LABORATORY METHODS OF SOIL ANALYSIS

CANADA-MANITOBA SOIL SURVEY

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Edited by

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ACKNOWLEDGEMENTS

This manual represents a revision and update of the methods of soil analysis currently in use by the Canada-Manitoba Soil Survey. The material was largely derived and updated from *Laboratory Methods of the Manitoba Soil Survey* (Beke, 1972), with additional information obtained from the *Manual on Soil Sampling and Methods of Soil Analysis* (McKeague, 1981) and *Methods of Soil Analysis* (Page, 1982). The last two provide more detailed information on these and other procedures.

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PREFACE

Values of error for some methods will indicate the precision and accuracy of these analyses. Rates of analysis will vary with different procedures, giving as approximate indication of the time required to perform them.

As a check on accuracy of lab data, standard or reference samples should be included routinely in batch analyses.

Accessories required for all procedures include balances (sensitive to four decimal places), distilled or deionized water, and glassware of various types, sizes and graduations. Also in use are manufactured reagents of specified concentration and purity, as opposed to reagents created in the lab.

I SAMPLE PREPARATION

1. When samples come in from the field arrange them:

   i) according to date;

   ii) within the dates organize by location and site number;

   iii) finally separate them by surveyor.

2. Once the samples are organized by first sampled with lowest site number and increasing depth then record the tag information on the daily sample sheets. Once you have done this take the sample number from the daily sample sheet and write it on the tag.

		Site No.		
Eg) for ordering	05/07/26	001	goes before 05/07/27	001
	05/07/26	001	goes before 05/07/26	002

NOTE: **IF** there is no letter in the “H” box then it is a “W”.

IF there is a number in the “Lit” or “Mod” box then the number goes BEFORE the horizon suffix (eg 1 Ckgj)

MAKE SURE TO PUT ALL INFORMATION THAT IS ON THE TAG ON THE DAILY SAMPLE SHEET.

IF you are missing data see the surveyor for their sample sheets.

3. Get a tray and a plastic bag (wipe down the bag with a moist towel) for the sample. Open the bag and pour the sample onto the tray **ON** the plastic bag. Place the tag near the edge of the bag so that you know which sample is which.

Spread the sample over the bag in order for the sample to dry evenly. Make sure to break down big clumps of soil so that they dry properly.

IF you have Electrical Conductivity tests requested for a sample take the soil out first, so that you don't have to add more H_20. Just make sure to fill the E.C. container full of soil.

4. Once the samples are dried take soil for the tests requested.

   10 grams in a 400 mL beaker = MECHANICAL

   15 grams in plastic 50 mL beaker = pH

   Half full metal tin = organic carbon and carbonate

   250 grams in plastic 400- mL beaker = E.C.

   IF complete is circled on the tag then the sample is a profile sample and should be entered on a profile sheet. It gets all the tests including C.E.C.

   IF the sample is stony, remove the stones and calculate the percent stones.

5. After the soil is taken for all the tests place the tag (WRITING FACING OUT) in a sample container and fill the container with soil. The extra soil is thrown out. Write the sample number on the container and its lid. Then put the samples in the sample drawer.

   IF there were stones after they have been weighed and the percent stones calculated place the stones in a little plastic bag and put it with the sample **in** the container.

Most procedures require that samples be air-dried. This entails spreading the soil on trays of plastic sheeting, mixing and rolling to break up clods. The sample is gently crushed and sieved until only coarse fragments (>2 mm) remain. These coarse fragments are weighed and recorded as “% stones”. Some procedures require samples to be oven-dried and ground to pass a .42 mm (40 mesh) sieve.

The procedures mentioned above are common in the preparation of mineral soil samples. In the case of organic samples, a portion of the soil in the undisturbed state must be stored in a freezer for fiber analysis. When the soil is to be analyzed for minor elements, nylon or stainless steel sieves should be used.

DETERMINATION OF RUBBED AND UNRUBBED FIBER (VOLUME BASIS)

The procedure outlined is designed to give approximate values for un-rubbed and rubbed fiber content on a volume basis of organic soils and is based on the method of Lynn and McKenzie. (1)

EQUIPMENT

- 5 cc half syringe

  a plastic syringe is modified by cutting away half of the cylinder wall in a longitudinal direction.

  NOTE: Brunswick and Monoject are brands of syringes which have calibration marks imbedded in plastic and are suitable for extended use.
- 500 mL beakers
- 100 mesh sieve

- Spatula

REAGENT

1% Sodium metaphosphate [$(NaPO_3)_6$], dispersing solution.

PROCEDURE FOR UNRUBBED FIBER CONTENT (%)

1. Prepare sample by placing about 25 cc of moist organic sample on a paper towel; roll up the paper and squeeze lightly to expel surplus water.
   NOTE: the objective is to dry the sample until it does not glisten, but is still very moist.
2. Pack the modified syringe (adjusted to 5 cc capacity level) by filling with sample and pressing with a spatula hard enough to expel air but not water.
3. Transfer all of the soil from the syringe into a 500 mL beaker using cold tap water. Add 100 mL of dispersing solution and bring volume to 400 mL. Stir for one minute and allow to stand overnight.
4. Stir several times and transfer onto a 100 mesh sieve. Wash with cold tap water from a faucet adjusted to deliver about 400 mL of water in 5 seconds until the water passing through the sieve appears clear.
5. Collect the sample together at one side of the sieve and transfer to a paper towel by inverting and tapping the sieve.

6. Remove excess water by lightly pressing a paper towel onto the sample surface. Pack sample into syringe and record volume as “percent unrubbed fiber.”

7. Transfer sample to 100 mesh sieve for determination of rubbed fiber content.

PROCEDURE FOR RUBBED FIBER CONTENT (%)

1. Rub sample between thumb and fingers under a stream of water until water passing through the sieve is clean.

   NOTE: Clean fibers will roll between thumb and fingers rather than slide or smear.

2. Dry sample and transfer it to syringe as outlined in procedure for unrubbed fiber content.

3. Record volume as “percent rubbed fiber”.

ERROR VALUE:

n	$\bar{X}$	SD	RSD(%)
8	92	4.0	4.4

Rate of analysis: 8-10 / day

REFERENCES

1) LYNN, W.C., and McKENZIE, W.E. 1971. Field tests for organic soil materials. U.S.D.A. Soil Cons. Serv., Lincoln, Nebraska.

PARTICLE SIZE ANALYSIS (PIPET METHOD)

Mechanical Procedure

1. Weigh 10 grams of soil into a numbered beaker. Record sample number on mechanical input sheet next to its corresponding beaker number.
2. In fume hood:

   a) slightly wet soil with distilled water. Add 5 mL of Hydrogen Peroxide to each sample and cover with watch glass.

   b) Once fizzing stops add 10 mL.

   c) Repeat b) until 35 mL of Hydrogen Peroxide has been added.

   d) Fill beaker to 150 mL (300 mL if left for weekend) with distilled water and leave overnight.
3. In fume hood: Place samples on the heater and turn the heater to 200^{o}C. Increase the temperature by 100^{o}C every 20 minutes. Add Hydrogen Peroxide (amounts vary by colour and reactivity) during the "cooking" process. Generally, samples will become lighter in colour with cooking, particularly with samples that start off dark brown or black. When no organic matter is left in the sample, remove from heat. and fill beaker to 300 mL. Leave overnight.

   Another thing to watch for is the type of bubbles produced when Hydrogen Peroxide is added. There will be some bubbling even if cooking is done, when Hydrogen Peroxide is added. These bubbles are bigger (and tend to be near the side of the beaker) and stop fast.

   Big bubbles are characteristic of carbonates.

Watch for foam or little bubbles forming. This indicates lots of organic matter (NOTE: some other chemicals will make this type of bubbling so watch colour change as well). No colour change means it is probably done.

Some warnings for cooking process:

Don't let samples boil over! If sample looks like it will boil over:

a) remove from heat OR

b) add a drop or two of octanol (inhibits reaction) OR

c) blow sample with air from an empty dispensing bottle (this works for bigger bubbles).

Don't let samples dry out! If it does then the beaker may break and the sample will be destroyed. Make sure to watch the level of the water and add distilled water often.

IF you leave for lunch add distilled water to bring the water level to 300 mL and decrease the heat to <100^{o}C,

IF you are in the mechanical room then your samples can be at ~ 150 mL.

IF you are doing other tests in different rooms then 200 mL should be the volume in the beakers (to avoid drying out) **BUT** make sure to check on it every **20 minutes**!

NOTE: Try not to add Hydrogen Peroxide to samples after 4:00 p.m. Turn heat off before 4:15.

4. After the samples have sat for a while (ideally overnight) check for salinity. To check, look at whether or not there is a clear liquid. If there is, the liquid can be

suctioned out. BE CAREFUL! DON'T SUCTION THE SAMPLE FROM THE BOTTOM AND WATCH FOR SOIL FALLING OFF THE SIDES OF THE BEAKER. If soil falls down from the sides, stop.

IF you think the solution is a little too cloudy (and it has sat overnight/long time) double check if salinity is present by tipping the beaker slightly.

IF the soil layer is fluid and moves then there is salinity present and suction it. Fill the beaker up to 300 mL again and watch for the solution to settle quickly.

IF the solution remains cloudy for a while (30 minutes or more) then it is ready for the next step.

5. Fill all the beakers to ~ 400 mL. It is important to keep concentrations constant so make all beakers the same volume. Pour about 250 mL into a "milkshake" container. Swirl the remaining contents and pour about 50 mL into the "milkshake" container. To clean the beakers/lids, rub with the spatula and rinse with water from the FINE POINT SQUIRT BOTTLE. Make sure that the sides and lid are extremely clean and rub the bottom of the beaker to loosen the soil. Pour the remainder of the solution into the "milkshake" container. Tilt the beaker and rinse it with the FINE POINT SQUIRT BOTTLE until all soil is in the "milkshake" containers and the beaker is clean.

   Add **10 mL** of dispersing agent to the "milkshake" container. Place the container on the mixer. Make sure the container is not loose! Set the timer **15 minutes**. The speed of the mixer should be enough to move the soil on the bottom of the container. IF it's too fast, the sample will splash. Too slow, the sample will not move much.

Once it's done remove the container from the mixer and rinse the mixer very well into the container.

DON'T FORGET TO ADD THE DISPERSING AGENT.

DON'T ADD TOO MUCH WATER OR IT WILL AFFECT THE CONCENTRATIONS.

6. Filter the sample into its corresponding graduated cylinder:

1 – 10 & 31 – 40 $\longrightarrow$ 1 – 10 graduated cylinder

11-20 & 41 – 50 $\longrightarrow$ 11 – 20 graduated cylinder

21 – 30 & 51 – 60 $\longrightarrow$ 21 – 30 graduated cylinder

BE CAREFUL NOT TO OVERFILL THE SIEVE. IF THE WATER WON'T DRAIN

a) Rub the bottom of the sieve with your finger to move the soil (rinse your finger after [into the sieve])

b) Place the spout of the squirt bottle near the bottom of the sieve and squirt some water. This will move the soil around.

Rinse the container very well; all around the top of the container (so all soil is at the bottom) and then tilt it while spraying at the back of the container.

Rinse the soil left in the sieve with the distilled water from a regular point squirt bottle. Rinse the soil until the water runs clear. If you tilt the sieve to get the sand to one side it will be easier for the next step. The graduated cylinder has clay and silt while the sieve has sand.

Weigh the sand beakers BEFORE putting the sand in! Take the sieve and rinse it into its corresponding beaker (same as graduated cylinder). To check if there is no more clay or silt with your sand: let the beaker contents settle and check if the

water is clear. If it is then the sand samples can go into the oven for the night. If it is not then pour it back into the sieve (over its corresponding graduated cylinder) and rinse some more until the water is clear.

Fill the graduated cylinder to **1000 mL** and place it in a 32^0C water bath for overnight. Also, put 2 water bottles filled with distilled water in the water bath. In all of the above steps be careful not to over fill or spill the samples.

NOTE: **IF** your water bath is not 32^0C, but close just note the temperature. The temperature affects the morning to afternoon wait in Step #7. A variance in temperature needs to be taken into account for the timing.

7. To determine amount of clay and silt:

Morning

a) Prepare the samples by filling the graduated cylinders to **1000 mL** from the squirt bottles in the water bath.

b) Fill a dedicated graduated cylinder with distilled water for the hand mixer.

c) Mix the graduated cylinder samples very well for 30 seconds. MAKE SURE TO MIX THE BOTTOM INTO SOLUTION OR YOUR FINAL RESULTS WILL BE WRONG.

d) Take the 25 mL volumetric pipette and place it in the graduated cylinder so that the black line is just under water. Take a 25 mL sample and place it in its corresponding beaker.

NOTE: you can set up the beakers in front of the water tank in order of the graduated cylinders to save time.

e) Rinse the pipette into the beaker with distilled water from the FINE POINT SQUIRT BOTTLE. Make sure to rinse the inside and the outside (where the pipette was in the water).

f) Place the beakers in a tray and put them into the oven for overnight.

NOTE: Before you start, check the temperature of the bath and check the time. The temperature affects when you will be sampling next. For example: if you start at 9:30 and the temperature is 32^{o}C you can sample after 6 hours (3:30). There is a sheet to obtain this data.

Afternoon:

Repeat Steps d), e) and f).

DON'T MIX THIS TIME!

DON'T FILL THE GRADUATED CYLINDER.

THE OVEN SHOULD BE AT 110^{O} C.

8 Remove the samples from the oven and place them in the desiccator to cool for **one hour**. Make sure to seal the desiccators properly.

FOR SILT AND CLAY BEAKERS:

Weigh the beakers to four decimal places and record the weight on the mechanical input sheet.

NOTE: Only take 2 beakers out at a time and you should seal the desiccators after opening it.

DON'T CLEAN THE BEAKERS

The final weight of the beaker will be the starting weight for the next sample in that beaker.

FOR SAND BEAKERS:

Weigh the beakers to three decimal places and record the weight on the mechanical input sheet.

NOTE: Only take 2 beakers out at a time and the desiccators should be resealed.

IF there is one gram or more of sand then you need to separate it into categories.

IF it is less than one gram then dump it into the sand waste beaker.

To separate the types of sand:

Scrape the beaker to release the sand, then pour it into the stack of sieves and place it in the shaker for **10 minutes**. Once it is done weigh each section's sand by placing it on a weigh dish. Make sure to zero the scale with the empty weigh dish on it. Tap the sieves to make sure all the sand is collected. Weight and record the results on the mechanical input sheet. Dump the sand in the corresponding flasks.

NOTE: Course fragments/stones are not soil. To determine if there are course fragments use the 2 mm sieve. Anything that doesn't pass though is a course fragment and should be weighed and recorded.

PARTICLE SIZE ANALYSIS (PIPET METHOD)

Particle size analysis by the pipet method involves the measurement of proportions of particles within stated size classes by relative weight. Fractionation of soil particles into distinct classes is achieved by sieving and sedimentation. Pretreatments

are employed to remove organic matter and soluble salts from the sample (carbonates are not removed in this procedure).

EQUIPMENT

- 50, 100 AND 400 mL beakers
- Watch glasses for 400 mL beakers
- 1000 mL graduated cylinders, polypropylene
- Set of 8 cm diameter sieves with openings in various sizes (in mm):
  - 1.0; 0.5; .25; .105; .053 (18, 35, 60, 140, 270 mesh)
- Additional sieves with openings of .074 mm (200 mesh) – used for AASHO and UNIFIED systems
- Electric stirrer with timer (Hamilton-Beach “milkshake” type)
- Constant temperature water bath with clips for holding 10-12 1000 mL cylinders
- Hot plate
- Sieve shaker and timer
- Oven
- Dessicator
- Stand for sieving
- Balance with 4 decimal place sensitivity

REAGENTS

- Hydrogen Peroxide (H_2O_2) 35%

- Anti-foaming agent – Octanol [$CH_3CHOH(CH_2)_5CH_3$]
- Dispersing agent – 35.7 g of Sodium metaphosphate [$NaPO_3)_6$] and
  7.94 g of Sodium carbonate [Na_2CO_2] per litre
- Silver nitrate ($AgNO_3$) .1N – used in spot testing for Cl^-.
- Barium chloride ($BaCl_2$).5N – used in spot testing for SO_4^{2-}.

