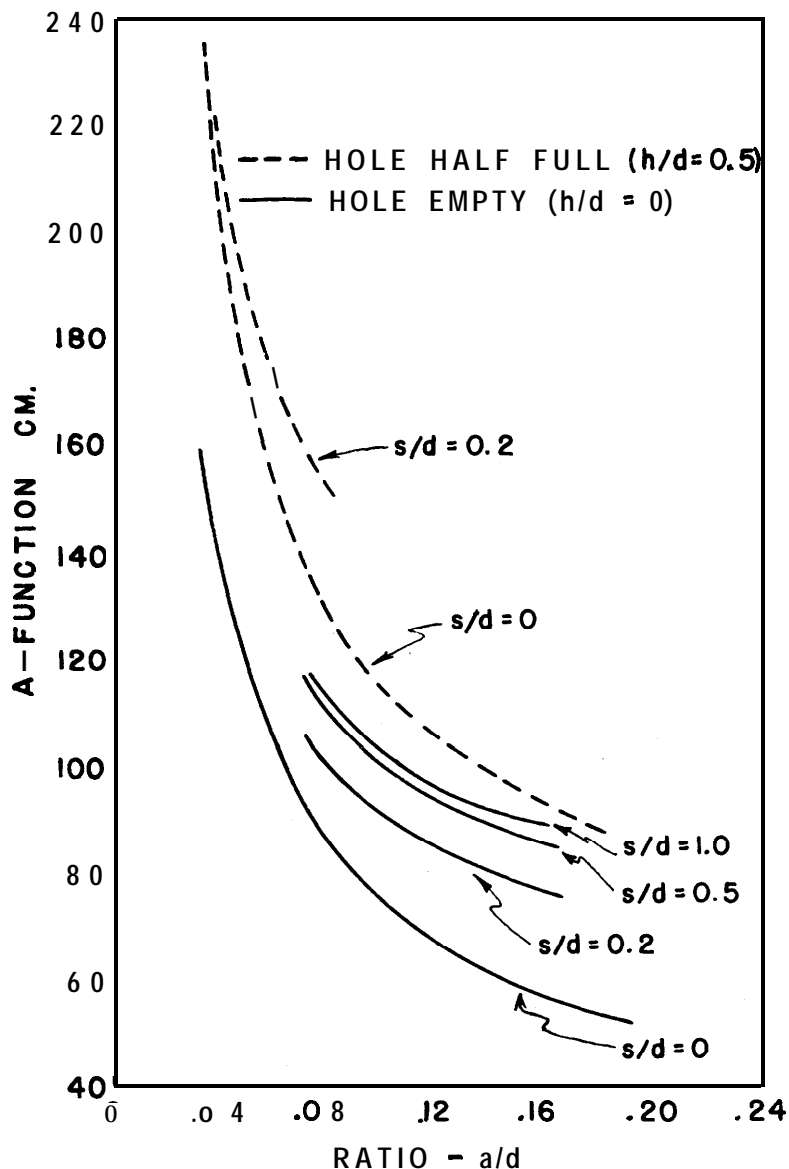


FIGURE 29.—Relation of the value of the A-function to the ratio a/d , for the auger-hole method of measuring hydraulic conductivity: a , radius of the hole; d , depth of the hole below the water table; h , depth of water in the hole; s , depth of an impervious layer below the bottom of the hole. The A values in the figure are for auger holes having a radius equal to 5 cm. For any other radius (x), multiply the A value read from the figure $x/5$. (Redrawn from Van Bavel and Kirkham, 1949.)

from the curve for the nearest value of s/d . When $s/d > 1$, use the curve for $s/d = 1$. The depth(s) to an impermeable layer is not a critical factor for the usual values of u/d . See Van Bavel and Kirkham (1949) for discussion of errors involved when, without knowledge of the depth to an impermeable layer, arbitrary values of s/d are used.

References

Diserens (1934), Hooghoudt (1936), Johnson, Frevert, and Evans (1952), Kirkham and Van Bavel (1949), Reeve and Kirkham (1951), and Van Bavel and Kirkham (1949).

(35) Hydraulic-Head Measurements in Saturated Soil

(35a) Piezometers Installed by Driving

Equipment

Iron pipe, $\frac{3}{8}$ in., galvanized or black, cut in 7, 10.5, 14 ft., or other lengths as desired; hand-operated driving hammer (Christiansen, 1943) or pneumatic driving hammer (Donnan and Christiansen, 1944); rivets, structural iron, $\frac{3}{8}$ in. in diameter by 1 in.; rivet punch-out rods (several lengths of $\frac{1}{8}$ in. in diameter iron pipe with male and female flush connections, or other rods to fit); 25 to 50 ft. of semirigid plastic tubing, $\frac{5}{16}$ in. in diameter;¹⁶ hand-operated bucket pump; 5-gal. water bucket; and carpenter's level.

After installation it is necessary to measure the depth to water in the piezometer. This can be done with a steel tape or other sounding device. Tapes with a dark oxidized surface show the water-level mark readily, or chalk can be used to make the water mark more visible.

If many readings are to be made, it is worth while to construct an electrical water-level sounder. For this, a length of flexible insulated wire is wound on a reel that has a socket for mounting on the top of the piezometer pipe. A straight segment of the wire slightly longer than 1 ft. should be exposed to view between the reel and the top of the pipe. The lower end of the wire is weighted with metal tubing to keep the wire taut, and the upper end is grounded to the reel and pipe through a battery and high-resistance voltmeter. The insulated wire is marked at 1-ft. intervals. Fractions of a foot can be read to the nearest 0.01 ft. from a scale attached to the reel mount. Readings are taken on the first mark on the wire appearing above the top of the pipe when the voltmeter indicates the lower end of the wire is at the water surface.

