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कृषि मन्त्र विद्यालय

Practical Manual

on

Fundamentals to Soil Science

Prepared by :

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Department of Soil Science & Agricultural Chemistry

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Exercise No.1

Title : Collection and preparation of soil for analysis

Objective : Collection and preparation of soil samples for analysis of soil parameters.

The first step of soil testing is collection of soil sample. Sampling is essential as the testing of whole farm soil is not possible. Satisfactory results cannot be obtained by analyzing the sample of the later if it is not a true representative of the farm area. The success of soil testing program depends upon the soil sample. There is an axiom "Soil Testing cannot be better than Soil Sampling". Reliable results can only be obtained by carefully collected samples. Otherwise the results will not be reliable. Collection of a perfect sample is a difficult job and therefore it is necessary to observe the following.

Time of Sample Collection

Soil can be tested round the year but summer season is most appropriate for this work. The condition of farms is ideal for sample collection in this season, and the results of testing can be obtained before the monsoon crop.

Sampling Area and Number of Samples

If all the conditions are uniform then the sample area should not exceed two hectares. Sample should be prepared by collecting soil from 6-8 sites per acre. Following precautions should be taken for site selection.

- Sample area should be divided into small uniform units according to soil color, topography, previous crop, type and quantity of fertilizers used, slope and erosion. Make sure that soil is collected from each of these units.
- Soil should not be collected from places close to irrigation channels, manure pits, bunds and below trees.

Sampling Equipments

Different type of equipments can be used for sampling. Soil auger or tube auger, garden hoe or spade, knife, clean bucket or shallow pan and cloth or polythene bags are used for sampling. Tube auger is used for soft moist soils, whereas, screw auger for hard and dry soils. Such augers are most useful for sampling because a uniform portion of soil from a particular depth can be drawn with accuracy and in less time. If these are not available, samples can be taken with a garden hoe or a spade but care should be taken that sampling depth and quantity of soil should be uniform at every site.

Method of Sample Collection

Clean the site from where soil sample is to be collected by removing un-decomposed organic materials, garbage, pebbles etc. (Taking care that the surface soil is not removed). Then insert the tube auger into the soil by rotating it and when it penetrates 15 cm inside then pull it out slowly. Collect the soil from the auger into a clean pan using a knife. When auger is not available, a 15 cm deep V-shaped pit is made with the help of a spade or garden hoe and soil is removed to expose the pit. Take out at least ½ inch thick soil slice (like bread-slice) from both the exposed surfaces of the V-shaped pit from top to bottom and remove the soil from the pit. While sampling empty fertilizer bags should never be used for collection of soil samples.

Collect the soil from every site and crush the large clods by hand or with the help of wooden plank followed by thorough mixing and making a cone-shaped heap. Divide this cone in four equal parts to obtain four quarters. Take two diagonally opposite quarters and mix thoroughly discarding the remaining two quarters. This process is repeated until 500 g soil is obtained.

Precautions in collection and storing of samples

Care in handling the soil samples against contamination is extremely important. Special precautions are necessary if the samples are to be tested for micronutrients also. Any possibility of contact with chemicals, fertilizers or manures must be avoided. Cotton, jute or plastic bags which had previously been containing fertilizer, salt or lime should not be used at any stage. Soil samples should preferably be stored in clean cloth or polythene bags. Glass, porcelain or polythene jars are useful for keeping special samples for long duration research purpose. The bags can be re-used only after thoroughly washing and drying.

Information sheet

In addition to the location, field number, name of cultivator and identification marks, relevant information regarding slope, drainage, irrigation, previous cropping history, fertilizer use etc. of the field(s), must be recorded. Prepare two copies of this information and keep one copy inside the bag with soil sample and tie the bag with another copy with the help of thread. Send this soil sample bag through VLW of the locality or directly to nearest soil testing laboratory for soil analysis.