PROCEDURE

1. Place 10 g of 2 mm air-dry sample into a 400 mL beaker with approx. 20 mL distilled water and 10 mL of 35% H_2O_2 and cover with a watch glass.

   NOTE: Spraying with distilled water from a wash bottle can be used to reduce frothing in the event of a violet reaction.

   CAUTION: Avoid contact with 35% H_2O_2.
2. After reaction has subsided, add another 5 mL of 35% H_2O_2 and leave sample overnight for digestion.
3. Place sample on a hot plate at a low temperature for further digestion, with additional H_2O_2, until frothing ceases. Octanol may be added as necessary to reduce frothing. Allow digestion process to continue on the hot plate to remove excess H_2O_2, but the sample should not be allowed to dry out.
4. Soil adhering to the sides of the beaker or watch glass is washed down with distilled water, bringing the volume up to approx. 400 mL. Allow the suspension to stand overnight.

5. Once the supernatant is clear, siphon off excess liquid and test on a spot plate for sulfates and chlorides with $BaCl_2$ and $AgNO_3$ respectively. (If salts are present, it may necessitate an additional washing with 300 to 400 mL of distilled water and allowing a day to settle, after which excess liquid is siphoned off and tested).
6. Transfer the contents of the beaker into a stirrer cup containing 20 mL of dispersing agent and stir for 10 minutes at medium speed.

SEPARATION OF SAND FRACTION

1. Place a sieve of 8 cm diameter with openings of 53 um (270 mesh) in a large funnel held above a 1000 mL bylinder. Transfer the contents of the cup into the sieve and wash until the water passing through is clear.

   NOTE: Work the sediment from side to side while washing to remove all silt and clay from the sand.
2. Transfer the sand into a tared 100 mL beaker. Inver the sieve in the funnel and remove any remaining sand by washing with distilled water.
3. Siphon off excess water from the beaker, dry the sand at 105^{o} C, cool in a dessicator and weigh. If the total sand weight is greater than 1.0 g, transfer the sample to a nest of 8 cm diameter sieves arranged in order: 1 mm (top; 0.5 mm, 0.25 mm, .105 mm (18, 35, 60, 140 mesh). Fit the next with top and pan and place onto a shaker for 3 – 5 minutes.
4. Weigh and record each sand fraction to 2 decimal places.

SEPARATION OF SILT AND CLAY

1. Bring the volume of the cylinder containing the silt and clay suspensions up to 100 mL by adding distilled water, cover with a watch glass and pace in a water bath. Allow cylinder to equilibrate and check for flocculation before sampling.
2. Insert a perforated plunger to the bottom of the cylinder and stir vigorously for several seconds to loosen any sediment.
3. Stir the suspension more methodically, using 20 slow, even vertical strokes, and record time at which stirring is completed.
4. Remove plunger and draw off a 25 mL aliquot from a depth of 10 cm. Place aliquot into a tared 50 mL beaker, along with rinse water from pipet.
5. Dry at 105^{o} C, cool in a dessicator and weigh to 4 decimal places. (Record as weight of silt, clay and dispersing agent).

CLAY SEPARATION

Maintain the cylinder in the controlled temperature water bath until the silt greater than 0.002 mm settles below 10 cm depth. (see Table 1).

Table 1.

TEMP ^{o} C	SEDIMENTATION RATE TIME	
	Hrs.	Min
25	7	4
26	6	56
27	6	47
28	6	38
29	6	29
30	6	20

31	6	11
32	6	0
34	5	42

2. Remove a 25 mL aliquot from a 10 cm depth below the suspension surface. Discharge aliquot into a 50 mL beaker along with rinse water from pipet.
3. Dry, cool and weigh as previously described. (Record as weight of clay and dispersing agent).
4. CORRECTION FACTOR: To allow for the weight of dispersing agent in the sample, dilute 10 mL of dispersing agent to 1000 mL in a graduated cylinder. Remove 25 mL of the liquid and dry, cool and weigh as with the silt and clay fractions (D.A. = dispersing agent)

CALCULATIONS

Wt. of silt & clay in aliquot (corrected) = Wt of silt & clay & D.A. – Wt. of D.A.

Wt. of clay in aliquot (corrected) = Wt of clay & D.A. – Wt. of D.A.

Total clay in sample = Wt. of clay in aliquot (corrected) x 40

Total silt in sample = [Wt. of silt & clay (corrected) – Wt. of clay (corrected)] x 40

Total sand in sample = Wt of sand fraction

Recovered Wt. of sample = Total Sand (S) + Total Silt (Si) + Total Clay ©

$$\%S = \frac{\text{Total S x 100}}{\text{Recovered Wt. of sample (g)}}$$

$$\%\ SI = \frac{\text{Total Si x 100}}{}$$

Recovered Wt. of sample (g)

$$\%C = \frac{\text{Total C} \times 100}{\text{Recovered Wt. of sample (g)}}$$

ERROR VALUES

	n	$\overline{X}$	SD	RSD (%)
S (%)	7	51.0	1.4	2.8
Si (%)	7	26.6	1.1	4.3
C (%)	7	22.4	1.6	7.2

Rate of analysis: 40-50/week

REFERENCES

1) CLARK, J.S. 1962. Notes on a pipetting assembly for the mechanical analysis of soils. Can. J. Soil Sci. 42: 316.

2) KILMER, V.J. and ALEXANDER, L.T. 1949. Methods of making mechanical analyses of soils. Soil Sci, 68: 15-24.

3) TOOGOOD, J.A. and PETERS, T.W. 1953. Comparison of methods of mechanical analyses of soils. Can. J. Agric. Sci. 33: 139-171.

4) TYNER, E.H. 1939. The use of sodium metaphosphate for dispersion of soils for mechanical analysis. Soil Sci. Soc. Amer. Proc. 4: 106-113.

DETERMINATION OF ATTERBERG LIMITS

A) LIQUID LIMIT (D423-66)

The liquid limit of a soil is the water content (expressed as a % of the weight of the oven-dry soil) at the boundary between the liquid and plastic states. This boundary is arbitrarily defined as the water content at which two halves of a soil cake will flow together over a distance of ½ inch (13 mm) along the bottom of a groove separating the two halves, when the cup is dropped 25 times for a distance of 1 dm at the rate of 2 drops per second.

EQUIPMENT

1. Place about 100 g of air-dry soil that has passed a No. 40 mesh (.42 mm opening) sieve into a 250 mL beaker.

   NOTE: Prepare the No. 40 mesh-sized sample from 2 mm soil by dry sieving and crushing aggregates but not primary particles.
2. Mix thoroughly with distilled water while stirring, kneading and chopping with a spatula.
3. Make further additions of water (few mL at a time) and mix thoroughly until the consistency has reached a point at which the mixture is almost liquid.
4. Place a portion of the mixture in the cup of the Liquid Limit Device. Squeeze the sample down and spread evenly with as few strokes of the spatula as possible.

Level the soil and trim it to a maximum thickness of 1 cm. Return the excess soil to the beaker.

5. Divide the soil in the cup with firm strokes of the grooving tool to form a clean, sharp groove of proper dimensions.

   NOTE: Up to six strokes may be used to make the groove, each one penetrating a little deeper until the last stroke scrapes the bottom of the cup clean. Make the groove with as few strokes as possible.

6. Operate the Liquid Limit Device so that the cup drops at a rate of 2 per second, until the two halves of the soil cake come in contact at the bottom of the groove along a distance of about ½ inch (13 mm). Record the number of drops required to close the groove.

7. Remove a slice of soil with the spatula at the right angles to the groove, including a portion where the groove has closed. Place the sample into a tared moisture can, weigh and oven-dry at 105^{o} C. Record weights to two decimal places.

8. Cool sample in dessicator and weigh. Calculate the water content of the soil as follows:

$$\frac{Ww - Wod}{Wod} \times 100$$

   Ww = weight of moist sample

   Wod = weight of oven-dry sample

9. Transfer the soil remaining in the cup to the 250mL beaker. Detach, wash and dry the cup and grooving tool; then re-attach the cup to the Liquid Limit Device.

10. Repeat steps 3-9 to obtain 2 or 3 readings between 15 and 35 crops.

NOTE: Always proceed from drier to wetter soil.

11. Plot a graph of water content (%) vs. number of drops on semilog paper, with water content (%) on the arithmetic scale. Draw a straight line through the plotted points.

12. Read the liquid limit as the water content (%) corresponding to the point of intersection of the 25 drops ordinate with the line plotted in step 11. Record the liquid limit as the nearest whole number.

B. PLASTIC LIMIT (D424-54T)

The plastic limit of a soil is the water content (expressed as a % of the oven-dry soil) at the boundary between plastic and semi-solid states. It is the lowest water content at which the soil can be rolled into threads 1/8 in. (3 mm) in diameter without the thread breaking into pieces.

EQUIPMENT

Evaporating dish

Ground glass plate

Moisture cans

Spatula

PROCEDURE

1. Place about 15 g of air-dry soil that has passed a No. 40 mesh (0.42 mm opening sieve. into an evaporating dish.

   NOTE: Prepare the No. 40 mesh-sized sample from 2 mm soil by dry sieving and crushing aggregates but not primary particles.

2. Mix thoroughly with distilled water until the soil becomes plastic enough to be shaped into a ball.

   NOTE: Alternatively a portion of the soil material prepared for the liquid limit test may be used.

3. Squeeze and form the sample into an ellipsoidal shape and roll the soil between the fingers and a ground glass plate with just enough pressure to form a thread of uniform diameter throughout its length.

   NOTE: The rate of rolling should be 80-90 strokes per minutes (a stroke is 1 complete back and forth motion).

4. When the diameter of the thread reaches about 3 mm, break the thread into 6 to 8 pieces. Squeeze the pieces together between the thumbs and fingers and form a uniform ellipsoidal shape and re-roll as before.

5. Repeat step 4 until the thread crumbles under the pressure required for rolling and the soil can no longer be rolled into a 3 mm strand.

6. Collect the pieces of crumbled soil, place them in a tared moisture container, weigh and oven-dry at 105^{o} C for 24 hours. Record weight to 2 decimal places.

7. Cool sample in dessicator and weigh as before.

8. Calculate the plastic limit as the water content in %, to the nearest whole number.

$$\text{PLASTIC LIMIT} = \frac{Ww - Wod}{Wod} \times 100$$

Ww = weight of moist sample

Wod = weight of oven-dry sample

C. PLASTICITY INDEX

The plasticity index of a soil is the difference between its liquid and plastic limits.

PLASTICITY INDEX (PI) = LIQUID LIMIT (LL) - PLASTIC LIMIT (PL)

Under certain conditions, the plasticity index cannot be calculated and is reported as NON-PLASTIC (NP):

a) when either the LL or PL cannot be determined

b) when the PL is equal to or greater than the LL.

- when the soil is extremely sandy, the PL test should be made before the LL.
- if the PL cannot be determined, report both PL and LL as NP.

Rate of analysis: 2/day for LL, PL and PI.

REFERENCES

1. American Society for Testing and Materials. 1971. The Annual Book of ASTM Standards. Part II, 225-232.

SHRINKAGE FACTORS OF SOIL (D427-39)

This method is intended for obtaining data from which the following sub-grade soil constants may be calculated: shrinkage limit, shrinkage ratio, volumetric change and lineal shrinkage.

EQUIPMENT

Evaporating dish (approx. 11.5 cm in diameter)

Shrinkage dish (circular porcelain dish with flat bottom, approx. 4.5 cm in diameter and 1.2 cm in height)

Plastic plate – rigid plate with 3 metal prongs for immersing the soil cake in mercury (as in Fig.1).

Spatula and straightedge

Silicon lubricant or petroleum jelly

Glass cup – about 5 cm in diameter and about 2.5 cm in height

REAGENT

Mercury (Hg)

PROCEDURE

1. Place about 30 g of air-dried soil that has passed a No. 40 (.42 mm opening) sieve into an evaporating dish.
2. Add distilled water and mix thoroughly to fill the soil voids completely with water and to make the soil moist enough to be readily worked into the shrinkage dish.

NOTE: The desired consistency is equal to or slightly greater than the liquid limit.

3. Coat the inside of the shrinkage dish with a thin layer of lubricant to prevent the adhesion of soil to the dish.

FIGURE 1. Apparatus for Determining the Volumetric Change of Subgrade Soils

4. Place an amount of wetted soil equal to about 1/3 of the dish's volume in its centre and allow soil to flow to the edge of the dish by tapping it on a firm surface.

5. Repeat step 4 until the dish is completely full, compacted, and all included air has been brought to the surface.

6. Remove excess soil with a straightedge, clean the outside of the dish and record the weight of the dish + wet soil (Ww).

7. Air-dry sample in dish for 12-24 hours, then oven-dry at 105^{o} C for 24 hours; cool in dessicator and weigh dish + dry soil (Ws).

8. Determine the volume of the dry soil cake by removing it from the shrinkage dish and immersing it in mercury in the following manner:

   i) Fill the glass cup with mercury (Fig. 1)

   ii) Remove excess mercury by pressing the plastic plate with prongs firmly over the edge of the cup - carefully wipe off any mercury adhering to the side of the cup.

   iii) Place the cup into a clean evaporating dish.

   iv) Place the soil cake on the surface of the mercury and force it down into the mercury with the plastic plate.

   NOTE: it is essential that no air be trapped under the soil cake.

   v) Measure the volume of mercury displaced and record as volume of dry soil (Vd).

   ALTERNATIVELY the volume of mercury (equivalent to soil volume) can be determined by weighing the mercury displaced and dividing the weight

by the density of mercury.

$$V = \frac{W}{D}$$

W = weight of mercury

D = density of mercury

V = volume displaced

9. Weigh the empty shrinkage dish (Wd).

10. Determine the volume of the empty shrinkage dish (equivalent to volume of wet soil cake).

- fill the dish with mercury and record as volume of wet soil cake (Vw).

CALCULATIONS

I Moisture content of soil when placed in dish (M)

$$M = \frac{(Ww - Ws)}{(Ws - Wd)} \times 100$$

Ww = weight of dish + wet soil

Ws = weight of dish + dry soil

Wd = weight of dish

II Shrinkage limit (S)

The shrinkage limit of a soil is the moisture content (expressed as a % of the weight of oven-dried soil) at which the volume of the soil mass will be affected by an increase in moisture content, but not by a decrease.

$$S = \frac{[\,(Vw - Vd)\,]}{[\,(Ws - Wd)\,]}$$

Vw = volume of wet soil cake

Vd = volume of dry soil cake

III Shrinkage ratio (R)

The shrinkage ratio of a soil is the ratio between a given volume change (expressed as a % of the dry volume) and the corresponding change in moisture content above the shrinkage limit (expressed as a % of the weight of the oven-dried soil).

$$R = \frac{(Ws - Wd)}{Vd}$$

IV Volumetric change (Cf)

The volumetric change of a soil for a given moisture content is the volume change (expressed as a % of the dry volume) of the soil mass when the moisture content is reduced from the stipulated % to the shrinkage limit.

$$Cf = (M - S)R$$

M = moisture content of soil when placed in dish

S = shrinkage limit

R = shrinkage ratio

V. Lineal shrinkage (LS)

The lineal shrinkage of a soil for a given moisture content is the decrease in one dimension of the soil mass (expressed as a % of the original dimension), when soil moisture content is reduced from an amount equal to the field moisture equivalent to the shrinkage limit.