Procedure

With an iron rivet in lower end of pipe, drive first length of pipe into soil. Additional lengths can be added with standard pipe couplings as driving pro-

¹⁶ Saran tubing, manufactured by the Dow Chemical Co., is suitable for this use and is often locally available at hardware stores.

gresses until pipe reaches the desired depth. Leave pipe extending approximately 1 ft. above ground surface. If hydraulic-head readings are desired at several depths at a given location, drive pipes of different lengths into the soil, spacing the pipes laterally with a separation of about 1 ft. Use the carpenter's level to set the tops of all pipes to the same elevation. This makes it convenient to record and interpret hydraulic-head readings. Pipe lengths up to 16 ft. long can be installed by driving if a stepladder is used. In some soils, the pipe can be pushed into the ground 5 or 6 ft. before driving is required so that 21-ft. lengths can sometimes be used. Insert punch-out rod in pipe and punch rivet a distance of 3 to 6 in. out of the end of the pipe. Push the plastic tubing, previously marked with paint or tape to indicate the pipe length, to the bottom of the pipe and by pumping water through the tube with the hand pump, flush out a cavity 3 to 6 in. long below the end of the pipe. Soil material and water will return to the surface in the annular space between the tubing and pipe. After the cavity at the base of the pipe is formed, test the piezometer for response rate by filling with water and observing the rate at which the water level drops. If the rate of change in the level of the water in the pipe is very low, repeat the flushing operation. In sands and gravels, the rate of drop may be so rapid that no overflow can be obtained during flushing; whereas, in clays the rate of drop may be so slow that it is hardly noticeable. In any event, the flushing should be repeated without unduly extending the plastic tube below the end of the pipe until the rate of change of the water level in the pipe after filling is perceptible. The level of the water in the piezometer should then be allowed to come to equilibrium with the ground water. It is important to make this test of the responsiveness of each piezometer because the reliability of readings depends directly upon the readiness with which the water level in the pipe responds to hydraulic-head changes in the ground water at the bottom of the pipe. Piezometers should be retested for responsiveness periodically and reflushed, if necessary.

In some soils, the rivet in the end may not be necessary. When the piezometer is driven, a soil plug from 3 to 12 in. in length may form in the lower end of the pipe, which can be removed by the flushing operation. In many soils, this soil plug can be flushed out in much less time than is required to punch out the rivet.

After the piezometers have been installed, flushed, and allowed to come to equilibrium with the ground water, the depth to water surface from the top of the pipe is measured and recorded.

Remarks

The hydraulic head of ground water at any given point, i. e., at the bottom of the pipe, is the equilibrium elevation of the surface of the water in the piezometer. This elevation can be referenced to any standard datum.

All hydraulic-head readings in a single ground-water system or locality should be referenced to the same

datum, mean sea level being commonly used. Water elevations at each site can be recorded as read from the top of the pipe. The elevations of the top of the pipe and the adjacent soil surface are determined by standard surveying methods.

The hydraulic gradient is the change in hydraulic head per unit distance in the direction of the maximum rate of decrease in head. The vertical component of the hydraulic gradient at a site where piezometers have been installed at several depths is equal to the difference in the equilibrium elevation of the water surface in two pipes divided by the difference in the elevation of the cavities at the bottoms of the pipes. This is an average value for the vertical component of the hydraulic gradient in the depth interval. (See Method 36 for graphical procedures that are useful in the interpretation of hydraulic-head readings.)

References

Christiansen (1943), Donnan and Christiansen (1944), and Richards (1952).

(35b) Piezometers Installed by Jetting

Equipment

Iron pipe, $\frac{3}{8}$ -in. in diameter, galvanized or black, 10.5-ft. lengths, threaded at both ends; power-driven pump, 300 to 600 lb./in.², 10 to 15 gal./min. capacity with a water tank of 300-gal. capacity, truck- or trailer-mounted (an auxiliary 300-gal. water tank, truck-mounted, is also desirable); 25 to 50 ft. high-pressure hose, $\frac{3}{4}$ -in. in diameter, with a swivel coupling for attachment to the pipe; driller's mud; and steel measuring tape or electrical sounder.

Procedure

The installation of piezometers by the jetting technique makes use of the eroding and lubricating properties of a stream of water issuing from the end of the pipe for opening a passage into the soil. Piezometers may be installed by hand or with simple hoisting and handling equipment, such as has been used in Coachella Valley, California, and described by Reger and associates (1950). During installation, the pipe is oscillated up and down from 1 to 2 ft. to facilitate the jetting. Water and soil material in suspension return to the surface around the outside of the pipe. The return flow acts as a lubricant for the upward and downward movement of the pipe and serves as a means for logging materials penetrated. An adjustable measuring tape used with the Coachella jetting rig serves to indicate depth of penetration to ± 0.1 ft. If the jetting is done without a rig, the pipe should be marked at 1-ft. intervals to facilitate logging.

An estimate of texture and consolidation of the material is made from (a) the nature of the vibrations in the pipe that are transmitted to the hands of the operator, (b) the rate of downward progress, (c) examination of sediments carried by the effluent, and (d)

observation of color changes of the effluent. Logging subsurface layers by this method requires experience that can be gained and checked by jetting in profiles for which data on stratigraphy are available from independent logging procedures.

Return flow may be lost and penetration may stop in permeable sands and gravels. A commercial preparation, **Aquagel**, a form of driller's mud, was found by Reger and associates (1950) to be effective for maintaining return flow in coarse materials. Approximately 10 lb. per 100 gal. of water was sufficient for jetting conditions encountered in the Coachella Valley. It is necessary to add this preparation to the water supply slowly and to agitate thoroughly as it is added.

A record of the depth and nature of material penetrated is kept as the jetting progresses. Where several hydraulic-head measurements are desired at different depths, the deepest pipe is usually installed first. The log from the first pipe serves for selecting depths at which additional pipes are to be installed. It is often desirable to terminate piezometers in sandy lenses to increase the rate at which they respond to hydraulic-head changes in the soil. Jetting is stopped immediately as each pipe reaches the desired depth, so that excessive washing of material from around the pipe will not occur. The material in suspension settles back around the pipe and usually provides a satisfactory seal. Several pipes that terminate in the soil at different depths may be installed as close as 1 ft. apart. Experience has shown that, under most conditions, the effect of leakage along the pipe or from one pipe to another is negligible.

After the piezometers are installed, they are flushed, reference elevations are set, and readings are made as outlined in Method 35a. (For details of jetting-equipment construction, refer to the article by Reger and others (1950).)

References

Pillsbury and Christiansen (1947), Reeve and Jensen (1949), and Reger and others (1950).

(35c) Observation Wells, Uncased or With Perforated Casing

Equipment

Soil auger; perforated tubing or pipe; steel tape, or electrical sounder.

Procedure

It is desired to measure depth to water table. An uncased auger hole can often be used to measure depth to water table. Where soils are sandy and will not stand or where a more permanent well is desired, an auger hole may be cased with perforated casing. Sometimes it is necessary to install the casing during the augering process.

Water-table observation wells are usually installed to a depth great enough to reach the minimum ex-

pected position of the water table. As a result, observation wells are sometimes installed to considerable depths and perforated throughout a portion or all of the underwater length. Under many conditions, water-level readings in such wells coincide with the water-table level, but if there is a vertical flow component of water in the soil, either upward or downward, water-level readings in an open or perforated casing well may not represent the true water-table level. Where vertical-flow conditions occur, the water level in a perforated cased well represents a steady-state flow condition within the well itself, and may not give useful information. Such a condition is more likely to occur where an observation well penetrates layers that differ greatly in permeability. Where such conditions occur or where there is any question about water-table readings, hydraulic-head determinations should be made at several depths in the profile by the use of piezometers.