NOTE: Moisture content of the soil when placed in the dish (M) is used instead of field moisture equivalent in the lineal shrinkage calculation.

Insert Formula

Rate of analysis: 15/20 / day

REFERENCES

1. Procedures for testing soils. Standard Method of Test for Shrinkage Factors for Soils. pp. 76-79. 1958. Published by the American Society for Testing Materials. (ASTM)

LABORATORY MEASUREMENT OF HYDRAULIC CONDUCTIVITY OF SATURATED SOIL (Constant-Head Method)

Soil samples with either disturbed or undisturbed structure are usually held in metal or plastic cylinders so that one-dimensional flow can be obtained.

The constant-head system is best suited to samples with conductivities greater than approximately 0.01 cm/min (0.6 cm/hr), while the falling-head system is best suited to samples with lower conductivity.

EQUIPMENT

An apparatus for maintaining a constant head (Fig. 1).

Metal cylinders

Cylindrical sample holders, plastic

Funnels

Beakers

Graduated cylinders

Thermometer

REAGENTS

Paraffin

Supply of de-aerated water

PROCEDURES

1. Place a core sample, trimmed flush with the metal cylinder at both ends, into a cylindrical sample holder and clamp tightly.

2. Use paraffin to seal the outside area of contact between the soil core and the sample holder.
3. Place the sample holder into a container filled with water to a depth just below the top of the sample (de-aerated water is preferred).
4. Allow the soil core to soak for at least 16 hours or until saturated.

Insert Figure 1 Constant Head Apparatus

5. Transfer the sample holder to a constant-head apparatus and start the siphon to maintain a constant head of water above the sample.

NOTE: Do not allow the water to drain from the top of the sample.

6. After the water level on top of the sample has become stabilized, collect the percolate in a beaker.

7. Measure the volume of percolate (Q) obtained from the sample in a known time interval (t).

8. Also measure the hydraulic head difference the cross-sectional area of the core (A) and the temperature of the water and use to calculate the hydraulic conductivity (K).

CALCULATIONS

$$K \text{ (cm/hr)} = \frac{Q\,L}{A(\Delta H)t}$$

K = hydraulic conductivity (cm/ hr)

Q = volume of percolate (cm^3)

L = height of core (cm)

H = hydraulic head difference (cm)

A = cross sectional area of core (cm^2)

t = time required to collect percolate (hours)

NOTE: The conductivity will be affected by the temperature of the water. The result may be corrected to a standard temperature by use of

the relation:

$$K_{ST} = K_T \frac{N_T}{N_{ST}}$$

K_{ST} = conductivity at standard temp.

K_T = conductivity at measured temp.

N_T = viscosity at measured temp.

N_{ST} = viscosity at standard temp.

ADDITIONAL NOTES

1. Gas bubbles entrapped in the soil pores may produce erratic results. If tap water is used the amount of entrapped gas may increase; it is therefore preferable to use air-free water. Small quantities of water may be de-aerated by boiling or by vacuum. Distilled water is not recommended for measurement of hydraulic conductivity.
2. Because of swelling clays and slaking of aggregates, soil that has been air-dried will undergo changes in structure when it is rewetted. To prevent such changes, soil cores should not be allowed to dry after sampling.
3. Leakage along the interfaces between the soil core and the sample container may be a source of error and must be eliminated (for satisfactory results).
4. Undisturbed cores with cracks and worm holes are unsuitable for the purpose of evaluation of conductivity.

5. Fragmented and repacked samples are useful in comparative studies and in dealing with soils where structure is of relatively little importance.

6. It is suggested that at least 3 cores of a given sample be used to obtain a reliable measure of hydraulic conductivity.

7. A conversion from conductivity to permeability (k) can be made by the use of the equation:

$$k = \frac{Kn}{pg}$$

k = permeability

K = conductivity

n = fluid viscosity

p = fluid density

g = gravitational constant

REFERENCES

1. KLUTE, A. Laboratory Measurement of Hydraulic Conductivity of Saturated Soil IN Agronomy No. 9. Methods of Soil Analysis, Part 1, Black, C.A., ed., pp. 210-221.

DETERMINATION OF SOIL WATER CONTENT AT 15 BARS

The moisture content (%) of soils at 15 bars has become a well known and widely used characteristic that can be correlated to a permanent wilting point (PWP) of soils. (1)

PWP = 0.0207 + .77468 x (15 bar % moisture).

EQUIPMENT

Pressure membrane extractor (0-30 bar)

Tank of nitrogen and regulator

Cellulose membrane (ave. pore radius of 24 Å and an air entry value greater than 100 bars.)

Soil sample containers (6 cm diam., 1 cm high, with cloth mesh base)

100 mL beakers

Drying oven

PROCEDURE

1. Prepare 2 mm size air-dried samples, in duplicate, and place into soil containers. (15-20 g for clayey soils, 25-30 for sandy soils).
2. Pre-wet samples and cellulose membrane with an excess of water in a large try for several hours or overnight.
3. Place cellulose membrane, and then samples, into a pressure extractor.
4. Close the extractor and connect a buret to the outflow tube in the screen drain plate.

5. Gradually apply pressure to a level of 15 bars.

   NOTE: Water will immediately start flowing from the extractor into the buret.

   NOTE: 15 bars is a measure of pressure roughly equivalent to 1500 Kpa, 220 psi, or 15 atm (1 bar = .9869 atm.)

6. After a day, apply a 4 psi pressure differential to the rubber diaphragm above the sample. (Some extractors may not have this feature).

   NOTE: A pressure differential is applied to provide and maintain good contact between the sample and the cellulose membrane.

7. Record the level in the buret periodically; when the level in the buret has ceased to change over a period of a day, equilibrium has been attained.

   NOTE: The time required to reach equilibrium will vary from 5 to 10 days depending on the texture of the soil samples.

8. Release pressure, remove samples and transfer the soil into tared 100 mL beakers.

9. Weigh and oven-dry at 105^{o} C for 24 hours, cool samples in dessicator and weigh again. Record weight to 2 decimal places.

CALCULATIONS

$$\% \text{ MOISTURE @ 15 bars} = \frac{Ww - Wod}{Wod} \times 100$$

Ww = weight of soil sample at 15 bars

Wod = weight of soil sample, oven-dry

ERROR VALUES:

n	$\overline{X}$	SD	RSD (%)
5	3.6	0.2	6.3

Rate of analysis: 20 samples per set; 1 set / 2 weeks.

REFERENCES:

1. SHAYKEWICH, C.F. 1965. The relationship between soil components and soil physical constants of some Manitoba soils. M.Sc. Thesis, University of Manitoba, Winnipeg, Manitoba.
2. Soil Moisture Equipment Corp., Technical Notes on Equipment. P.O. Box 30025, Santa Barbara, Ca. 893105 U.S.A.

X-RAY DIFFRACTION FOR MINERAL IDENTIFICATION AND MINERALOGICAL COMPOSITION

X-ray diffraction analysis has become a useful method for mineralogical characterization of layer-silicate species in soils. Pretreatment of samples and subsequent fractionation into particle size classes is necessary for improving results.

Qualitative interpretation of diffraction patterns involves identification of crystalline species from the array of diffraction maxima obtained from a sample. Identification may be accomplished by measurement of diffraction spacings and comparison of these spacings with known spacings of standard minerals.

Quantitative analysis of minerals by x-ray diffraction can be reliable, but variations in chemical composition, crystal perfection and amorphous substances are difficult to evaluate and compensate for in analysis of soil or clay samples. In most cases, estimation of mineral percentages from x-ray diffraction patterns is only semiquantitative. For more reliable and accurate estimation, the use of x-ray diffraction analysis in conjunction with other methods, such as differential-thermal, integral-thermal, infrared, surface area, and other elemental analysis, is advisable.

EQUIPMENT

Centrifuge

Supercentrifuge and peristaltic pump

X-ray Diffractometer with chart recorder (Philips)

Electric stirrer (Hamilton-Beach "milkshake" type) with containers

Hot plate

Hot water bath

Beakers - 50, 100 and 400 mL

Centrifuge bottles, 250 mL

Centrifuge tubes, 15 mL

REAGENTS

Citrate bicarbonate buffer

- dissolve 125 mL of 1M Sodium bicarbonate ($HaHCO_3$) @ 84 g/L, to each litre of 0.3M Sodium citrate ($Na_3C_6H_5O_7$ 2 H_2O) @ 88.4 g/L

Sodium carbonate (Na_2CO_3)

- 2% solution: 20 g Na_2CO_3 per litre
- dilute solution: dissolve 1 g Na_2CO_3 in 9 L of distilled water.

Sodium acetate (NaOAc) 1N, neutral and pH 5 *

- dissolve 27 mL of Acetic acid, glacial (HOAc) and 92 g NaOAc per L.

Glycerol-alcohol mixture

- dissolve 10 mL of Glycerol ($CH_2OHCHOHCO_2OH$) in 90 mL of Isopropyl alcohol.

Sodium dithionite powder ($Na_2S_2O_4$)

Hydrogen peroxide (H_2O_2), 35%

Sodium chloride (NaCl) 1N and saturated solutions

Magnesium chloride ($MgCl_2$) 1N and 1ON

Magnesium acetate (Mg $[OAc]_2$) 1N

Potassium chloride (KCl) 1N and 4N

Potassium acetate (KOAc) 1N

Isopropyl alcohol ($CH_3CHOHCH_3$), 50% and 99%

PROCEDURE

I PRETREATMENT OF SAMPLES

A. Removal of Carbonates

1. Weigh a 10 g sample (more if clay content is low) into a 250 mL centrifuge bottle.
2. Treat with a 150 mL of 1N NaOAc buffer (pH 5) in a boiling water bath, with intermittent stirring, for at least 30 minutes.
3. Centrifuge, decant and save supernatant for analysis.

   NOTE: Repeat steps 2 + 3 for highly calcareous samples.
4. After treatment wash twice with 1N NaOAc buffer (pH 5).

B. Destruction of Organic Matter and Removal of Divalent Cations

1. Transfer the carbonate-free sample to a 400 mL beaker and treat with 35% H_2O_2 until organic matter is destroyed.
2. Boil to remove any excess H_2O_2 .
3. Transfer sample back to centrifuge bottle, wash 3 times with 100 mL of 1N NaOAc (pH 5), then twice with 100 mL of neutral IN NaOAc to remove divalent cations.

C. Removal of Fe Oxides and Na-saturation

1. Add 150 mL of Citrate-bicarbonate buffer to the sample in the centrifuge bottle.
2. Place in a water bath at 70-80^{o} C, add 3 g of Sodium dithionite ($Na_2S_2O_4$) and mix well.

   NOTE: A water bath temperature of over 80^{o} C may result in the formation of Iron sulfide (FeS) as a black precipitate.
3. Stir intermittently over a period of 20 minutes.
4. Add 10 mL of saturated NaCl, centrifuge and decant, saving the supernatant if chemical analysis is desired.
5. Wash 4 times with 100 mL 1N NaCl, combining the first 2 washes with the supernatant if chemical analysis is to be performed.
6. Wash once with 100 mL of 99% Isopropyl alcohol to remove the bulk of the NaCl.

D. Removal of Amorphous Material

1. Transfer sample from (C) to a "milkshake container" with 200 mL of 2% Na_2CO_3 and mix for 3 minutes with an electric stirrer.
2. Cover the container and boil for 5 minutes on a hot plate.
3. Transfer back to the centrifuge bottle with a minimum volume of 2% Na_2CO_3 and centrifuge until supernatant is clear. Discard supernatant unless Si and Al analyses are required.
4. Resuspend the sample in 100 mL of H_2O.

II. FRACTIONATION OF SAMPLES

A. Separation of 53u - wet sieve using 270 mesh sieve (page 6?????)

B. Separation at 5u

1. Bring the level of the pretreated sample suspension to 10.9 cm from the bottom of the 250 mL centrifuge bottle with dilute Na_2CO_3 and mix well.

2. Centrifuge at 300 RPM for 5.02 minutes, using an IEC centrifuge with a 242 head (length of fall is 8.3 cm).

NOTE: Time of centrifugation depends on the distance from the centre of the centrifuge to the top of the suspension (S) and to the top of the sediment (R). Centrifugation time is given by the expression:

$$t(min) = \frac{63.0 \times 10^8 n \log 10(R/S)}{(Nm)^2 (Du)^2 (\square S)}$$

Nm = RPM

Du = particle diameter in microns

ΔS = difference in specific gravity between solvated particle and suspension liquid

n = viscosity

For 250 mL centrifuge bottles and 242 head:

R= 15.55 cm, S= 7.24 cm

3. Draw off the supernatant to the proper depth and collect it as "material less than 5u".

4. Wash the sample repeatedly with dilute Na_2CO_3 and repeat the centrifugation until the supernatant is essentially clean.

   NOTE: Usually, 6-8 washings should suffice.

C. Separation at 2u

1. Centrifuge the “less than 5u” suspension step by step at 700 RPM for 5.77 minutes, as above, until all the suspension has been transferred.
2. Wash the sediment with dilute Na_2CO_3 repeatedly, with centrifugation and collection of supernatant until it becomes as clear as possible.

   NOTE: In both the 5u and 2u separations, the supernatant does not become absolutely clear, but nearly so. Four to six washings will suffice in the 2u separation.
3. Collect the sediment as fine silt (2-5u in diameter).

D. Separation at 0.2u

1. Put the “less than 2u” suspension through a supercentrifuge running at 25 000 RPM with a flow rate into the bowl of 415 mL/min.

   NOTE: A constant flow rate to the supercentrifuge can be provided by a peristaltic pump.
2. After all the suspension is processed, put 200 - 250 mL of distilled water through to displace the suspension still in the centrifuge bowl (approximately 200 mL).
3. Collect the sediment deposited on the inner surface of the bowl as coarse clay (0.2-2u in diameter).

4. Treat the effluent suspension with 15 g NaCl to cause flocculation.

5. Draw off the clear supernatant and wash the sediment, initially with about 100 mL of 50% Isopropyl alcohol and then repeatedly with 100 mL of 99% Isopropyl alcohol, until all NaCl is removed (as evidenced by a negative result to testing for chlorides with $AgNO_3$). Collect the sediment as fine clay (less than 0.2u in diameter).

   NOTE: An alternative procedure for separating coarse and fine clay involves normal centrifugation as previously described. Using 250 mL bottles and a 242 head and centrifuging at 2000 RPM for approximately 78.4 minutes, separation at 0.2u may be achieved. Though the procedure is very time consuming, it is more quantitative than the supercentrifuge method.

III. SAMPLE PREPARATION AND X-RAY ANALYSIS

A. Suspension Concentrations

1. Take 5 mL aliquots of the fine silt (Fsi), coarse clay (CC) and find clay (FC) suspensions.
2. Wash Fsi and CC aliquots with about 50 mL of 0.05N HCl to remove Na_2CO_3.
3. Oven-dry aliquots, weigh out and calculate suspension concentrations and aliquots required to provide 50 mg samples.

B. Pretreatment with Slightly Acidic Buffer (1N NaOAc, pH 5)

1. Take duplicate aliquots containing 50 mg of each fraction of each sample and place in 50 mL beakers.
2. Add 10 mL of 1N NaOAc (pH 5) to each sample and boil for 5 minutes.

C. Mg-Saturation and Glycerol Solvation

1. Take one set of samples from (B) and flocculate each sample with 3 mL of 10N $MgCl_2$.
2. Place samples in 15 mL centrifuge tubes, wash once with 10mL of 1N $MgCl_2$
3. Wash once with 10 mL of 50% Isopropyl alcohol, then with 10 mL volumes of 99% Isopropyl alcohol until all salt is removed (negative chloride test).
4. Add Glycerol (as a 10% v/v solution in alcohol) to each Mg-saturated sample. Use 0.11 mL for Fsi, 0.22 mL for CC and 0.7 mL for FC.