The elevation of the water table can be determined by a graphical method as follows. Plot the elevation of the terminal points of the piezometers in the soil as a function of the corresponding pressure heads, i. e., the lengths of the columns of water standing in the piezometers. Extrapolate this curve to zero pressure-head to obtain the water-table elevation. Abrupt changes in soil permeability with depth in the vicinity of the water table complicate the use of this method and make it necessary to install piezometers at or near the water table.

(36) Ground-Water Graphical Methods

(36a) Water-Table Contour Maps

Equipment

Drafting instruments and supplies

Procedure

On a scale map of the area being investigated, write in the water-table elevations at locations at which water-table level measurements have been made. By standard mapping procedures used for ground-surface contour plotting, i. e., interpolation and extrapolation, draw in lines of equal water-table elevations. The principles that apply in surface contour mapping also apply for water-table contours. Where slopes change abruptly, more points are required to locate the contours accurately. Conversely, in areas of little change in slope, measurement points may be farther apart. In areas of rolling or varied topography where water tables in general follow surface slopes, the number of data required to construct water-table contours may be prohibitive. A water-table contour line is the locus of points on the water-table surface for which the hydraulic head is constant. In a three-dimensional flow system, such a line represents the intersection of an equal hydraulic-head surface with the surface of the water table.

Water-table contour maps provide direct visual information on the slope of the water table, and it is to be expected that generally there is a horizontal movement of ground water in the direction of slope of the water table. In the absence of subsurface artesian conditions and if the areal application of water to the soil surface is uniform, a region of steep slope of the water table would be expected to occur where barriers to the horizontal movement of ground water occur or where the hydraulic conductance of the soil strata below the water table is low. On the other hand, areas of low slope in the ground-water table may indicate the presence of aquifers that permit the ready transfer of ground water in the horizontal direction. Such information is pertinent to the analysis and solution of drainage problems.

(36b) Water-Table Isobath Maps

Equipment

Drafting instruments and supplies.

Procedure

On a scale map of the area, write in depths to water table from the ground surface at locations at which water-table and ground-surface elevations have been obtained. Construct isobath lines, i. e., lines of equal depth to water table, by the standard mapping procedures that are used for ground-surface and water-table contour mapping, i. e., interpolation and extrapolation, and other procedures. Where either surface topography or water-table slopes change abruptly, more points of measurement are required for accurate construction of equal depth-to-water lines.

Depth to water table may also be shown by circumscribing areas within which depth to water table is in a specified range. On a scale map, note depths to water table as above. Select a convenient number of depth ranges, such as 0 to 2, 2 to 4, 4 to 10, 10 to 20, > 20, and delineate areas within which depth to water table is in the designated ranges. Distinguish between areas with a crosshatch, color, or other convenient code system. Maps such as the foregoing provide graphic information on the adequacy of drainage and, therefore, aid in showing areas in which artificial drainage may be needed.

(36c) Profile Flow Patterns for Ground Water

Equipment

Drafting instruments and supplies.

Procedure

On a profile section showing the soil surface and available information on subsoil stratigraphy, write in hydraulic-head values at points where hydraulic-head

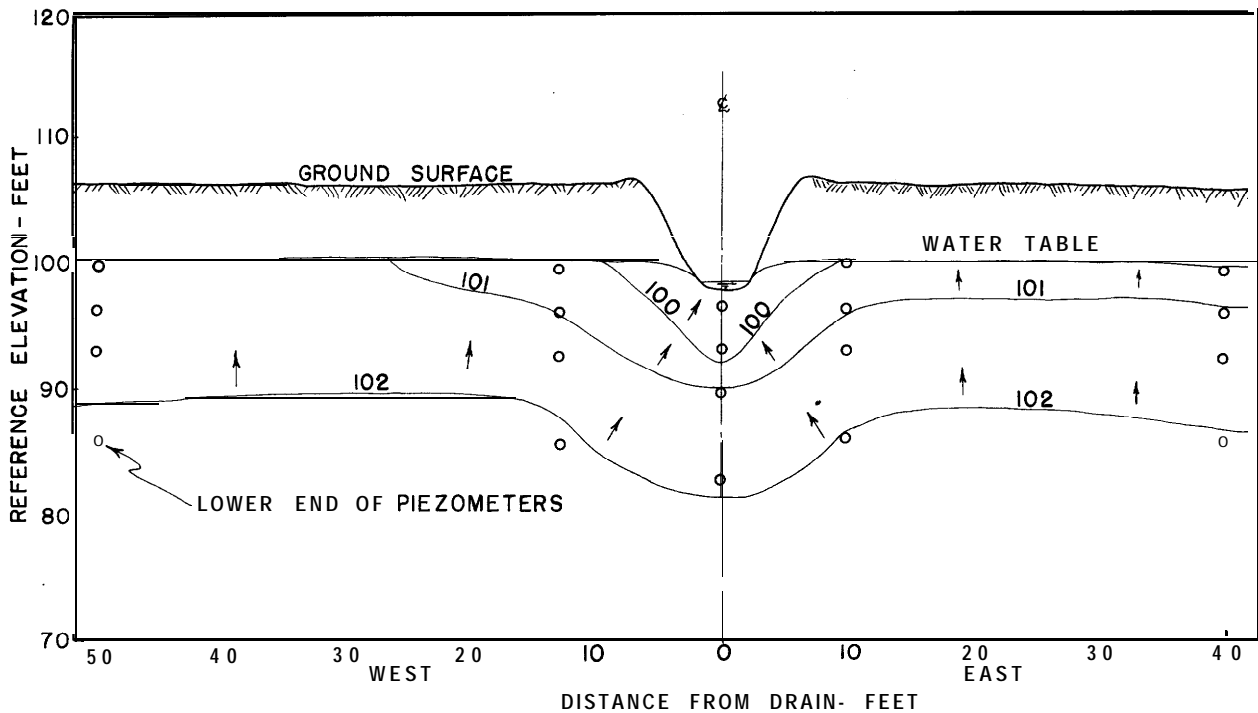


FIGURE 30.—Equal hydraulic-head lines below a water table on a profile section in the vicinity of an open drain. Example from Delta area, Utah. The direction of the hydraulic gradient is represented by arrows and indicates upward water movement from an underlying source.

measurements have been made, i. e., points where piezometers terminate in the soil (fig. 30). By standard methods, which are used for contour mapping, interpolation, and extrapolation, draw lines to connect points of equal hydraulic head. Convenient hydraulic-head intervals may be selected, extending over the range of measured values for hydraulic head. Usually an interval is selected that allows a number of equal hydraulic-head lines to be sketched on the same profile. The component of flow in the plane of the profile is normal to lines of equal hydraulic head, if the profile section is plotted to a 1 : 1 scale. With the 1 : 1 scale, flow lines can be sketched in at right angles to the equal hydraulic-head lines, with arrows to show the direction of flow. If the vertical scale is exaggerated, the relation between stream lines and equal hydraulic-head lines on the plotted profile is no longer orthogonal. Where the vertical and horizontal scales are not equal, therefore, the hydraulic-head distribution may be properly plotted, but flow lines should not be indicated.