   NOTE: Use less Glycerol for FC samples low in montmorillonite.

D. K-Saturation

1. Proceed with the other set of samples as described in “C” except:
   - flocculate with 10 mL of 4N Kcl.
   - use Kcl and KOAc instead of $MgCl_2$ and Mg $(Oac)_2$.
   - no Glycerol solvation.

E. Slide Preparation

1. Add to the samples volumes of liquid equal to their sediment volume (alcohol for Mg-saturated sample, water for K-saturated samples).

2. Mix samples well and pipet material onto 27 mm x 46 mm glass slides.

3. Label each slide with sample, fraction and saturating cation.

   NOTE: For labelling slides, the use of heat-resistant ink such as TECH-PEN is recommended, since temperatures up to 550^{o} C are involved.

F. X-ray Diffraction Analysis

1. X-ray the Mg and K slides using an x-ray diffractometer scanning the range 20 = 2^{o} to 36 ^{o}.

2. After x-raying the air-dried K slide, heat the slide to 300^{o} C for 1 hour, cool in a dessicator and x-ray again.

3. Heat the same slide to 550 ^{o} C for 1 hour, cool and repeat the above analysis.

G. Qualitative Interpretation of X-Ray Diffractograms

D-SPACING (Å)	ORDER *	MINERAL
32	1	Alternating montmorillonite and chlorite (or vermiculite)
28	1	Alternating montmorillonite – mica or chlorite-vermiculite
24	1	Alternating vermiculite – mica
19.2-19.5		Random mixture 10 + 17.7 Åm etc,
17.7	1	Montmorillonite, glycerol solvated
17.2	1/1	Random 50:50, 10 + 17.7 Å
14.2	1	Chlorite or vermiculite
13.3	1/1	Random 50:50, 10 + 14 Å
10.0	1	illite, micas
9.5	½	Random 60:40, 10:17.7 Å
9.2	½	Random 40:60, 10:17.7 Å
8.85	2	Montmorillonite
8.40-8.48	1	Amphiboles
7.2	1	Kaolinite
7.0-7.2	2	Chlorite (or vermiculite)
5.0	2	Muscovite
4.7-4.9	3	Chlorite
4.6	3	Vermiculite
4.21		Quartz
4.05		Cristobalite
3.54	5	Montmorillonite, glycerol solvated
3.52-3.58	4	Chlorite
3.50-3.55	4	Vermiculite
3.35		Quartz
3.33	3	Muscovite, illite
3.21-3.28		K-feldspar
3.12-3.23		Plagioclase feldspars

* - where two numbers are given, 1st refers to first mineral, 2nd to other.

NOTES:

- 9.5 Å and 9.2 Å peaks can, if diffraction peaks are sufficiently broad, be created by partial overlapping of 10 Å and 8.85 Å peaks.
- montmorillonite (17.7 Å) and vermiculite (14.2 Å) collapse to 10 Å spacings on K-saturation and heating to 300^{o} C: chlorite (14.2Å) does not.

- chlorite (14.2 Å) peak is enhanced at 550^{o} C.
- kaolinite peaks disappear at 550^{o} C (crystal structure destroyed).

H. Semi-quantitative Interpretation

1. Select a diagnostic peak for each mineral to be identified.
2. Run standard samples of minerals, using size fractions and treatments as with regular analysis. Note intensities of diagnostic peaks.
3. Compute factors for diagnostic peaks so that peak intensities may be directly compared to each other. (If the intensity of the diagnostic peak of mineral A is twice that of mineral B, divide the intensity of the mineral A peak by two.)
4. Measure diagnostic peak intensities from diffractogram.
5. Adjust these to a common basis, using factors described in (3).
6. Add corrected intensities and calculate percentages.

Rate of analysis:
- pretreatment, 1 week.
- fractionation, 1 week.
- x-ray preparation, 4 days.
- x-ray, 8 days @ 12/day
- interpretation, 1-2 days.
- TOTAL - 5 weeks per set of 8.

REFERENCES

1. JACKSON, M.L. 1956. Soil Chemical Analysis – Advanced Course. University of Wisconsin. pp. 30-75.

2. McKEAGUE, J.A. 1978. Manual on Soil Sampling and Methods of Analysis, 2nd edition, Canadian Society of Soil Science, pp. 189-204.

3. WHITTIG, L.D. 1965. X-ray Diffraction Techniques for Mineral Identification and Mineralogical Composition IN Agronomy No. 9, Methods of Soil Analysis, Part 1, Black, C.A. ed., pp. 671-698.

MEASUREMENT OF SOl pH

1. Place 15 grams of dried sample in a 50mL plastic beaker. Record the sample number on the pH sheet beside the corresponding beaker number.
2. Take the samples to Room 339. Add 30 mL of 0.01 M $CaCl_2$ to the beakers and stir the solution very well. Wait 20 minutes.
3. Stir the solution very well again and wait 30 minutes.
4. White you are waiting you can calibrate the pH meter.
   a) Select “pH” from the START screen.
   b) Select “STD” and press “CLEAR”.
   c) Remove the cap from the pH electrode and rinse it very well with distilled water.
   d) Fill the calibration bottles with the appropriate buffers.
   e) Place the electrode in the bottle filled with pH 4 buffer.
   f) Press “STD”, wait till it stabilizes and is near the correct pH then press “STD” again to accept the value.
   g) Remove the electrode from the solution and rinse it very well with distilled H_20.
   h) Repeat e, f, g for pH 7 and pH 10.
   i) After the machine is calibrated place the electrode in pH 4 until you are ready to measure your samples.

   USE FRESH pH SOLUTIONS EVERY TIME YOU CALIBRATE.
5. After 30 minutes have passed, remove the electrode from the buffer and rinse it.

*Place the electrode in the solution so as the eye is covered, but soil does not touch it. Be careful not to disturb the soil.

*Repeat starting here (Step 7).

6, Press “MEAS” and wait for the reading to stabilize. Once it stabilizes hit “MEAS” again to see if the number changed. If no change then record the value on the pH sheet in the corresponding spot. Remove the electrode from the solution and rinse the electrode.

7. For the remaining samples repeat Step 5 and 6.

8. When all the samples are done, press “MODE” and rinse the electrode. Place 2 drops of the pH 4 buffer in the cap and place the cap on the electrode.

*IF your sample continues to change in its value then carefully move the beaker around (side to side) and press “MEAS”. Continue until it stabilizes.

*Personally, I like to obtain 3 identical readings before accepting a value.

Soil pH is a measure of the activity of ionized H in the soil solution. The pH of a solution is defined as the negative logarithm to the base 10 of the H ion activity.

Soil pH is an indicative measurement of the chemical properties of a soil. A strong relationship exists between soil pH and the solubility of various compounds, the relative exchange capacity and the activity of micro-organisms.

Measurement of soil pH in water, saturated pastes, and solutions of $CaCl_2$ is mot satisfactory for non-saline soils (1). Measurements made with glass electrodes/saturated calomel cells yield accurate, reproducible values which are largely

independent of the soil-solution ratio, and errors due to the liquid junction potential are minimized because the soil suspensions are flocculated.

EQUIPMENT

pH meter equipped with glass and calomel electrodes

50 mL plastic beakers

Stirring rods

REAGENTS

Calcium chloride solution ($CaCl_2$) 0.01M

Standard buffer solutions: pH 4.0, 7.0

PROCEDURE

1. Measure out 15 g of mineral soil into a 50mL beaker with a graduated scoop. Add 30 mL of 0.01M $CaCl_2$ and stir into suspension. Stir again after 15-20 minutes and allow to stand for 30 minutes to allow sediment to settle.

   NOTE: For organic samples a 1:4 soil-solution ratio is used, as organic soil tends to absorb more solution.

2. Standardize the pH meter as follows:

   i) set the pH meter at pH 7.0 with standard buffer solution of pH 7.0 and set the temperature compensator at the temperature of the buffer.

   ii) check the meter with a pH 4.0 buffer solution and adjust if necessary.

3. Immerse the glass and calomel electrodes into the partly settled suspension (do not immerse electrodes to the bottom of the container) and record pH when reading has been stabilized.

ERROR VALUE

n	$\bar{X}$	SD
10	6.0	±.10

Rate of analysis: 80-100/day.

REFERENCES

1. SCHOFIELD, R.K. and TAYLOR, A.W. 1955. The measurement of soil pH. Soil Sci. Soc. Proc. 19: 164-167.

DETERMINATION OF ELECTRICAL CONDUCTIVITY AND % SATURATION

Electrical Conductivity Procedure

1. Place 250 grams of DRIED SOIL into a plastic 400 mL beaker

   OR

   Fill the plastic 400 mL beaker ¾ of the way full with wet soil.

2. Record the sample number and beaker number on the Moisture Data Sheet.

3. Add DISTILLED H_2O to the soil while mixing. You want the soil to glisten, have little clumps, slide off the spatula (used for mixing) and flow slightly when beaker is tipped.

4. Once the sample reaches that stage cover the beaker with parafilm and let it sit overnight.

5. The next day check whether or not there is free water collected on the surface.

   IF there is, then mix in more soil.

   IF no free water has collected check to see if the soil still behaves like it did for Step 3.

   IF it does not then add more water and mix it. Let it stand for 4 hours.

   IF it does move on to the next step.

6. Weigh 50 grams of the paste in a 50mL beaker. Record the initial weight of the empty beaker and the final weight of the beaker and paste on the Moisture Data Sheet. Place the beaker and paste in the oven and leave it to dry overnight.

7. Take a conductivity test tube and place it in the Erlenmeyer flask that is attached to the vacuum. Place a rubber seal on top of the flask then place a Buchner

funnel on top of the flask and seal. Place a filter paper in the funnel and wet it slightly with distilled water.

NOTE: Record the number of test tube on the Moisture Data Sheet.

NOTE: If you have a large amount of soil use a large Buchner funnel so that the process of collecting water goes faster.

8. Add the paste to the centre of the funnel and spread it around evenly. Turn the vacuum on.

   Make sure a seal is formed. You should press the funnel onto the flask. You can check that a seal formed by lifting the funnel - if it remains attached to the flask a good seal has formed.

9. Turn the vacuum off once enough liquid has entered the test tube (up to the BLACK line). Carefully take apart the apparatus and dispose of the soil. Take the collected liquid over to the conductivity meter and measure its electrical conductivity. Record.

10. From Step. 6…….after the samples have dried weigh them and record their weights. Place them in the computer to obtain the % moisture content.

    Let samples cool before weighing the samples. You don't need to cool them in the dessicator.

    Measurement of electrical conductivity (EC) of soil water extracts obtained from field water content is not practical for routine purposes. Because the absolute and relative amounts of solutes are influenced by the soil/water ratio at which the extract is made, the ratio must be standardized to obtain results that can be applied and interpreted universally. Soil salinity is conventionally defined and

measured on aqueous extracts of saturated soil pastes, which is the preferred method as it gives a closer approximation of the salinity in the natural soil solution than does analysis of extracts of soil/water of 1:2, 1:5, etc. (1)

EQUIPMENT

400 mL plastic beakers

Spatulas (stainless)

Vacuum pump

Vacuum manifold with filter flasks, Buchner funnels, filter paper (Whatman No. 1)

500 mL beakers

Test tubes

Conductivity bridge and cell (YSI Model 32 conductance meter)

REAGENT

1. Place 200-300 g of soil into a 400 mL plastic beaker:

   NOTE: Soil samples should not be oven-dried before extracting for determination of soluble salts, because heating to 105□ C converts at least part of the gypsum ($CaSO_4.2\ H_2O$) to plaster of paris ($CaSO_4\ .\ ½\ H_2O$). The latter hydrate has a higher solubility in water than does the former.

2. Add distilled water to the soil while stirring until it is nearly saturated. Allow the sample to stand for several hours to permit the soil to imbibe the water, and then add more water to achieve a saturated paste. At saturation the soil pate glistens and reflects light, flows slightly when the container is tipped, slides freely and

cleanly off a spatula, and consolidates easily by tapping or jarring the container after trench is formed in the paste with the side of a spatula. After mixing, allow the sample to stand (preferably overnight, but at least 4 hours) and then recheck the criteria for saturation. Free water should not collect on the surface, nor should the paste stiffen markedly or lose its glisten. If the paste is too wet, add additional soil to mixture.

NOTE: With fine-textured soils, enough water should be added immediately, with a minimum of mixing, to bring the sample nearly to saturation. This minimizes the formation of clumps of soil during stirring, speeds the mixing process, and helps attain a more definite endpoint. Care should also be taken not to overwet coarse-textured soils. The presence of free water on the surface of the paste after standing is a very important indication of oversaturation in the case of coarse-textured soils.

3. Allow the saturated paste to stand for 4 or more hours:

   a) Place approx. 50 g of saturated paste into a tared 50 mL beaker, record weight, oven-dry, reweigh and calculate % moisture at saturation.

   b) Transfer remaining sample to a Buchner filter funnel fitted with Whatman No. 1 filter paper.

4. Apply vacuum and collect filtrate in test tube; terminate filtration when air begins to pass through filter, or sooner if sufficient extract has been collected.

   NOTE: 25 mL of extract is usually adequate for cation and anion analysis.

5. Measure the EC of the saturation extract using a conductivity meter. Record conductivity as millisiemens / cm at 25^{o} C.

NOTE: Standardize the conductivity cell against 0.01N KCl at 25° C.

6. Retain those samples with an EC greater than 1.5 mS/cm for cation and anion analysis.

NOTE: Add 1 drop of 0.5% $(NaPO_3)_6$ solution for each 25 mL of extract, to prevent precipitation of calcium carbonate ($CaCO_3$).

NOTE: Soil water extracts should be stored at approx. 4□ C until analyzed.

ERROR VALUES

	n	$\overline{X}$	SD	RSD(%)
% MOISTURE	6	29.3	1.4	4.8
AT SATURATION	5	96.6	6.3	6.5
EC (mS/cm)	6	1.1	0.2	18.2
	4	13.1	1.0	7.9

Rate of analysis: 80-100/week.

Calculating Soil Water Content

$$100 \times \frac{[\text{Wet Sample} - \text{Dry Sample}]}{[\text{Dry Sample} - \text{Container Wt.}]} = \text{Soil Water Content}$$

REFERENCES

1. RICHARDS, L.A. 1954. Diagnosis and Improvement of Saline and Alkali Soils. U.S. Salinity Laboratory, U.S. Dept. Agric. Hbk. 60, pp. 160.

DETERMINATION OF WATER SOLUBLE CATIONS AND ANIONS

The major solutes of interest found in soil water and aqueous extracts of salt-affected soils are: Ca^{2+}, Mg^{2+}, Na^{+}, K^{+}, CO_3^{2-}, HCO_3^{-}, SO_4^{2-}, Cl^{-} and NO_3^{-}. Methods of analysis range from wholly automated to manual. The choice of method is usually determined by the number of samples to be processed and the availability of analysts, automated equipment, etc.

1. DETERMINATION OF CALCIUM (Ca), MAGNESIUM (Mg) and SODIUM (Na)

EQUIPMENT

Atomic Absorption Spectrometer (IL-257)

Volumetric flasks, 50 mL

Adjustable micro-pipet (10-100 uL) and disposable tips

REAGENTS

Lanthanum oxide solution (La_2O_3), 5%

- dissolve 50 g of La_2O_3 in 100 mL of concentrated Hydrochloric acid (HCl), cool and dilute to 1L with deionized water.

NOTE: Lantanum or Strontium at concentrations of 0.1 to 1% (w/v) are added to sample and standards to control interferences and simultaneously control a slight ionization interference which occurs when Ca and Mg are determined in an air-acetylene flame.

NOTE: Partial ionization of Na and K in an air-acetylene flame can be suppressed by adding 0.15N Lithium chloride (LiCl) to samples and standards to give 10% by volume of this LiCl solution in the final dilution.

CERTIFIED ATOMIC ABSORPTION STANDARDS: 1000 ug/mL of Ca, Mg and Na.

PROCEDURE

1. Pipet an appropriate aliquot of sample into a 50 mL volumetric flask.

EC of SAMPLE	ALIQUOT
1.5 – 4.0	100 uL
4.1 – 10.0	50 uL
10.1 – 20.0	25 uL
20.1 +	10 uL

2. Add 2 mL of 5% La_2O_3 solution to volumetric flask and dilute to volume with deionized water.
3. Prepare a blank and a range of standards of Ca, Mg and Na.

   NOTE: Suitable range – 0.100 to 4.000 ug/mL.
4. Adjust the atomic absorption spectrometer controls and settings for the cation to be analyzed, as recommended by the manufacturer.
5. Analyze sample and record results in meq/L.

CALCULATIONS

$$Ca^{2+}(meq/L) = \frac{ug/mL\ Ca \times d.f.}{eq.\ wt.\ of\ Ca}$$

$$Mg^{2+}(meq/L) = \frac{ug/mL\ Mg \times d.f.}{eq.\ wt.\ of\ Mg}$$

$$Na^{+}(meq/L) = \frac{ug/mL\ Na \times d.f.}{eq.\ wt\ of\ Na}$$

d.f. = dilution factor of sample

SODIUM ABSORPTION RATIO (SAR)

$$SAR = \frac{Na^{+}}{\sqrt{\frac{(Ca^{2+} + Mg^{2+})}{2}}}$$

(concentration of Ca^{2+}, Mg^{2+} and Na^{+} as expressed in meq/L)

ERROR VALUES

	n	$\bar{X}$	SD	RSD(%)
Ca^{2+} (meq/L)	6	20.5	1.9	9.2
Mg^{2+}(meq/L)	5	11.8	0.3	2.8
Na^{+} (meq/L)	4	14.2	1.1	7.8

Rate of Analysis: 60-100/dayt for Ca, Mg and Na.

2. DETERMINATION OF CARBONATE (CO_3^{2-}) and BICARBONATE (HCO_3^-)

EQUIPMENT

Automated titration system (Mettler DL40RC)

Combination pH electrode (calomel and glass)

Beakers, 100 mL

Pipet

REAGENTS

Standard Nitric acid (HNO_3), approximately 0.050N

Standard buffer solutions, pH 4.00 and 7.00

PROCEDURE

1. Pipet an aliquot (usually 5 mL) of sample into a 100 mL beaker and dilute to 50 mL with distilled water.
2. Calibrate the instrument with electrode immersed in pH buffer 7.00 and then in pH 4.00; rinse electrode.
3. Immerse electrode into sample, titrate and record volume of titrant delivered to reach endpoints set pH 89.3 and pH 4.5 respectively.
4. Blank of 50 mL of distilled water is also titrated.

CALCULATIONS

$$CO_3^{2-}\ (meq/L) = \frac{2P \times N \times 1000}{\text{aliquot of sample (mL)}}$$

$$HCO_3^{-}\ (meq/L) = \frac{(T - 2P) \times N \times 1000}{\text{aliquot of sample (mL)}}$$

P = mL of std. HNO_3 of Normality N to reach endpoint pH = 8.3

T = total mL of std. HNO_3 of Normality N to reach endpoint pH = 4.5

ERROR VALUES

	n	$\overline{X}$	SD	RSD(%)
HCO_3^{-} (meq/L)	6	2.6	0.3	13.3

Rate of analysis: 100-120/day.

3. DETERMINATION Of CHLORIDE (Cl^-)

A potentiometric titration is used for Cl^- determination. (1)

EQUIPMENT

Automated titration system (Mettler DL40RC)

Electrodes, calomel (mercurous sulfate) and silver

Beakers, 100 mL

Pipet

REAGENTS

Standard Silver nitrate solution ($AgNO_3$) , approximately 0.05ON

- dissolve 8.4944 g of $AgNO_3$ in distilled water and dilute to 1 L.

NOTE: Check normality of $AgNO_3$ by titration of a standard NaCl or KCl solution.

PROCEDURE

1. Pipet an aliquot (usually 5 mL) of sample into a 100 mL beaker and dilute to 50mL with distilled water.

   NOTE: Sample aliquot used for CO_3^{2-} and HCO_3^- determination may be used for the Cl^- determination.

2. Set instrument parameters for Cl^- measurement and titrate the sample with $AgNO_3$ solution to the endpoint (70 mv).

3. Titrate a blank of 50 mL of distilled water with each set of samples.

CALCULATIONS

$$Cl^- \text{ (meq/L)} = \frac{(Vs - Vb) \times N \times 1000}{\text{aliquot of sample (mL)}}$$

Vs = mL of $AgNO_3$ used to reach endpoint of sample

Vb = mL of $AgNO_3$ used to reach endpoint of blank.

N = Normality of $AgNO_3$

ERROR VALUES

	N	$\overline{X}$	SD	RSD(%)
Cl^- (meq/L)	4	10.3	0.6	6.0

Rate of analysis: 100-120/day.

REFERENCES

1. DAVEY, B.G. and BEMBRICK, M.J. 1969. The potentiometric estimation of chloride in water extracts of soils. Soil Sci. Soc. Amer. Proc. 33 : 385-387.

4. DETERMINATION OF SULFATE (SO_4^{2-})

A sulfate solution is titrated with Lead nitrate $Pb(NO_3)_2$ in the presence of ferro-cyanide / ferric-cyanide redox system, using platinum (Pt) and silver (Ag) electrodes. In an excess of SO_4^{2-} the ratio of Fe^{2+} to Fe^{3+} cyanide remains constant and the redox potential of the system also remains constant. When all the SO_4^{2-} is precipitated as $PbSO_4$, additional $Pb(NO_3)_2$ will result in a precipitate of Lead ferro-cyanide, leaving the ferric-cyanide unaffected. A sharp potential change can be detected in the system. The procedure used is adapted from D.Y. Golkin et al. (1)

EQUIPMENT

Automated titration system (Mettler DL40RC)

Electrodes, platinum (Pt) and silver (Ag)

Beakers, 100 mL

Pipet

REAGENTS

Standard Lead nitrate solution [$Pb(NO_3)_2$], approx. 0.02ON

- dissolve 3.312 g of $Pb(NO_3)_2$ in distilled water and dilute to 1 L.

NOTE: Check normality of $Pb(NO_3)_2$ by titration of a standard Potassium sulfate (K_2SO_4) solution.

Potassium ferro-cyanide trihydrate [$K_4Fe(CN)_6.3\ H_2O$] .02ON

- dissolve 2.115 g of $K_4Fe(CN)_6.3\ H_2O$ in distilled water and dilute to 1 L.

Potassium ferric-cyanide [$K_3Fe(CN)_6$] 0.3ON

- dissolve 32.926 g of $K_3Fe(CN)_6$ in distilled water and dilute to 1 L.

Isopropyl alcohol ($CH_3CHOHCH_3$), 99%

PROCEDURE

1. Pipet a suitable aliquot into a 100 mL beaker, and add distilled water to bring the volume to 25 mL.

EC OF SAMPLE	ALIQUOT
1.6 – 4.0	3 ml
4.1 – 8.0	2 mL
8.1 +	1 mL

2. Add 25 mL of isopropyl alcohol, 1 mL of $K_3Fe(CN)_6$ and 0.10 mL of $K_4Fe(CN)_6$.
3. Set instrument parameters for sulfate (SO_4^{2-}) measurement and titrate slowly to the endpoint (320 mv).

   NOTE: Titrate a standard solution of SO_4^{2-} and plot a curve of mL of titrant vs mv to determine exact endpoint.
4. Titrate a blank of 25 mL of distilled water and reagents as per sample.

CALCULATIONS

$$SO_4^{2-}\ (meq/L) = \frac{(Vs - Vb) \times N \times 1000}{\text{aliquot of sample (mL)}}$$

Vs = mL of $Pb(NO_3)_2$ used to reach endpoint of sample

Vb = mL of $Pb(NO_3)_2$ used to reach endpoint of blank.

N = Normality of $Pb(NO_3)_2$

ERROR VALUES

	n	$\overline{X}$	SD	RSD(%)
SO_4^{2-} (meq/L)	5	38.2	3.1	8.1

Rate of analysis: 20-30/day

REFERENCES

1. GOLKIN, D.Y., LOZANOVA, L.N., PINAYEVA, I.B., ALEKSANDROVA, L.V. 1960. Quick and simple electronic method for determining sulfate in salinized soils. Soviet Soil Science No. 1 - Jan. 1960: 198-200.

DETERMINATION OF $CaCO_3$ EQUIVALENT AND ESTIMATES FOR CALCITE & DOLOMITE

Carbonate Procedure

1. Grind samples
2. Determine reactivity of samples. This is done by placing a small amount of sample into a well of a spot plate. Add a drop or two of 4N HCl near the edge of the sample. Observe the bubbling and rate it as: 0.5 grams, 1.0 grams, 2.0 grams, 3.0 grams, 4.0 grams, 5.0 grams, none.

◄----------►

order of increasing reactivity

ex) vigorous bubbling would be 0.5 to 1.0 grams. this means you would weigh 0.5 to 1.0 grams (whichever you decide) in a container.

ex) very little bubbling would be 5.0 grams, etc.

3. Record the sample‘s information on the Calcium Carbonate sheet. Weigh out the corresponding amount of soil in a disposable microbeaker. Make sure to zero the scale with the empty microbeaker on it.

   NOTE: When weighing the samples it must be ± 0.0005.

4. Take the Calcium Carbonate beaker (marked 1 or 2) and fill it with 30 mL of 4N Cl.

   Mark which beaker was used on the Calcium Carbonate sheet.

5. CAREFULLY place the microbeaker filled with soil in the beaker using tongs. DON’T SPILL OR YOU HAVE TO RESTART!

6. Place the beakers in the water bath (~26^{o} C) and attach them to the shaker. Take an elastic band and attach the two beakers together. Allow the beakers to sit for 10 minutes in the bath.
7. After the 10 minutes have passed, move the rulers so that they line up at zero with the mercury level.
8. Turn the timer on (sometimes samples can go for an hour so turn it on for 60 minutes) and flip the lever to 10. Leave it at 10 for 15 seconds then turn it down to 5.

   AS SOON AS THE LEVER IS FLIPPED START THE STOPWATCH. YOUR FIRST READING WILL BE AT 30 SECONDS1
9. Take your readings accordingly and record them on the Calcium Carbonate sheet.

   Make sure to tap the columns containing the mercury <u>before</u> taking the reading as the mercury tends to stick in the column.

   You are done taking readings once you obtain 3 successive readings of the same value.
10. Clean the beakers.

When carbonates are treated with hydrochloric acid in a closed system under constant volume and temperature, the increase in pressure as read on a manometer is related linearly to the content of CO_2 in the carbonates. Using a pressure calcimeter as described by Skinner and Halstead (1,2), constant volume is maintained with reaction bottles of similar volume, and temperature is controlled by a constant temperature water

bath. If dolomite is present in a sample, the readings should continue for approximately 45 minutes. Clay-sized dolomite cannot be distinguished from calcite by this method. (A slight reading may be obtained with a carbonate-free sample, probably due to the heat of wetting.)

EQUIPMENT

- Pressure calcimeter – a 700 mL wide-mouth bottle fitted with a single-hole stopper and appropriate tubing connected to a mercury manometer.
- Write-action shaker (Burrell)
- Constant temperature water bath
- 5 mL polystyrene micro-beakers
- Tongs or forceps

REAGENTS

Hydrochloric acid (HCl) 4N

Distilled water adjusted to a slightly basic pH

PROCEDURE

1. Prepare an oven-dry sample ground to pass a No. 35 (0.5 mm opening) sieve (sample capable of passing 80 mesh is also acceptable).
2. Clamp reaction bottle onto wrist-action shaker so it is partially immersed in the adjacent constant temperature water bath, which is maintained at 25^{o} C.

NOTE: Exact temperature of 25^{o} C is not vital but samples and standards must be run at same temperature.

3. Add 30 mL of 4N HCl to reaction bottle and weigh into micro-beaker sufficient sample to obtain a manometer reading of 2.5 to 8 cm of mercury.
4. After moistening (not too much) with distilled water, lower micro-beaker into reaction bottle, stopper bottle tightly, allow several minutes for the temperature of the bottle to equilibrate with the temperature of the water bath, then adjust manometer to zero.
5. Start shaker at full speed, reducing to half speed after the sample and the HCl have reacted (about 15 seconds).
6, Record readings at 30 second intervals for 2 minute, then at minute intervals until 5 minutes have elapsed. Take additional readings every 5 minutes until no change in readings occurs.

CALCULATIONS

1. Manometer readings (Ht) recorded at each time interval, are subtracted from the final reading (H∞).
2. The log values (H∞ - Ht) are plotted on a vertical axis (ordinate) against time (minutes) on a horizontal axis (abscissa).
3. The straight line portion of the curve occurring after approximately 1 minute is extrapolated to zero time, and the intercept value (Hd) represents CO_2 derived from dolomite.

4. The difference between the final reading (H∞) and the intercept represents the CO_2 from calcite(Hc).
5. Values for H∞, Hc and Hd are converted to grams of CO_2 by means of a standard curve obtained from measurements of CO_2 evolved from different amounts of pure $CaCO_3$.
6. The weight of CO_2 obtained from H∞ and Hc is converted to $CaCO_3$ equivalent and calcite respectively by using the theoretical CO_2 content of 43.9%. The weight of CO_2 obtained from dolomite is converted by using the theoretical CO_2 content of 47.73%.

ERROR VALUE

	n	$\overline{X}$	SD	RSD%
$CaCO_3$	16	14.3	0.5	3.2

Rate of analysis: 10-15 samples/day

REFERENCES

1. SKINNER, S.I.M and HALSTEAD, R.L. 1958. Notes on rapid method for determination of carbonates in soils. Can. J. Soil Sci. 38: 187-188.
2. SKINNER, S.I.M., HALSTEAD, R.L., BRYDEN, J.E. 1959. Quantitative manometric determination of calcite and dolomite in soils and limestones. Can. J. Soil Sci. 39: 197-204.

3. TURNER, R.C. and SKINNER, S.I.M. 1960. An investigation of the intercept method for determining the proportion of calcite and dolomite in mixtures of the two. Can. J. Soil Sci. 40: 232-241.

DETERMINATION OF ORGANIC CARBON

Organic Carbon Procedure

1. Place the dried sample in a metal tin. Fill the tin about half full.
2. Grind the sample.
3. Write the sample number on the organic carbon sheet. Take the corresponding 180 mL beaker and place 0.5 g in it.
   - Use the scale that weighs to 4 decimal places.
   - Tare the scale with the empty beaker on it.
   - When you get close to 0.5 g (ie 0.4980 and up) close the scale's door to see the actual weight.

   BE CAREFUL NOT TO ADD TOO MUCH SAMPLE! Taking the beaker off the scale leads to error.
   - For this test the weight can be ± 0.005 g.
4. In fume hood: Add 10 mL of 1N $K_2Cr_2O_7$ to the sample, mix it well. Next, add 20 mL of H_2SO_4 **SLOWLY** to the beaker. Mix very well. Let the sample sit for 30 minutes.
   - MAKE SURE THERE ARE NO AIR BUBBLES IN THE DISPENSERS.
   - When adding the chemicals start at the back and work your way to the front. (The reaction gives off heat).
   - Make sure your sample is away from the dispensers so as no liquid falls into the beaker.

   GOGGLES AND GLOVES MUST BE WORN WHEN DEALING WITH THE CHEMICALS.