For cases where hydraulic head changes in a vertical direction, indicating a vertical component of flow, the elevation of the water table can be determined by piezometers that terminate at the water-table level, or by extrapolation from a series of known points below the water table, as outlined in Method 35c. Draw equal hydraulic-head lines to intercept the water table at the respective equal hydraulic-head elevations.

An equal hydraulic-head line may intercept the water table at any angle, depending upon the flow direction. The water table is not necessarily a flow line as is often assumed, although it may be. A component of upward flow that exists below the water table may continue upward through the soil above the water table to the soil surface by capillarity. Likewise, downward flow may occur in the unsaturated soil above a water table.

References

Christiansen (1943), Reeves and Jensen (1949).

(36d) Water-Table Isopleths for Showing Time Variations in the Elevation of the Water Table

Equipment

Drafting instruments and supplies.

Procedure

A large seasonal variation in the water table often occurs in irrigated areas. In such cases it may be useful to show graphically the variations, both in space and time, by the use of water-table isopleths.¹⁷ By this

¹⁷ Private communication from M. Ram, Water Utilization Division, Ministry of Agriculture, Tel Aviv, Israel.

method a series of observation wells is established on a straight line across the area under investigation and water-table elevations are recorded over a period of time.

The graphical representation of the data is accomplished as follows: On a vertical scale at the left margin of a sheet of tracing paper, make a time scale on which the dates are shown for the various sets of readings of the observation wells. Start the time scale near the top of the sheet with the initial set of readings. Draw horizontal constant-time lines across the sheet at the time values for the various sets of readings. Across the top of the sheet, draw a profile of the elevation of the soil surface along the line of observation wells. Use any convenient vertical scale and mark the location of the observation wells on the horizontal scale. Draw in horizontal lines representing convenient elevation intervals over the range of variation in the elevation of the water table and plot the water-table profile for the initial set of readings. Project the points of intersection of this profile curve with the elevation scale lines downward to the horizontal constant-time line of the initial set of readings. Place elevation numbers above these points on the constant-time line. Repeat this process, locating successive elevation points on successive constant-time lines for each set of well readings. Connect constant water-table elevation points on successive constant-time lines with smooth curves. These curves are called isopleths and show the variation with time of the points of equal water-table elevation along the line of water-table observation wells.

The sources of ground water as well as subsurface stratigraphy must be taken into account in the interpretation of isopleths. The method provides a convenient graphical summary of water-table observations and can be used to advantage in showing the rate of subsidence of the water table following an irrigation season. This information relates directly to the drainability of soils.

Physical Measurements

(37) Intrinsic Permeability

(37a) Permeability of Soil to Air

Apparatus

The apparatus for this measurement is shown in figure 31. Compressed air is admitted through a calcium chloride drying tube to an airtight tank of constant volume. An outflow tube leads to a water manometer and to the soil sample container. The soil sample container consists of a tinned iron can with an extension made from a 4-cm. section of brass tubing counter-bored to give a snug fit on the top of the can. Punch an outlet hole, approximately $\frac{5}{32}$ in. in diameter in the bottom of the can. Use a disk of brass screen, 20- or 40-mesh, with 2 layers of fiberglass sheet

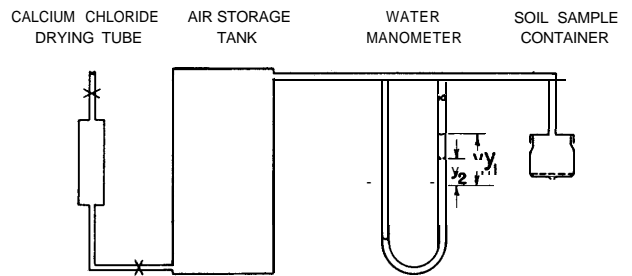


FIGURE 31.—Apparatus using air flow to measure the intrinsic permeability of soil by Method 37a. The manometer readings y_1 and y_2 represent successive heights of the free-water surface above the rest position.

as a filter in the bottom of each can. The soil-packing machine, mentioned below, is useful for this measurement.

Remarks

This method for measuring air permeability can be used for either disturbed or undisturbed samples. The following procedure is for disturbed soils. In order to get consistent results for comparing one soil with another and to determine the effect of various treatments, a standard procedure for preparing and packing the soil must be followed. Make determinations in triplicate.

Procedure

Pass air-dried soil through a wire-mesh sieve with 1-mm. openings. Obtain representative subsamples as outlined in Method 1. Attach the brass cylinder to the top of a can and fill the container about three-fourths full of soil. Dump the soil, which has been well mixed, into the container rather than by pouring or scooping. With a spring-load of 3-kg. wt. on top of the soil, drop the container 200 times on a solid block of wood from a height of 2.5 cm. A cam-operated mechanical dropper has been used for this purpose. Remove the brass cylinder from the can and use a spatula to strike the soil off level with the top of the can. Cover the soil with a disk of filter paper, place a lid on the can, and complete the seal with a tight rubber band or beeswax. Connection to the air source is made by means of a short piece of copper tubing soldered to the lid of the can. Pass compressed air through the drying tube to the tank until a manometer displacement of 40 to 50 cm. of water is attained. Record the air temperature in the tank and measure the rate of drop of the manometer level as air is allowed to flow from the tank through the soil sample. It is desirable to allow an initial 10-cm. drop of the manometer level before height and time readings are started.

Calculations

The intrinsic permeability of the soil using air is given by the equation :

$$k'_a = \frac{2.30 LVS \eta}{A P_a}$$

in which

k'_a = Intrinsic permeability with air, cm.²

L = Length of soil column, cm.

V = Volume of tank, cm.³

η = Viscosity of air at the temperature at which the determination was made, dyne sec. cm.⁻² (poises).

A = Cross-sectional area of sample, cm.²

P_a = Atmospheric pressure, dynes/cm.²

S = Slope of log γ vs. time curve =

$$\frac{\log_{10} \gamma_1 - \log_{10} \gamma_2}{At}$$

At

γ = Displacement of the water surface in one arm of the manometer, cm.

Δt = Time interval in seconds for the water surface in the manometer to drop from γ_1 to γ_2 .

The time for a convenient and measurable drop in manometer level can be controlled by the volume of the tank used. For most soils prepared as outlined above, a 213-liter (55-gal.) drum gives a convenient time interval for a measurable manometer change. For soils of lower permeability, a 24-liter tank is used.