5. Fill the beaker up to the top of the white circle with DEIONIZED water. Let it stand for 15 minutes.

   - There should be 3 blanks made up of 10 mL of 1N $KrCr_2O_7$ and 20 mL of H_2SO_4. (the first blank is not important for the average concentration of $FeSO_4$). Add deionized H_2O to the blanks.

   - The blanks are made the same as the samples.

6. After the samples have sat for the 15 minutes take the samples to the autotitrator. Place the blanks in first. The samples are placed in next, by numerical order. The autotitrator runs counterclockwise. Place the yellow magnet at the last sample beaker. Make sure it clocks into place and is centered at the beaker. Place a beaker ¾ full of deionized water after the last sample.

7. BEFORE you start: make sure the $FeSO_4$ bottle has enough solution in it for the samples (at least half a bottle). Make sure the printer is turned on.

8. To start:

   a) Push “RUN”

   b) Enter the number of samples to be titrated (include the blanks) and push “F5”.

   c) For the next menu screen push “F5” unless the method has to be chanced.

   d) The titrations will start now.

   - Make sure to watch the titration of the blanks and note the final amount of $FeSO_4$ used. The amounts of $FeSO_4$ used for the blanks should differ by only 0.500 mL.

- The average of the two blanks gives the concentration of the $FeSO_4$ to be used in the calculation of the amount of organic carbon in the sample.

9. When the samples have finished the printer will print the results. On the paper write the corresponding lab and blank numbers beside their results. Then enter the amounts of $FeSO_4$ used to neutralize the samples on the organic carbon sheets.
10. Then enter the information into the computer program to obtain the percent of organic carbon and percent of organic matter. Record these values on the organic carbon sheet.

- MAKE SURE THE RESULTS PRINTED <u>BEFORE</u> YOU RESET THE AUTOTITRATOR OR ELSE YOUR RESULTS ARE LOST. If it didn't print then obtain your results by scrolling down the values.
- <u>IF</u> you get an ERROR instead of an amount of $FeSO_4$ then half the amount of soil used until a value is obtained.

The procedure used for determining organic carbon is the Walkley-Black method, modified for automatic titration, based on the oxidation of organic matter in soil by $Cr_2O_7^{2-}$ (2, 3, 4). Heat generated by the addition of concentrated H_2SO_4 to a soil and $Cr_2O_7^{2-}$ solution promoted oxidation of soil organic matter. Excess $Cr_2O_7^{2-}$ is titrated with Fe^{2+}, with the endpoint in the titration determined by potentiometer measurement using platinum and calomel electrodes (1). This method is subject to interference by chloride (Cl^-), ferrous iron (Fe^{2+}), and higher oxides of manganese (MnO_2) when present in significant amounts in soils.

Certain assumptions must be made in the use of this method. Firstly, 75% of the organic matter present is oxidized; thus a correction factor of 1.33 is used. Secondly, organic carbon in soil organic matter has an average oxidation state of zero and an equivalent weight of 3 g per equivalent.

EQUIPMENT

200 mL tall-form beakers

Acid burret

10 mL pipet

Mettler DL40 RC titration system

Platinum and calomel electrodes

REAGENTS

Potassium dichromate solution ($K_2Cr_2O_7$) 1N.

- dissolve 49.04 g of reagent-grade $K_2Cr_2O_7$ (dried at 105^o C) in water, and dilute to 1000 mL.

Ferrous sulfate heptahydrate ($FeSO_4.7\ H_2O$) 0.05N

- dissolve 140 g of reagent-grade $FeSO_4.7\ H_2O$ in 40 mL of concentrated Sulfuric acid (H_2SO_4), cool and dilute to 1000 mL.

Sulfuric acid (H_2SO_4) concentrated – not less than 96%.

PROCEDURE

1. Grind a sample to pass a No. 35 (0.5 mm opening) sieve and oven-dry at 105˚ C.

NOTE: Avoid using iron or steel grinders for preparing the sample.

2. Transfer a weighed sample containing 10-25 mg (0.5000 g) or organic carbon, but not in excess of 10 g of soil, into a 200 mL beaker (see Table 1).
3. Add 10 mL of 1N $K_2Cr_2O_7$ and swirl the beaker gently to disperse the soil in the solution.
4. Rapidly add 20 mL of concentrated H_2SO_4, directing the stream of acid into the suspension. Immediately, swirl the beaker by hand for 1 minute and place the beaker in a fume hood for 30 minutes.
5. Add 75-100 mL of distilled water and allow the contents to cool.
6. Prepare a blank determination following the previously described steps.
7. Set up automatic titration system and titrate blank and sample.
8. Repeat determination with less sample if more than 75% of the dichromate has been reduced.

CALCULATIONS

$$\% \text{ ORGANIC CARBON} = \frac{B - V}{B} \times \frac{Vo \times No \times 0.4}{\text{weight of sample (g)}}$$

B = mL of $FeSO_4$ used for blank

V = mL of $FeSO_4$ used for sample

Vo = mL of $K_2Cr_2O_7$ added

No = normality of $K_2Cr_2O_7$

NOTE: Approximate conversion of organic C to organic matter

% ORGANIC MATTER = 1.7 X % ORGANIC CARBON

Table 1

HORIZON	WEIGHT OF SAMPLE
Om, Of, Oh	0.1 g *
Ah, Ae	0.25 – 0.5 g
Bm, Bh, Bt	1 – 3 g

* (double quantities of $K_2Cr_2O_7$ and H_2SO_4)

ERROR VALUE

	n	$\overline{X}$	SD	RSD%
ORGANIC CARBON (%)	17	2.49	.17	7.1
	10	24.80	1.07	4.3
	10	44.43	.97	2.2

Rate of analysis: 40-60/day

REFERENCES

1. RAVEH, A. and AYNIMELECH, Y. 1973. Potentiometer determination of soil organic matter. Soil Sci. Am. Proc. 36: 967.

2. WALKLEY, A. 1935. An examination of methods for determining organic carbon and nitrogen in soil. J. Agric. Sci. 24: 598:609.

3. WALKLEY, A. 1947. A critical examination of a rapid method for determining organic carbon in soils – effect of variations in digestion conditions and of inorganic soil constituents Soil Sci. 63: 251-263.

4. WALKLEY, A. and BLACK, I.A. 1934. An examination of the Degtjareff method for determining soil organic matter, and a proposed modification of the chromic acid titration method. Soil Sci. 37: 29-38.

DETERMINATION OF PYROPHOSPHATE-SOLUBLE ORGANIC MATTER INDEX FOR ORGANIC SOILS

The degree of humification of organic soils can be related to a degree by sodium pyrophosphate-soluble organic matter. The colorimetric procedure of Kaila (1) was used by Schnitzer and Desjardins (2) in a comparison of procedures.

EQUIPMENT

Shaking bottles

Filter funnels and stand

Shake

Spectrophotometer (Unicam SP 600)

REAGENTS

Sodium pyrophosphate decahydrate solution ($Na_4P_2O_7.10\ H_2O$) 0.025M

- dissolve 11.152 g of $Na_4P_2O_7.10\ H_2O$ in distilled water and dilute to 1 L.

PROCEDURE

1. Weigh 0.5 g air-dried soil into a shaking bottle and add 50 mL of 0.025M $Na_4P_2O_7$ solution.
2. Shake for 18 hours, filter and dilute filtrate to 250 mL with distilled water.
3. Measure the absorbance of this solution at 550 mu in a spectrophotometer.

CALCULATIONS

Multiply the absorbance by 100 to give cardinal numbers. (Spec. reading x 100).

REFERENCES

1. KAILA, A. 1956. Determination of the degree of humification in peat soils. Maataloust Aikak. 28: 18-35.
2. SCHNITZER, M. and DESJARDINS, J.G. 1965. Carboxyl and phenolic groups in some organic soils and their relation to the degree of humification. Can. J. Soil Sci. 45: 257-264.

LOSS-ON-IGNITION

Loss-on-ignition provides a reliable and simple estimation of soil organic matter (1,2). Loss of CO_2 from carbonates and loss of structural water from clay minerals, except gibbsite, are minimal at temperatures of 350˚-450˚ C. (600˚ C).

EQUIPMENT

Muffle furnace

drying oven

porcelain crucible

PROCEDURE

1. Weigh 20.00 g of mineral (2.000 g of organic) oven-dried sample into a tared porcelain crucible.
2. Place crucible and contents into muffle furnace and gradually increase the temperature to 400˚ C (or 600˚ C). Leave for 12-24 hours.
3. Cool crucible and contents in a dessicator and weigh.

CALCULATIONS

$$\text{LOSS-ON-IGNITION (\%)} = \frac{(Wod-Wi) \times 100}{Wod}$$

Wod	=	weight of oven-dried sample
Wi	=	weight of sample after ignition @ 400˚ C.
Loss-on-ignition	=	organic matter content

% ASH = 100 - % loss-on-ignition.

REFERENCES

1. BALL, D.F. 1964. Loss-on-ignition as an estimate of organic matter and organic carbon in non-calcareous soils. Journal of Soil Science. 15: 84-92.
2. DAVIES, B.E. 1974. Loss-on-ignition as an estimate of soil organic matter. Soil Sci. Amer. Proc. 38: 150.

DETERMINATION OF NITROGEN KJELDAHL

The Kjeldahl method of nitrogen determination involves wet digestion of a sample to convert organic nitrogen to NH_4^+-N and then determining the nitrogen content.

Digestion is accomplished by heating a sample with concentrated Sulfuric acid (H_2SO_4). Addition of a salt such as Potassium sulfate (K_2SO_4) increases the temperature of digestion, and addition of catalysts such as mercury (Hg), copper (Cu) or selenium (Se) increases the rate of oxidation of organic matter (1). When NH_4^+-N in the digest is distilled in the excess of NaOH, the ammonia (NH_3) released in the distillation is trapped in Boric acid (H_3BO_3) and then titrated with a standard acid solution.

EQUIPMENT

KJELTEC AUTO 1030 Analyzer

Tecator Digestor 20/40 with built-in thermostat

Accessories - digestion tubes, stand, exhaust manifold, heat shield, stand for rapid cooling of tubes, retainer plate, boiling chips

REAGENTS

Sulfuric acid (H_2SO_4), concentrated and 0.05N standard

KJELTABS (3.5 g K_sSO_4 + .0035 g Se)

Sodium hydroxide (Na0H) 35-40%

Boric acid (H_3BO_3) – 1% with bromocresol green-methyl red indicator

- dissolve 100 g of boric acid in 10 L deionized water (1% solution)

- add 100 mL bromocresol green solution (100 mg in 100 mL methanol)
- add 70 mL methyl red solution (100 mg in 100 mL methanol)
- add 5 mL NaOH (alkali is necessary to achieve a positive blank value).

PROCEDURE

NOTE: The quantity of regents and samples described in this procedure are based on the "SEMI-MICRO" method.

1. Grind a sample to pass a No. 35 (0.5 mm opening) sieve and oven-dry at 105˚ C.
2. Transfer a 1.50 g mineral sample (0.30 g of organic sample) into a 100 mL digestion tube.

   NOTE: Samples of clay soils are wetted with distilled water prior to addition of concentrated H_2SO_4. (2)
3. Add 1 KJELTAB and 9 mL of conc. H_2SO_4. so that the sample is completely immersed at the bottom of the tube. Boiling chips are added to reduce bumping during digestion.
4. Pre-heat block digestor to 420˚ C (may require up to 3 hours). Allow sample to digest for 45 minutes at a temperature of 420˚ C, then allow 30 minutes to cool.
5. Add 50 mL of deionized water.

   NOTE: If digest has solidified it must be redissolved.
6. Insert and read a blank reagent sample with the KJELTEC after the instrument has been calibrated with deionized water.
7. Insert digestion tube with sample into KJELTEC and obtain reading.

CALCULATIONS

$$\% \text{ NITROGEN} = V \times N \times \frac{1.4}{\text{Weight of sample (g)}}$$

V = Volume of .05N H_2SO_4

N = Normality of H_2SO_4

ERROR VALUE

	n	$\overline{X}$	SD	RSD(%)
N (%)	5	.278	.007	2.5
	10	2.230	.071	3.2

Rate of analysis: 30-40/day

REFERENCES

1. BREMNER, J.M. 1960. Determination of nitrogen in soil by the Kjeldahl method. J. Agric. Sci. 55: 11-31.
2. MORAGHAN, J.T., REGO, T.J., SAHRAWAT, K.L. 1983. Effect of water pretreatment on total nitrogen analysis of soils by the Kjeldahl method. Soil Sci. Soc. Am. J. 47: 213-217.

DETERMINATION OF CATION EXCHANGE CAPACITY AND EXTRACTION OF EXCHANGEABLE CATIONS (MINERAL SAMPLES)

Cation exchange capacity (CEC) is a measure of the quantity of readily exchangeable cations neutralizing negative charge in the soil.

Ammonium acetate (NH_4OAc), pH 7.0, has been employed widely for determining soil CEC (1). Significant errors result due to ammonium fixation by some soils, and the presence of calcium carbonate, gypsum and other salts in carbonated and saline soils. Methods of CEC determination and their suitability are discussed by Rhoades (2).

EQUIPMENT

Vacuum pump (Welch, 2 stage)

Vacuum manifold (8-10 outlets) with filter flasks & Buchner funnels (5.5 cm)

Filter paper (Whatman No. 42)

100 mL beakers

Wash bottles, 500 mL

100 mL graduated cylinder or 100 mL volumetric flask

Distillation unit (KJELTEC Auto 1030 Analyzer)

REAGENTS

Ammonium acetate (NH_4OAc) 1N, pH 7.0

- dissolve the pH to 7.0 by adding Acetic acid (HOAc) or ammonium hydroxide (NH_4OH).

- adjust the pH to 7.0 by adding Acetic acid (HOAc) or ammonium hydroxide (NH_4OH)

Isopropyl alcohol ($CH_3cHOHCH_3$), 99%

Sodium chloride (NaCl), acidified

- dissolve 58.46 g NaCl in distilled water, add HCl to make the solution approximately 0.005N with respect to acidity, dilute to 1L.

Sodium hydroxide (NaOH), 35-40%

Boric acid (H_3BO_3) – 1% with bromocresol green-methyl red indicator solution

- dissolve 100 g of Boric acid in 10 L deiionized water (1% solution).
- add 100 mL of bromocresol green solution (100 mg in 100 mL methanol) + 70 mL methyl red solution (100 mg in 100 mL methanol).
- add 5 mL 1N NaOH (alkali is necessary to achieve a positive blank value).

Sulfuric acid (H_2SO_4) 0.05N

PROCEDURE

1. Weigh 10 g of 2mm, air-dried soil into a 100 mL beaker.
2. Add 30 mL of 1N NH_4OAc solution and stir 2 or 3 times (cover and allow to stand overnight.
3. Filter the supernatant liquid though the Buchner funnel, then wash the soil from the beaker to the funnel with NH_4OAc from a wash bottle.

   NOTE: Do not allow soil to dry.
4. Leach the soil with small portions of NH_4OAc until the total volume of extract is 80-90 mL.

5. Transfer the leachate to a 100 mL graduated cylinder or volumetric flask, make up to volume with NH_4OAc and keep for analysis of exchangeable or extractable cations.

   NOTE: The term "exchangeable" is not applicable for soil containing free calcium carbonate ($CaCO_3$), gypsum ($CaSO_4.2\ H_2$)) or high amounts of soluble salts.

6. Wash out excess NH_4OAc from the soil with 100 mL of 99% isopropyl alcohol, discard the alcohol filtrate.

7. Leach the ammonium-saturated soil with acidified NaCl, adding small portions and allowing each portion to pass through the sample before more is added until about 95 mL of extract has been collected.

8. Transfer extract to 100 mL volumetric flask, rinse filter flask with a small portion of NaCl, add to volumetric flask and dilute to volume.

9. Adjust instrument for blank and analyze 25 mL aliquot of NaCl extract.

CALCULATIONS

$$\text{CEC (meq / 100 g)} = \frac{(Vs - Vb) \times N}{\text{aliquot}} \times \frac{100}{\text{Wt. of sample (g)}}$$

Vs = mL of H_2SO_4 used for sample aliquot

Vb = mL of H_2SO_4 used for blank

N = normality of H_2SO_4

ERROR VALUE

	n	$\overline{X}$	SD	RSD(%)
CEC (meq/100 g)	6	42.3	1.1	2.5

Rate of analysis: 75/233k

REFERENCES

1. CHAPMAN, H.D. 1965. Cation-exchange capacity, IN Agronomy No. 9, Methods of Soil Analysis, Part 2, Black, C.A., ed., pp. 891-901.
2. RHOADES, J.D. 1982. Cation exchange capacity. IN Agronomy No. 9, Methods of Soil Analysis, Part 2, Page, A.L., Miller, R.H., Keeney, D.R. eds., pp. 149-157.

DETERMINATION FO CATION EXCHANGE CAPACITY (ORGANIC SAMPLES)

To provide a measure of the total amount of exchangeable cations that can be held by organic soil, the method of Thorpe was adapted. (1)

EQUIPMENT

125 Ml Erlenmeyer flasks

250 mL beakers

Buret assembly

Vacuum pump

Vacuum manifold (8-10 outlets) with filter flasks, Buchner funnels (5.5 cm diam.), filter paper (Whatman No. 42)

Shaker

REAGENTS

Hydrochoric acid (HCl) 0.5N

Standard Sodium hydroxide (NaOH), approx. 0.50N

NOTE: Check normality of NaOH by titration with standard acid (or a primary standard.

Barium acetate [$Ba(OAc)_2$] 1.0N

- dissolve 127.7 g $Ba(OAc)_2$ in distilled water and dilute to 1L
- adjust pH to 7.0

Silver nitrate ($AgNO_3$) 0.10N

- dissolve 4.25 g of $AgNO_3$ in distilled water and dilute to 1L

Thymol blue indicator solution – 5% in ethanol

PROCEDURE

1. Weigh 2 g of air-dried organic sample into a 125 mL Erlenmeyer flask and add 100 mL of 0.5N HCl.
2. Shake the flask for 2 hours and allow to stand overnight.
3. Filter sample and wash with distilled water until the filtrate is free of chlorides (test leachate with $AgNO_3$ for white precipitate).
4. Transfer filter paper and sample into a 250 mL beaker.
5. Add 100 mL of 1.0N $Ba(OAc)_2$ and allow to stand for 2 hours.
6. Filter and wash sample with 1.0N $Ba(OAc)_2$, bring to final volume of 150 mL.
7. Titrate leachate to pH 7.0 with 0.5N NaOH and thymol blue as an indicator.

   NOTE: Endpoint can also be determined potentiometrically.
8. Titrate a blank containing an equivalent volume of 1.0N $Ba(OAc)_2$ solution.

CALCULATIONSB

$$\text{CEC (meq/200 g)} = \frac{(Vs - Vb) \times N \times 100}{\text{Weight of sample (g)}}$$

Vs = volume of NaOH used to titrate sample leachate to pH 7.0

Vb = volume of NaOH used to titrate blank to pH 7.0

N = normality of NaOH

Rate of analysis: 16/day

REFERENCES

1. THORPE, V.A. 1973. Collaborative study of the cation exchange capacity of peat minerals. Journal of the AOAC, 56: 154-156.

EXTRACTION OF EXCHANGEABLE CTIONS (ORGANIC SOIL)

EQUIPMENT

Vacuum pump

Vacuum manifold (8-10 outlets) with filter flasks, Buchner funnels (5.5 mm diam.) and filter paper (Whatman No. 42)

100 mL beakers

Wash bottles, 500 mL

REAGENTS

Ammonium acetate (NH_4OAc) 1N, pH 7.0

- dissolve 77.08 g of NH_4OAc in distilled water and dilute to 1L
- adjust the pH to 7.0 by adding Acetic acid (HOAc) or Ammonium hydroxide (NH_4OH)

PROCEDURE

1. Weigh 1 g of air-dried organic soil into a 100 mL beaker and add 30 mL of 1N NH_4OAc .
2. Stir the sample, cover, and allow to stand overnight.
3. Filter the soil with suction through a Buchner funnel, leach the soil with small portions of 1N NH_4OAc until the total volume of extract is 80 mL.
4. Transfer the leachate to a 100 mL volumetric flask, rinse filter flask with a small portion of 1N NH_4OAc , add to volumetric flask and dilute to volume.
5. Save for analysis of Ca, Mg, Na and K.

ANALYSIS FOR Ca, Mg, Na and K in NH_4OAc EXTRACT

EQUIPMENT

Atomic Absorption Spectrometer (IL-257)

Volumetric flasks, 50 mL

Adjustable micro-pipets (10 to 100 mL) and disposable tips

REAGENTS

Lanthanum oxide solution (La_2O_3) 5%

- dissolve 50 g of La_2O_3 in 100 mL of concentrated hydrochoric acid (HCl), cool and dilute to 1L with deionized water.

NOTE: Lanthanum or strontium at concentrations of 0.1 to 1% (w/v) are added to sample and standards to control interference and simultaneously control a slight ionization interference which occurs when Ca and Mg are determined in an air-acetylene flame.

Lithium chloride solution (LiCl) 0.15N

- dissolve 6.358 g of LiCl and make up to 1 L with deionized water.

NOTE: Partial ionization of Na and K in an air-acetylene flame can be suppressed by adding LiCl solution to samples and standards.

Certified Atomic Absorption Standards

- 1000 ug/mL solutions of Ca, Mg, Na and K.

PROCEDURES

1. Pipet an appropriate aliquot of sample into a 50 mL volumetric flask.

NOTE Suitable aliquot – MINERAL SOIL: .20 mL for Ca & Mg

2.00 mL for Na & K

ORGANIC SOIL: 1.00 mL for Ca & Mg

10.00 mL for Na & K

2. Add 2 mL of 5% La_2O_3 solution, 5 mL of 0.15N LiCl solution to volumetric flask and dilute to volume with deionized water.

3. Prepare a blank and a range of standards of Ca, Mg, Na and K.

   NOTE: Suitable range – 0.100 to 10.00 ug/mL.

4. Adjust the atomic absorption spectrometer controls and settings for the cation to be analyzed as recommended by the manufacturer.

5. Analyze sample and record result at concentration of cations in original sample in meq/100 g.

CALCULATIONS

$$\text{Ca (meq/100 g)} = \frac{\text{ug/mL Ca x ER x df}}{\text{10 x EQ.Wt. of Ca}}$$

$$\text{Mg (meq/100 g)} = \frac{\text{ug/mL Mg x ER x df}}{\text{10 x EQ.Wt. of Mg}}$$

$$\text{Na (meq/100 g)} = \frac{\text{ug/mL Na x ER x df}}{\text{10 x EQ.Wt. of Na}}$$

$$\text{K (meq/100 g)} = \frac{\text{ug/mL K ER x df}}{\text{10 x EQ.Wt. of K}}$$

ER = extraction ratio $\frac{\text{(Solution)}}{\text{(Soil)}}$

df = dilution factor

ERROR VALUES

	N	$\overline{X}$	SD	RSD(%)
Ca (meq/100 g)	5	30.4	2.3	7.7
Mg (meq/100 g)	5	14.4	0.6	4.3
Na (meq/100 g)	5	0.22	0.04	18.2
K (meq/100 g)	6	1.04	0.13	12.5

Rate of analysis: 70-80/week

DETERMINATION OF EXCHANGE ACIDITY

Exchangeable acidity is a fairly arbitrary quantity composed of H ions obtained from the hydrolysis of exchangeable and nonexchangeable Al^{3+}, weakly acidic groups on organic matter and exchangeable H. (2)

The procedure described is a modification of Mehlich's $BaCl_2$-triethanolamine as reported by Peech et al. (1)

EQUIPMENT

Vacuum pump

Vacuum manifold (8-10 outlets) with filter flasks, Buchner funnels (5.5 cm diam.), and filter paper (Whatman No. 42)

Buret assembly

125 mL Erlenmeyer flask

REAGENTS

0.5n Barium chloride dehydrate ($BaCl_2.2\ H_2O$) - 0.05N triethanolamine [$N(CH_2CH_2OH)_3$]

- weigh 1100 g of $BaCl_2.2\ H_2O$ into a 20 L bottle and dissolve in CO_2-free distilled water.
- add 133 mL of C.P. conc. triethanolamine, and 36 mL of conc. HCl.
- mix well and make volume to 18 L.
- adjust pH to 8.0 ± .1 with HCl or triethanolamine.

- protect from in CO_2 contamination by attaching a tube containing soda lime to the air intake.

Mixed Indicator solution

- dissolve 0.22 g of bromocresol green and 0.075 g of methyl red in 96 g of ethanol (95%), add 3.5 mL of 0.1N NaOH and mix.

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

NOTE: Check normality of HCl by titration with a primary standard or with a standardized NaOH solution.

PROCEDUREB

1. Weigh 10 g of 2mm, air-dried mineral soil (2 g for organic soil) into a 125 mL Erlenmeyer flask and add 100 mL of 0.5N $Bacl_2$ –triethanolamine solution.
2. Stopper, mix and allow to stand overnight.
3. Transfer the contents of the flask to a Buchner funnel fitted with filter paper. Filter and rinse several times with $Bacl_2$ –triethanolamine solution until a volume of 190 mL has been collected.

   NOTE: Allow each portion of the solution to drain before adding the next portion; do not allow soil to dry.
4. Add 2-3 drops of indicator and titrate leachate with 0.1N HCl to the endpoint of the indicator.

   NOTE: Endpoint can also be determined potentiometrically.

5. Titrate a blank containing an equivalent volume of $BaCl_2$-triethanolamine solution.

CALCULATIONS

EXCHANGEABLE ACIDITY: meq/100 G = $(Vb) - Vs) \times N \times \frac{100}{\text{sample wt. (g)}}$

Vb = mL of HCl required to titrate blank to endpoint.

Vs = mL of HCl required to titrate soil sample extract to endpoint.

N = normality of HCl

Rate of analysis: 100-125/week

REFERENCES

1. PEECH, M., COWAN, R.L., BAKER, G.H. 1962. A critical study of the $BaCL_2$-triethanolamine and the Ammonium acetate methods for determining the exchangeable hydrogen content of soils. Soil Sci. Soc. Proc. 26: 37-40.
2. THOMAS, G.W. Exchangeable cations. IN Agronomy No. 9. Methods of Soil Analysis, Part 2, Page, A.L., Miller, R.H., Keeney, D.R. eds., pp 161-163.

DETERMINATION OF TOTAL ELEMENTS (Fe, Mn, Zn, Cu)

Elemental analysis of soils necessitates their decomposition into soluble forms by acid digestion or fusion in Na_2CO_3. Numerous methods are documented for acid digestion of soil and the determination of elements in solution by atomic absorption spectrometry (1, 3, 4). The procedure outlined below is an acid digestion (HNO_3, HF and $HCLO_4$) and elemental determination by atomic absorption spectrometry spectrometry.

EQUIPMENT

Atomic Absorption Spectrometer (IL-257) with background corrector

Stainless steel fume hood

NOTE: If $HClO_4$ is used in a fume hood that previously was (or later is) used for organic materials, an explosive reaction can occur.

Teflon beaker (100 mL capacity)

Electric hot plate

Volumetric flasks (50 mL capacity)

Polypropylene bottles (50 mL capacity)

REAGENTS

Hydrofluoric acid (HF), 48%

Perchloric acid ($HClO_4$), 70-72%

Nitric acid (HNO_3), 10% and 70% solution

Water, redistilled or deionized

Standard stock solutions (1000 ug/mL) of various elements

PROCEDURE

1. Weigh 1.000 g of oven-dry soil, 300 mesh size (0.05 mm sieve opening) into a 100 mL Teflon beaker (Weigh 1.000-5.000 g of organic soil).
2. Add 10 mL of conc. HNO_3, cover, boil gently for about 30 minutes on a hot late at 100-150˚ C, allow contents to cool.

   NOTE: Treatment with HNO_3 to destroy organic materials is required to prevent danger of an explosion by reaction with $HClO_4$.
3. In a stainless steel fume hood, add 5 mL $HClO_4$ and 15 mL HF, cover, and boil gently for 60 minutes on a hot plate at 150-225˚ C.
4. Remove cover and continue to boil until the volume is reduced to 2-3 mL.

   NOTE: It is important not to dry the contents completely.
5. Cool and wash down with 5-10 mL of deionized water, cover and bring to a boil for about 20 minutes.

   NOTE: If a precipitate is present, filter through a Whatman GF/B filter. Filtration is desirable to keep the solution free of solid particles that cause clogging of burner capillaries of the atomic absorption spectrometer.
6. Wash sample solution into volumetric flask and bring up to volume. Transfer the contents into 100 mL polypropylene bottles for storage (until determination of elements is made).

   NOTE: It is recommended that a 48-hour soak with 10% HNO_3 be used for both the preliminary cleaning of new bottles and for routine cleaning. (4)

7. Prepare standards of elements by dilution of stock solution with the same matrix solution as the sample.

8. Determine elements by atomic absorption spectrometry.

   NOTE: Prepare and analyze a reagent blank.

CALCULATIONS

$$\text{ug/g in soil} = \frac{\text{Reading in ug/mL x 50}}{\text{wt. of sample (g)}}$$

$$\% = \frac{\text{Reading in ug/mL x 50}}{\text{wt of sample (g) x 10 000}}$$

ERROR VALUES

	n	$\overline{X}$	SD	RSD(%)
Fe (%)	5	1.79	.12	6.7
Mn (ug/g)	5	423	16	3.7
Zn (ug/g)	5	54	3	5.6
Cu (ug/g)	5	12	1	8.3

REFERENCES

1. BAJO, S. 1978. Volatilization of Arsenic (III,V), Antimiony (III.V) and Selenium (IV.VI) from mixtures of hydrogen fluoride and perchloric acid solution; application to silicate analysis. Anal. Chem. 50: 649-651.

2. LAXEN, D.P.H. and HARRISON, R.M. 1981. Cleaning methods for polythene containers prior to the determination of trace metals in freshwater samples. Anal. Chem. 53: 345-350.

3. LEVESQUE, M. 1966. A rapid digestion method for total iron and aluminum in soil. Can. J. Soil Sci. 46: 205-206.

4. PAWLUK, S. 1967. Soil analyses by atomic absorption spectrophotometry. Atomic absorption Newsletter. 6: 53-56.

EXTRACTION AND DETERMINATION OF Fe, Al, AND Mn

Extraction by dithionite-citrate-bicarbonate, acid ammonium oxalate, and sodium pyrophosphate provide a basis for an approximate differentiation of the forms of Fe, and to a lesser degree, or Al and Mn in soils.

I DITHIONITE-CITRATE-BICARBONATE EXTRACTABLE Fe, Al AND Mn (1)

This extraction removes finely-divided hematite (Fe_2O_3) and goethite ($HFeO_2$) amorphous inorganic Fe and Al oxides and organic-complexed Fe and Al. Only small amounts of Fe and Al from silicates are removed. The extraction provides an estimate of “free” (non-silicate) Fe in soils, but sand-sized goethite and hematite, and magnetite are not dissolved.

EQUIPMENT

Constant temperature water bath

Centrifuge with head for 15 mL centrifuge tubes

Centrifuge tubes, 15 mL, graduated

Volumetric flasks, 100 mL

Stirring rods

Vortex mixer or shaker

REAGENTS

Sodium dithionite powder ($Na_2S_2O_4$)

Sodium chloride (NaCl), saturated solution

Citrate-bicarbonate solution:

- mix 500 mL of 1M sodium bicarbonate solution (42 g of $NaHCO_3$ in 500 mL) with a 4 L 0.3M sodium citrate dihydrate (353 g of $Na_3C_6H_5O_7$. 2 H_2O in 4 L).