While c. g. s. units are suggested above, any consistent set of units can be used.

References

Kirkham (1947), Soil Science Society of America (1952).

(37b) Permeability of Soil to Water

Apparatus

Constant-level water-supply reservoir, glass siphon tubes, and rack for supporting samples; soil containers made from 3-oz. tinned iron soil cans with $\frac{5}{32}$ -inch hole punched in bottom and brass cylinder extensions, 4-cm. high; 50- or 100-ml. graduates.

Procedure

The procedure for preparing and packing samples is as outlined in Method 37a. Water-permeability determinations may be made on the same samples used for air permeability. Care must be exercised to avoid disturbance of the sample in handling.

Following the air-permeability determination, place a brass cylinder extension on top of the can containing the soil sample and seal in place with an elastic band or beeswax. Place the soil sample on the rack, cover the soil surface with filter paper, and admit water to the sample from the supply reservoir with the siphon. The water level is adjusted so that the height of soil plus water column is 2 times the soil column, giving a hydraulic gradient of 2.

Record the water temperature, the time at which water is admitted to the container, and the time at which water first percolates through the sample. Meas-

ure the volume of percolate for a number of successive time intervals. The amount of water passed through the soil and the number of volume measurements made will depend upon the purpose of the determination. Usually 3 to 6 in. is used, a depth corresponding to an irrigation. For comparing one soil with another and for determining the effect of various treatments on a given soil, use a value obtained after the hydraulic conductivity has become more or less constant.

Calculations

Intrinsic permeability of the soil using water is given by the equation:

$$k'_w = \frac{\eta}{d_w g} k = \frac{\eta}{d_w g} \frac{VL}{A \Delta h \Delta t}$$

in which

k'_w = Intrinsic permeability with water, cm.²

k = Hydraulic conductivity, cm./sec.

V = Volume of percolate in time, Δt , cm.³

L = Length of soil column, cm.

Δh = Difference in hydraulic head between the inflow and outflow ends of the soil column, cm.

A = Cross-sectional area of the soil column, cm.²

Δt = Time interval for volume of percolate V to pass through the soil, sec.

η = Viscosity of water at the recorded temperature, dyne sec. cm.⁻² (poises).

d_w = Density of water, gm./cm.³

g = Acceleration of gravity, cm. sec.⁻²

Intrinsic permeability is related to hydraulic conductivity as indicated by the above equation. If desired, hydraulic conductivity may first be calculated and then converted to intrinsic permeability by multiplying by the ratio $\eta/d_w g$. While c. g. s. units are suggested above, any consistent set of units can be used.

Remarks

Soils may be compared with respect to permeability on the basis of values obtained at a fixed time after wetting or after a specified amount of water has passed through the sample. For comparing changes in structure between different soils, the time basis has been found to be preferable.

Reference

Soil Science Society of America (1952).

(38) Bulk Density

Apparatus

Balance, drying oven, moisture, boxes, and core sampler. The latter can be anything from an elaborate power-driven machine to a short section of thin-walled brass tubing with an internal closely fitting ring of clock spring soldered in place to form the cutting lip. (See drawing of soil sampler in Appendix.)

Procedure

Details of procedure will depend on the type of core sampler and soil conditions. Usually a flat soil surface, either horizontal or vertical, is prepared at the desired depth, and the core sampler is pressed or driven into the soil. Care should be taken to see that no compaction occurs during the process, so that a known volume of soil having field structure is obtained. The oven-dry weight of the sample is then determined.

Calculations

Bulk density (d_b) = (wt. of oven-dry soil core) / (field volume of sample). Bulk density is expressed as pounds per cubic foot or grams per cubic centimeter. For practical purposes, the latter is equal numerically to apparent specific gravity or volume weight.

(39) Particle Density

Apparatus

Balance, vacuum desiccator, and pycnometers.

Procedure

Weigh a pycnometer when filled with air (W_a), when filled with water (W_w), when partially filled with an oven-dried sample of soil (W_s), and when completely filled with soil and water (W_{sw}). To exclude air, pycnometers containing the soil with enough water to cover should be subjected to several pressure reductions in a vacuum desiccator and then allowed to stand for a number of hours under reduced pressure before completely filling with water for weighing W_{sw} . The particle density (d_p) of the soil in gm. cm.⁻³, is then given by the formula:

$$d_p = d_w (W_s - W_a) / (W_w + W_s - W_a - W_{sw})$$

where d_w is the density of the water in gm. cm.⁻³.

Slightly different and perhaps better values will be obtained for d_p if a nonpolar liquid such as kerosene, xylene, or acetylene tetrachloride is used for the displacing liquid.

(40) Porosity

The porosity of soil is the fraction of the soil space not occupied by soil particles. The porosity (n) may be calculated from the formula:

$$n = (d_p - d_b) / d_p$$

if the bulk density (d_b) and the particle density (d_p) are known. Solutions of this equation may be found graphically by use of the nomograms given at the right of figure 8 (ch. 2).

(41) Particle-Size Distribution

Remarks

The method as given is essentially that described by Kilmer and Alexander (1949), except that the 0.005-

mm. determination has been omitted. The names used at present by the United States Department of Agriculture for the soil separates are as follows: The diameters 2.0 to 1.0, 1.0 to 0.5, 0.5 to 0.25, 0.25 to 0.1, and 0.1 to 0.05 mm., respectively, separate very coarse, coarse, medium, fine, and very fine sands; particles from 0.050 to 0.002 mm. are called silt, and particles with effective diameters less than 0.002 mm. are designated as clay. With the International System, the diameters 2.0, 0.2, and 0.02, respectively, separate the classes represented by the numerals I, II, and III, while particles of diameters less than 0.002 mm. are represented by IV.

Apparatus

Set of sieves; size openings, 2-, 1-, and 0.5-mm. round hole; 60-, 80-, 140-, and 300-mesh per in. Pyrex nursing bottles, 8-oz., with rubber stoppers; Lowy 25-ml. automatic pipet; haw pipet rack; Pasteur-Chamberland filters, short, "F" fineness. Analytical balance, drying oven, steam chest, motor stirrer, reciprocating shaker, desiccator, beakers, and evaporating dishes.

Reagents

A. Hydrogen peroxide, 30 percent solution.

B. Dispersing agent. Dissolve 35.7 gm. sodium metaphosphate and 7.94 gm. sodium carbonate in water and dilute to 1 liter. The sodium metaphosphate is prepared as follows: 125 gm. of monosodium phosphate ($\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$) is slowly heated in a platinum dish to 650° C. This temperature is held for 1½ hr. The platinum dish and its contents are removed from the furnace and the sodium metaphosphate is cooled rapidly by pouring it out in narrow strips on a clean marble slab. The sodium carbonate is used as an alkaline buffer to prevent the hydrolysis of the metaphosphate back to the orthophosphate which occurs in acidic solutions.

Procedure

GENERAL STATEMENT.—Samples are routinely run in sets of eight; the necessary equipment is designed accordingly. The sample is treated with hydrogen peroxide, washed, filtered, and dispersed. The sand is separated from the silt and clay by washing the dispersed sample through a 300-mesh sieve. The various sand fractions are obtained by sieving, while the 20- μ and 2- μ fractions are obtained by pipeting. Organic matter is determined on a separate sample by the dichromate reduction method (Peech and co-workers, 1947).

PREPARATION OF THE SAMPLE.—The air-dried sample is mixed and quartered. The quarter reserved for analysis is rolled with a wooden rolling pin to break up the clods. The sample is then passed through a sieve with 2-mm. round holes. Rolling and sieving of the coarse material are repeated until only pebbles are retained on the sieve. The material not passing the sieve is weighed and reported as a percentage of the air-dry weight of the whole sample.

REMOVAL OF ORGANIC MATTER.—A 10-gm. sample of the air-dry soil containing no particles larger than 2 mm. is weighed on a rough balance and placed in a 250-ml. electrolytic Pyrex beaker. About 50 ml. of water is added, followed by a few milliliters of 30 percent hydrogen peroxide. The beaker is then covered with a watch glass. If a violent reaction occurs, the cold hydrogen peroxide treatment is repeated periodically until no more frothing occurs. The beaker is **then heated to about 90° C.** on an electric hot plate. Hydrogen peroxide is added in 5-ml. quantities at about 45-min. intervals until the organic matter is essentially removed as determined by visual inspection. Heating is then continued for about 30 min. to remove any excess hydrogen peroxide.

REMOVAL OF DISSOLVED MINERAL MATTER.—Following the hydrogen peroxide treatment, the beaker is placed in a rack and about 150 ml. of water is added by means of a jet strong enough to stir the sample well. The suspension is filtered by means of a short Pasteur-Chamberland filter of "F" fineness. Five such washings and filterings are usually sufficient except for soils containing much coarse gypsum. Soil adhering to the filter is removed by applying a gentle back-pressure and using the forefinger as a policeman. The beaker is then dried on a steam bath, placed overnight in an oven at 110° C., cooled in a desiccator, and then weighed to the nearest milligram. After the sample is transferred to a nursing bottle for dispersion, the oven-dry weight of the beaker is obtained. Weight of oven-dry organic-free sample is used as the base weight for calculating percentages of the various fractions.

DISPERSION OF THE SAMPLE.—To the oven-dry sample is added 10 ml. of sodium hexametaphosphate dispersing reagent B, and the sample is transferred to an 8-oz. Pyrex glass nursing bottle by means of a funnel, a rubber policeman, and a jet of water. The volume is made to 6 oz., and the bottle is stoppered and shaken overnight on a horizontal reciprocating shaker with 120 oscillations per minute. A similar volume of dispersing agent is placed in a liter cylinder, the volume made to 1,000 ml. and well mixed. A sample is taken with the pipet, dried, and weighed to obtain the weight correction referred to in the section on calculations. This weight correction is obtained for each new solution of sodium metaphosphate.

SEPARATION OF THE SANDS FROM SILT AND CLAY.—**The dispersed sample is washed on a 300-mesh sieve, the silt and clay passing through the sieve into a 1-liter graduated cylinder.** The sieve is held above the cylinder by means of a clamp and a stand. Jets of water should be avoided in washing the sample. The sieve clamp is tapped gently with the side of the hand to facilitate the washing procedure. Washing is continued until the volume in the cylinder totals about 800 ml. The sands and some coarse silt remain on the sieve. It is necessary that all particles of less than 20 μ diam. be washed through the sieve. The sieve is removed from the holder, placed in an aluminum pan, and dried at 110° to 120° C. While the sands are

drying, another sieve is used for the next sample. The material on the sieve is then brushed into a platinum dish and further dried for about 2 hr. The dish is then placed in a desiccator, the contents to be sieved and weighed when convenient. The silt and clay suspension in the cylinder is made up to 1 liter with distilled water, covered with a watchglass, and set aside until the pipetings are to be made.

PIPETING.—Pipetings are made for the 20 μ and 2 μ particles in the order named. The 20 μ particles are pipeted at a 10-cm. depth, the sedimentation time varying according to the temperature. The 2 μ fraction is pipeted after a predetermined settling time (usually 6 to 6½ hr.), the depth varying according to the time and temperature. A Lowy 25-ml. automatic pipet with a filling time of about 12 sec. is used. Prior to each sedimentation process, the material in the sedimentation cylinder is stirred for 6 min. with a motor-driven stirrer (8 min. if the suspension has stood for more than 16 hr.). After removal from the stirrer, the sedimentation cylinder is surrounded with insulating material and the suspension is stirred for 30 to 60 sec. with a hand stirrer, an up-and-down motion being used. This stirrer is made by fastening a circular piece of perforated brass sheeting to one end of a brass rod. A wide rubber band is placed around the edge of the brass sheeting to prevent abrasion. The time is noted at completion of the stirring. About 1 min. before the sedimentation is complete, the tip of the 25-ml. pipet is lowered slowly into the suspension to the proper depth by means of a Shaw pipet rack. The pipet is then filled and emptied into a 60-ml. weighing bottle having an outside cover. One rinse from the pipet is added. A vacuum is used to dry the pipet for use on the next sample. The weighing bottle is dried in an oven at 95° to 98° C. and then further dried for about 4 hr. at 110°. The initial drying is done at a lower temperature to prevent spattering of the suspension. The weighing bottle is then cooled in a desiccator containing phosphorus pentoxide as a desiccant and weighed.

SIEVING AND WEIGHING THE SAND FRACTIONS.—The dry sands, including some coarse silt, are weighed and brushed into a nest of sieves. Sieves and specifications are as follows:

Sieve Opening (mm.)	Specifications
1.0-----	Perforated brass plate, round holes, No. 3 straight, 0.04-in. diam. holes, 240 holes per in. ²
0.5-----	Perforated brass plate, round holes, No. 00 staggered, 0.02-in. diam. holes, 714 holes per in. ²
0.25-----	60-mesh, Bureau of Standards (Phosphor Bronze wire cloth)
0.177----	80-mesh, Bureau of Standards (Phosphor Bronze wire cloth)
0.105----	140-mesh, Bureau of Standards (Phosphor Bronze wire cloth)
0.047----	300-mesh (Phosphor Bronze wire cloth), 0.0015-in. wire.