PROCEDURE

1. Weigh 0.500 g of soil ground to pass a No. 100 (.15 mm opening) sieve into a 15 mL centrifuge tube (use 0.200 g for clays and 1.000 g for coarse textured soils).
2. Add citrate-bicarbonate buffer to the 5 mL mark.
3. Add about 0.2 g of $Na_2S_2O_4$ (use a calibrated scoop), mix well with a stirring rod and place the tube into a water bath at 80° C. Stir every 2-3 minutes throughout a 15 minute extraction period.

   NOTE: Avoid overheating as FeS forms at higher temperature.
4. Remove the tube from the bath, add 1 mL of saturated NaCl solution and mix. Wash off stirring rod, adding wash to the tube.
5. Centrifuge for about 5 minutes at 1500-1600 rpm. Pour off the centrifugate into a 100 mL volumetric flask.
6. Repeat the extraction (steps 2-5). A shaker or vortex mixer can be used to loosen the soil cake after centrifugation.
7. Wash the soil twice with 5 mL of citrate-bicarbonate buffer solution and 1 mL of saturated NaCl, centrifuge after mixing add the wash solutions to the volumetric flask.

8. Bring the extract to volume with distilled water, mix, and save for Fe, Al and Mn analysis.

REFERENCES

1. MEHRA, O.P. and JACKSON, M.I. 1960. Iron oxide removal from soils and clays by a dithionite-citrate system buffered with sodium bicarbonate. 7th Nat'l. Conf. Clays and Clay Minerals. pp. 317-327.

II ACID AMMONIUM OXALATE EXTRACTABLE Fe, Al and Mn

Acid ammonium oxalate extraction removes amorphous inorganic Fe and Al oxides, and organic-complexed Fe and Al from soils. Silicate minerals (except olivine), goethite and hematite are only slightly affected; however, a considerable amount of magnetite (Fe_3O_4) is extracted. Some crystalline oxide forms of Mn are also extracted by this procedure.

EQUIPMENT

Centrifuge with head for 15 mL centrifuge tubes

Centrifuge tubes, 15 mL

Shaker

REAGENTS

Acid ammonium oxalate solution

(A) Prepare a 0.2M solution of ammonium oxalate [$(NH_4)_2\ C_2O_4\ .\ H_2O$].

- dissolve 28.3 g and dilute to 1 L.

(B) Prepare a 0.2M solution of oxalic acid ($H_2C_2O_4\ .\ 2\ H_2\ O$).

- dissolve 25.2 g and dilute to 1 L.
- mix about 700 mL of A and 535 mL of B, check pH and adjust to 3.0 by adding either A or B.

PROCEDURE

1. Weigh 0.250 g of soil ground to pass a No. 100 (.15 mm opening) sieve into a 15 mL centrifuge tube (use 0.125 g for soils with more than 2% extractable Fe or Al).
2. Add 10 mL of acid ammonium oxalate solution and stopper the tube tightly.
3. Place the tube horizontally in a covered box and shake for 4 hours (the extraction must be done in the dark.)
4. Centrifuge for about 10 minutes at 1500 – 1600 rpm; decant the clear centrifugate into a suitable container and store for analysis.

REFERENCES

1. BLUME, H.P. and SCHWARTZMANN, V. 1969. Genetic evaluation of profile distribution of aluminum, iron and manganese oxides. Soil Sci. Soc. Am. Proc. 33:438-444.
2. McKEAGUE, J.A. and DAY, J.H. 1966. Dithionite- and oxalate-extractable Fe and Al as aids in differentiating various classes of soils. Can. J. Soil Sci. 46: 13-22.

III SODIUM PYROPHOSPHATE EXTRACTABLE Fe, Al and Mn

Sodium pyrophosphate extracts organic-complexed Fe and Al from soils. The removal of crystalline, amorphous and silicate Fe and Al is slight. (2,3)

EQUIPMENT

Centrifuge, high speed

Plastic centrifuge tubes, 50 mL

Shake

REAGENTS

Sodium pyrophosphate decahydrate solution ($Na_4P_2O_7.10H_2O$) 0.1M

- dissolve 44.6 g of $Na_4P_2O_7.10H_2O$ in distilled water and dilute to 1 L.

SUPERFLOC – flocculating agent, 0.1% in water.

- prepare by shaking 0.10 g in 100 mL of distilled water (intermittently) for several days to bring into solution.

PROCEDURE

1. Weigh 0.300 g of soil ground to pass a No. 100 (.15 mm opening) sieve into a 50 mL plastic centrifuge tube (use 1.000 g for samples low in extractable Fe and Al).
2. Add 30 mL of 0.1M Sodium pyrophosphate solution, stopper and shake overnight.

3. Centrifuge at 20,000 x gravity or more for 10minutes with high speed centrifuge or add 0.5 mL of 0.1% SUPERFLOC and centrifuge at 1500 rpm for 10 minutes. (4)

   NOTE: A comparison of SUPERFLOC versus high speed centrifugation has also been reported by Ballantyne et al. (1)

4. Decant a portion of the clear centrifugate into a container and store for analysis.

REFERENCES

1. BALLANTYNE, A.K., ANDERSON, D.W. and STONEHOUSE, H.B. 1980. Problems associated with extracting Fe and Al from Saskatchewan soils by pyrophosphate and low speed centrifugation. Can. J. Soil Sci. 690: 141-143.
2. BASCOMB, C.L. 1968. Distribution of pyrophosphate-extractable iron and organic carbon in soils of various groups. J. Soil Sci. 19: 251-268.
3. McKEAGUE, J.A. 1967. An evaluation of 0.1M pyrophosphate and pyrophosphate-dithionite in comparison with oxalate as extractants of the accumulation products in Podzols and some other soils. Can. J. Soil Sci. 47: 95-99.
4. SHELDRICK, B.H. and McKEAGUE, J.A. 1975. A comparison of extractable Fe and Al data using methods followed in the U.S.A. and Canada. Can. J. Soil Sci. 55: 77-78.

IV DETERMINATION OF Fe, Al and Mn BY ATOMIC ABSORPTION SPECTROMETRY

EQUIPMENT

Atomic absorption spectrometer (IL-257)

Volumetric flasks, 50 mL and 100 mL

Adjustable micro-pipets and disposable tips

REAGENTS

Extracting solutions

- dithionite-citrate-bicarbonate
- acid ammonium oxalate
- sodium pyrophosphate

Certified atomic absorption standards

- 1000 ug/mL solution of Fe, Al and Mn

PROCEDURE

1. Filter extract if suspended material is present.
2. Pipet an aliquot of sample extract into a 50 mL volumetric flask and make up to volume with extracting solution.
3. Prepare a blank and a range of standards using the appropriate extracting solution.

4. Adjust the atomic absorption spectrometer controls and settings for the element to be analyzed, as recommended by the manufacturer.

   NOTE: An air-acetylene flame is suitable for the determination of Fe and Mn, and a nitrous oxide-acetylene flame is used for Al.

5. Analyze sample and record result in % for Fe and Al, and in ug/g for Mn.

CALCULTIONS

$$\text{Fe (\%)} = \frac{\text{ug/mL Fe x d.f.}}{\text{10,000 x sample wt. (g)}}$$

$$\text{Al (\%)} = \frac{\text{ug/mL Al x d.f.}}{\text{10,000 x sample wt. (g)}}$$

$$\text{Mn (ug/g)} = \frac{\text{ug/mL Mn x d.f.}}{\text{sample wt. (g)}}$$

d.f. = dilution factor

ERROR VALUES

			n	$\overline{X}$	SD	RSD(%)
Dithionite-citrate-bicarbonate	Fe	(%)	3	.76	.02	2.7
	Al		3	.11	.006	5.4
Acid ammonium oxalate	Fe	(%)	3	.24	.01	4.8
	Al		3	.15	.006	3.8
Sodium pyrophosphate	Fe	(%)	3	.35	.02	6.7
	Al		3	.40	.01	2.4

APPENDIX I **UNITS OF MEASURE**

UNIT	METRIC EQUIVALENT	ENGLISH EQUIVALENT	S.I.UNIT
LENGTH			
1 M	metre (m)	3.28 ft	metre (m)
10^{-1}m	decimetre (dm)	3.937 in.	decimetre (dm)
10^{-2}m	centimetre (cm)	0.394 in.	centimetre (cm)
10^{-3}m	millimetre (mm)	3.9 x 10^{-2}in.	millimetre (mm)
10^{-6} m	micron (u)	3.9 x 10^{-5} in	micrometre (um)
10^{-9} m	millimicron (mu)	3.9 x 10^{-8} in	nanometre (nm)
10^{-10}m	Ångstrom (Å)	3.9 x 10^{-9} in	0.1 nm
VOLUME			
$10^{-3}m^3$	litre (L)	61.023 cu. in	litre (L)
$10^{-6}m^3$	millilitre (mL)	6.1 x 10^{-2} cu. in.	litre (L)
$10^{-9}m^3$	microlitre (uL)	6.1 x 10^{-5} cu. in.	microliter (uL)
WEIGHT			
1 kg	Kilogram (kg)	2.20 lb avdp.	Kilogramme (kg)
10^{-3}kg	gram (g)	3.53 x10^{-2} oz. avpd.	gramme (g)
10^{-6}kg	milligram (mg)	3.53 x 10^{-5}oz.avdp.	milligramme (mg)
10^{-9}kg	microgram (ug)	3.53 x 10^{-8} oz.avdp.	microgramme (ug)
10^{-12}kg	millimicrogram (mug)	3.53 x 10^{-11}oz.avdp.	nanogramme (ng)

ELECTRICAL CONDUCTANCE

-measured in millisiemens/centimetre (mS/cm), which is analogous to mmhos/cm.

-under S.I. usually measured in decisiemens/metre (dS/m).

ELECTRICAL POTENTIAL

-measured in millivolts; under S.I, measured in kg m^2 s^{-3} A^{-1}.

CATION EXCHANGE CAPACITY (CEC)

-measured in milliequivalents per 100 grams (meq/100 g)

-under S.I., measured in mol kg^{-1}.

APPENDIX II SYSTEME INTERNATIONAL (S.I.) OF MEASURES

BASIC UNITS

QUANTITY	NAME	SYMBOL
Length	Metre	m
Mass	Kilogramme	kg
Time	Second	s
Temperature	Kelvin	K
Electric Current	Ampere	A

MULTIPLES

Tera	(T)	10^{12}	Deci	(d)	10^{-1}
Mega	(M)	10^{6}	Centi	(c)	10^{-2}
Kilo	(k)	10^{3}	Milli	(m)	10^{-3}
Deca	(da)	10^{1}	Micro	(u)	10^{-6}
Nano	(n)	10^{-9}			

DERIVED S.I. UNITS

QUANTITY	S.I. UNITS	S.I.NAME	NON-S.I. NAME
Density	kg m^{-3}	--	g/cc
Pressure	kg m^{-1} s^{-2}	pascal (Pa)	atmospheres, bars, psi
Concentration	mol m^{-3}	--	g/L
Conductivity	kg^{-1} m^{-3} s^{3} A^{2}	Siemens/metre	mmhos/cm
Molality	mol kg^{-1}	--	moles/1000 g
Volume	m^{3}	--	litres (L)

Elec. Potential	$kg\ m^2\ s^{-3}\ A^{-1}$	--	millivolts (mv)
CEC	$mol\ kg^{-1}$	--	meq/100 g
Elec. Conductance	$kg^{-1}\ m^{-2}\ s^3\ A^2$	Siemens	mho
Avagadro Constant	mol^{-1}	--	atoms/gram-atom, ions/gram-ion

APPENDIX III

CONCENTRATION OF SOLUTIONS

1. MOLARITY (M) = No. of gram-molecular weights (mole) of solute per litre solution.

$$M = \frac{\text{No. moles solute}}{\text{Litres solution}} \quad \text{OR} \quad M = \frac{\text{grams solute} \div \text{mol. Wt. solute}}{\text{litres solution}}$$

2. NORMALITY (N) = No. of gram-equivalent weights of solute per litre of solution.

$$N = \frac{\text{No. g-eq. wt. solute}}{\text{Litres solution}} \quad \text{OR} \quad N = \frac{\text{g solute} \div \text{g-eq. wt. solute}}{\text{litres solution}}$$

PROPOSED S.I. UNITS

- Concepts of Normality, equivalent weights, and litres will no longer be used in determining concentration.

VOLUME

mL = cm^3 /mL = cm^{-3}

cc = cm^3

L = dm^3 /L = dm^{-3}

(eg.) - standard solution of NaCl (2.314 g/L) = 2.314 dm^{-3}

PARTS PER MILLION

- for solutions : ppm = ug cm^{-3}
- for solids : ppm = ug g^{-1}

MOLES

-for monovalent cations : 1 milliequivalent (meq) = 1 millimole (mmol).

APPENDIX IV

CONVERSION OF PHYSICAL AND CHEMICAL PROPERTIES TO S.I. UNITS

QUANTITY	PRESENT UNITS	FACTOR	S.I. UNITS
1. Bulk Density	g/cc	x 10^3	kg m^{-1}
	Eg 1.50 g/cc	⟶	1500 kg m^{-1}
2. CATION EXHANGE CAPACITY (CEC) -monovalent	meq/100 g	x 10^{-2} ⟶	mol kg^{-1}
	(eg) 1.50 g/cc	⟶	0.500 mol kg^{-1}
-divalent	meq/100 g	5 x 10^{-3} ⟶	mol kg^{-1}
	(eg) 50 meq/100 b	⟶	0.250 mol kg^{-1}
3. CATIONS		---- SAME AS CEC	
4. EXTRACTABLE ACIDITY			
5. ELECTRICAL CONDUCTANCE	mS/cm	x 1 ⟶	dS m^{-1}
	mS/cm	x 10^{-1} ⟶	S m^{-1}
6. SOLUBLE SALTS monovalent	meq/1	x 10^{-3} ⟶	mol dm^{-3}
divalent	meq/1	5 x 10^{-4} ⟶	mol dm^{-3}

APPENDIX V

SIZE OF SIEVE OPENINGS IN mm AND MESH

OPENINGS IN mm	MESH No.
2.00	10
1.00	18
0.50	35
0.42	40
0.25	60
0.21	70
0.18	80
0.15	100
0.11	140
0.080	170
0.070	200
0.060	230
0.053	270
0.050	300

APPENDIX VI

COARSE FRAGMENT CONVERSTION FROM % BY VOLUME TO % BY WEIGHT

Percent, By volume	Percent, by weight			
	Bulk density of soil			
	1.3	1.5	1.7	2.0
10	19	17	15	13
15	27	25	22	19
20	34	31	28	25
25	41	38	34	31
30	47	44	40	37
35	53	50	46	42
40	58	55	51	47
45	63	60	56	52
50	68	64	61	57
55	72	69	66	62
60	76	73	70	67
65	80	77	75	71
70	83	81	79	76

1. Using formula $\frac{2.7G}{B.D.\ (100-G) + 2.7G} \times 100$:

Where G is percent of coarse fragments (by volume) and 2.7 is assumed

average specific gravity of coarse fragments and B.D. is bulk density of fine earth fraction (less than 2 mm).

For bulk densities other than those above, the approximate adjustment is 3% for each bulk density change of 0.2 g/cc in the soil.

Similar adjustment for coarse fragment densities other than 2.7 (granitic equivalents) can be made; adjust the basic formula to the appropriate coarse fragment density.

To use this table for conversion of material coarser than 3 inches, appropriate adjustment, generally upward, must be made in the assumed bulk density to include material, if any, between 2 mm and 3 inches.