(An 80-mesh sieve is included in order to obtain International Society of Soil Sciences fraction I.) The sands are then shaken for 3 min. on a shaker having vertical and lateral movements of $\frac{1}{2}$ in., making 500 oscillations per minute. For a different shaker, the time of shaking would have to be determined by microscopic study. The summation method of weighing is used. The first sand fraction is weighed, the second fraction added to it, the total weight determined, and so on. If the sum of the weights of the fractions is equal to the total weight, it is assumed that no weighing error has been made.

Calculations

PIPETED FRACTIONS.—(A - B) KD = percent of pipeted fraction where A = weight in gm. of pipeted fraction, B = weight correction for dispersing agent in gm.),

$$K = \frac{1,000}{\text{volume contained by pipet}}$$

and

$$D = \frac{100}{\text{organic-free oven-dry weight of total sample}}$$

(The' 20μ fraction) - (the 2μ fraction) = International Society of Soil Sciences fraction III. United States Department of Agriculture silt is obtained by subtracting the sum of the percentages of sand and clay from 100. International Society of Soil Sciences fraction II is obtained by subtracting the sum of the percentages of fractions I, III, and IV from 100.

SAND FRACTIONS.—

$$\frac{(\text{Weight in grams of fraction on sieve} \times 100) \div (\text{organic-free oven-dry weight of total sample})}{\text{percent of fraction}}$$

References

Kilmer and Alexander (1949), Peech and others (1947), and Tyner (1940).

(42) Aggregate-Size Distribution

(42a) Wet Sieving

Remarks

This is a modification of the mimeographed tentative method that was distributed in August 1951 by the Committee on Physical Analyses of the Soil Science Society of America. The method in brief consists of placing a sample of soil on a nest of sieves that is oscillated vertically under water. The amount of soil remaining on the individual screens is determined, and aggregation is expressed as the mean weight-diameter of the aggregates and primary particles. After weighing, the aggregate separates are combined and dispersed and washed through the nest of sieves. The

resulting separates make it possible to correct the previous separates of aggregates for primary particles and to calculate the aggregation index. This is a single-value index of the aggregation of a soil.

Apparatus

Yoder-type wet-sieving apparatus, sieve holders, 4 sets of 5-inch sieves with 2-, 1-, 0.5-, 0.25-, and 0.10-mm. openings (corresponding to United States Screens Nos. 10, 18, 35, 60, and 140), drying oven, moisture cans, balance Pyrex watchglasses, and 6-in. diameter porcelain funnel.

Procedure

Collect the soil sample with spade or garden trowel, preferably when the soil is moist, avoiding excessive compaction or fragmentation of soil. Dry the sample slowly and, when sufficiently friable, pass it gently through an 8-mm. sieve and air-dry. If the soil is stony, pass the sample through a 4-mm. sieve and discard all primary material greater than 4 mm. in size. Mix the soil and take subsamples in accordance with Method 1. Make determinations in duplicate on 40- to 60-gm. subsamples. Weigh the subsamples to the closest 0.1 gm. and determine the moisture content by drying a separate subsample at 105° C.

Install the nests of sieves in the water slowly and at a moderate angle to avoid entrapping air bubbles below the sieves. Adjust the mechanism so that the top sieve makes contact with the water surface when the oscillation mechanism is at the top of its stroke. Distribute the sample on the top sieve so that wetting occurs by capillarity and wait 5 to 10 min. after the soil surface appears wet to insure saturation of the aggregates. Oscillate the sieves for 30 min. with a stroke of 3.8 cm. and a frequency of 30 cycles per minute, keeping soil submerged at all times. Some attention may be required during the first few minutes of operation, in order to prevent water from spilling over the top sieve, and later, to prevent the top sieve from rising above water level.

Remove the sieves from the water and drain for a few minutes in an inclined position. Remove excess water from the bottom of the screens with absorbent tissue and place the sieves on watchglasses. Dry in a circulating oven at not higher than 75° C. because high temperatures cause some soils to adhere. Then remove the soil from the sieves, dry at 105°, and weigh.

In order to determine how much of the soil retained on the individual sieves represents aggregates and how much is gravel and sand, the oven-dried soil taken from the five sieves is dispersed and washed through the sieves with a stream of water. The oven-dry weight of the primary particles remaining on each sieve is then determined.

Calculations

The amount of soil remaining on each sieve is expressed as percentage of the total sample. Prepare a

graph, plotting the accumulated percentage of soil remaining on each sieve as ordinate against the upper limit of each fraction in millimeters as the abscissa, and measure the area shown by the curve connecting these points and by the ordinate and the abscissa. If 1 mm. (sieve size) represents 1 unit of the abscissa and 10 percent a unit on the ordinate, a square unit will represent 0.1 mm. mean weight-diameter of the aggregates of the sample. Multiplying the number of square units of the area by 0.1 gives the mean weight-diameter of the entire sample, including the material that has been washed through the smallest sieve.

The results from the wet sieving of the dispersed sample are plotted and calculated in the same way. The difference between the mean weight-diameters of the original and the dispersed samples gives the aggregation index.

Remarks

The water container in which the sieve nest is oscillated can be of any desired size or shape, providing its area is at least 1.6 times the area of the sieves. The temperature of the water should be in the range 20° to 24° C., and the water should not be excessively saline. Fresh water should be used for each set of determinations. Rubber bands cut from old inner tubes are convenient for holding loosely fitting sieves together.

References

Russell (1949), Van Bavel (1950).

(42b) Aggregation of Particles Less than 50 Microns

Remarks

This procedure measures the degree of aggregation of the silt and clay (less than 50 μ) fraction for those soils that do not contain enough large aggregates to be adequately characterized by wet-sieving. The method involves measuring the concentration of two suspensions of the same soil, one of which is dispersed by any standard dispersion procedure to give total silt plus clay. The other suspension, prepared by mild (end-over-end) agitation of the sample in water, gives a measure of the unaggregated silt plus clay. The difference in concentration between the two suspensions provides a measure of the amount of silt plus clay particles that is bound into water-stable aggregates larger than 50 μ in size.

This procedure may also be used as a rapid exploratory test to determine the effect of various soil-aggregating chemicals in producing water-stable aggregation.

Apparatus

Dispersion apparatus with high-speed stirring motor: metal cup, 1-liter hydrometer jars, thermometer, and

Bouyoucos hydrometer or hydrometer-pipet. If the pipet procedure is used, a Lowy automatic pipet, a Shaw pipet rack, and tared moisture boxes are needed.

Procedure

Weigh two 50-gm. subsamples of air-dried soil prepared as in Method 42a. Make a moisture determination on a separate subsample.

TOTAL SILT PLUS CLAY.—Disperse one of the subsamples in the dispersion apparatus. Transfer to a hydrometer jar and dilute with distilled water to the required volume. For procedure *a* (below) the final volume is 1,130 ml., determined with the hydrometer in the suspension; for procedures *b* and *c*, the volume is 1,000 ml. (Note: If either procedure *b* or *c* is to be used, stopper and invert the cylinder 2 or 3 times and record the temperature. This is necessary in order to determine in advance the settling time used.) Stopper, invert, and shake the cylinder vigorously several times and determine the total silt plus clay in the suspension as directed under procedures *a*, *b*, or *c*, given below.

UNBOUND SILT PLUS CLAY.—Incline the hydrometer jar containing the second subsample to a nearly horizontal position and shake lightly to spread the sample over a distance of 10 or 12 cm. along the side of the jar. Add distilled water slowly and in such manner as to favor wetting by capillarity rather than by flooding. When soil is completely wetted, dilute to the appropriate volume, as given above, but do not allow water to fall directly on soil. Allow the soil to slake for at least 15 min. Record the temperature. Stopper the cylinder and gently invert it 20 times (do not shake) within a period of about 40 sec., requiring about 1 sec. for inversion with a 1-sec. interval between inversions. After the required settling period, determine the amount of unbound silt plus clay in suspension by the same procedure *a*, *b*, or *c* used for the total silt plus clay measurement.

PROCEDURES FOR MEASURING CONCENTRATION OF SUSPENSIONS.—(a) *Hydrometer.* After final mixing, insert the hydrometer and take a reading after 40 seconds, as prescribed by Bouyoucos (1936). Immediately record the suspension temperature in degrees F. and apply a temperature correction as follows: Add 0.2 to the hydrometer reading for each degree above 67° F.; subtract if below.

The corrected hydrometer reading gives the grams per liter of silt plus clay in suspension.

(b) *Hydrometer-pipet.* The hydrometer-pipet (Hellman and McKelvey, 1941) measures the concentration of soil particles in grams per liter of a suspension that has been pipeted from a known depth and that is uniform throughout when measured by the hydrometer contained within the pipet. The settling time and depth of sampling are the same as for the pipet procedure *c*, and the hydrometer readings require the same temperature corrections as for the hydrometer procedure *a*.

After final mixing and a few seconds prior to time of sampling, squeeze the bulb and insert the hydrometer-pipet into the suspension to a depth of 12.5 cm. Start filling the pipet after the prescribed time interval in seconds, as indicated under procedure c. Care should be taken to keep the suspension well mixed by occasionally allowing a bubble to rise through the suspension in the pipet. Read the hydrometer at the top of the meniscus and apply the proper temperature correction as prescribed for the Buoyocous hydrometer.

The corrected hydrometer-pipet reading gives the grams per liter of silt plus clay in suspension.

(c) *Pipet.* After final mixing, insert the Lowy or other suitable pipet into the suspension to a depth of 12.5 cm. Start filling the pipet after the time-interval in seconds indicated by the accompanying data. Other depth-time-temperature relationships may be obtained from the nomograms of Tanner and Jackson (1948).

Temperature :	Time to sample at 12.5-cm. depth (seconds)
"C. or "F.	
20 (68) -----	57
22 (71.5) -----	54
24 (75) -----	51
26 (79) -----	49
28 (82.5) -----	46
30 (86) -----	44

The weight in grams of oven-dry (105° C.) material in the 25-ml. aliquot is multiplied by 40 to give grams per liter of silt plus clay in suspension.

Calculations

Percent aggregation = (wt. of total silt plus clay in dispersed suspension, gm., minus wt. of silt plus clay in undispersed suspension, gm.) $\times 100$ / (wt. of total silt plus clay in dispersed suspension, gm.).

References

Bouyoucos (1936), Hellman and McKelvey (1941), and Tanner and Jackson (1948).

(43) Modulus of Rupture

Remarks

In the following procedure, the maximum force required to break a small specially molded briquet of soil is measured, and from this breaking force the maximum fiber stress in the standard sample is calculated.

Shrinkage of soil material on drying is a pertinent property and can be determined from the dimensions before and after drying of the briquet samples used in this test.

Apparatus

The machine for breaking the sample makes use of 2 parallel bars 5 cm. apart for supporting the sample. The breaking force is supplied from a third overlying bar centrally located and parallel with respect to the supporting bars. The bar above and one bar below are

self-aligning to accommodate to any slight lack of parallelism in the line of bearing on the sample. The bars are coated with a strip of soft rubber with a cross section 0.16 cm. square. The breaking machine is mounted on the platform of a beam balance. The briquet sample is broken by upward motion of the two lower bars, which are supported by the platform of the balance. Upward motion of the upper bar is constrained by a cross frame above the balance. The breaking force is supplied by water accumulating in a vessel hung from the end of the balance beam. The breaking force is applied at the rate of 2,000 gm.-wt./min., and the beam motion that occurs when the sample breaks can be used to automatically stop the accumulation of water in the vessels.

The briquet molds are precision made from $\frac{3}{8}$ -in. brass strip with inside dimensions of $3\frac{1}{2}$ cm. by 7 cm. by 0.952 cm. high. Rectangles of hard white photographic blotting paper are cut to the size 5 cm. by $8\frac{1}{2}$ cm. A screen-bottomed tray, 50 cm. square, is made by pulling brass window screen taut and soldering it to a rigid $\frac{1}{2}$ -in. galvanized pipe frame. A pan or other water container slightly larger than 50 cm. square. Graduated cylinder. Tremie funnel with straight cylindrical tube 2 cm. diam. and 10 cm. high. (See drawing of apparatus in Appendix.)

Procedure

Make the determination on 6 replicate samples of soil that have been passed through a 2-mm. round-hole sieve, using the subsampling procedure outlined in Method 1. Samples should be just slightly larger than will fill the briquet molds. Cover the inside of the molds with a thin layer of Vaseline so that the soil will not stick to the mold. Place the screen-bottomed tray in the pan. Place the molds on the blotting paper on the screen. Rest the tremie on the blotting paper at one end of the mold. Dump all of a soil subsample into the tremie. Move the funnel around inside the mold while raising continuously so as to give a uniform smooth filling of the mold.

Strike off excess soil level with the upper surface of the mold. Add water to the pan until free water surrounds every mold. Allow samples to stand for 1 hour after all the soil samples become wet. Raise the screen very carefully so as not to jar the samples and transfer to a forced-draft oven at 50° C. After drying the briquets to constant weight, remove from the molds and determine the breaking strength.

Calculations

Use the formula $s = (3FL) / (2bd^2)$, where s is the modulus of rupture (in dynes per sq. cm.), F is the breaking force in dynes (the breaking force in gm.-wt. $\times 980$); L is the distance between the lower two supporting bars, b is the width of the briquet, and d is the depth or thickness of the briquet, all expressed in cm.