Registration of samples

As soon as the samples arrive at the laboratory, these are to be serially registered giving the date and other relevant particulars furnished in the information sheet. The sample register, being the most valued permanent document of the soil testing laboratory, all entries must be meticulously recorded in a systematic manner.

Processing of soil samples for analysis

The air-dry soil is normally passed through 2 mm sieve for analysis. Before sieving, the soil clods should be lightly crushed in wooden pestle and mortar. Plant residue, gravel and other foreign matter retained on the sieve should be removed.

Exercise No. :2

Title : Soil Profile Study

**Objective : 1) To classify and describe soils within the study area.
2) To determine nutrient and carbon contents from the soil surface to C horizons.**

The vertical section of the soil from the surface to the parent material is known as soil profile.

SOIL HORIZONS

Soil profile has a sequence of different layers, known as horizons. A soil horizon may be defined as a layer of soil, approximately parallel to the soil surface, formed due to soil forming processes. The major horizons also called as master horizons are grouped under five heads :

- O horizon** It is comprised of organic matter that form above the mineral soil. It result from litter derived from dead plants and animals. O horizons usually occur in forested areas and are generally absent in cropland and grassland regions.
- A horizon** It is topmost mineral horizon which lies at or near the surface. It contains a mixture of partially decomposed organic matter, which tends to impart a dark color than that of the lower horizons.
- E horizon** It characterize the zone of maximum leaching or the zone of eluviations (e = out) of clay, iron, and aluminum oxides, which leaves resistant minerals, such as quartz, in the sand and silt sizes. An E horizon is generally lighter in colour than the A horizon and is found under the A horizon.
- B horizon** It occurs immediately below the 'E' horizon in which maximum accumulation of materials, such as iron and aluminum oxides and the silicate clays, occurs. In arid and semiarid regions. CaCO_3 , CaSO_4 , and other salts may accumulate.
- C horizon** It is the unconsolidated material underlying the B horizon. C horizon is less weathered than the 'A' and 'B' horizons and consequently has not been subjected to horizon differentiation. It is parent material from which soil formed, sometimes, in the soil of arid and semi-arid regions the accumulation of calcium compounds may take place in the C horizon.

Transition Horizons

These horizons are transitional between the master horizons (O, A, E, B, and C). They may be dominated by properties of one horizon but may also have prominent characteristics of another. Both capital letters are used to designate these horizons e. g. AE, EB, BE, and BC, the dominant horizon, being listed first than the subordinate one. Combination such as A/E are used to designed transition horizons where distinct parts of the horizon have properties of A and other parts have properties of E.

Solum

The 'A', E and B' horizons together are called the solum. This portion of the profile is developed by the soil forming processes and is distinguished from the parent material immediately below it.

A theoretical mineral profile showing the major horizons is described below:

Oi	This horizon consist of the original plant and animal residues only slightly decomposed.
O2	This horizon has residues intermediately decomposed.
Oa	Consist of highly/completely decomposed or wetted organic debris On grass land Oi, Oe, and Oa are usually absent
A	A dark coloured mineral, horizon with organic matter mixed intimately with mineral soil. When soil is ploughed and cultivated, the upper 15-20 cm becomes the furrow slice. This may designated as Ap.
E	A light coloured mineral horizon resulted from leaching (horizon of maximum eluviations)
EB	A transitional layer, often absent . When it is present, it is more like 'E' than 'B'
BE	Also a transitional horizon and absent from most soils. But if present it is more like 'B' than 'E'
B	A horizon of illuviation, maximum accumulation of silicate clay minerals of iron, aluminum and organic matter. In arid region soils , this horizon is often characterised by maximum development of blocky or prismatic structure.
BC	Transitional to C. It may not be present.
C	Unconsolidated parent material similar to that from which the solum has developed. This may come from the bedrock below or it may have been transported from elsewhere and deposited on the bedrock.
R	Consolidated bedrock such as granite, sandstone, limestone etc.

To indicate certain additional characteristics, few suffixes as follows are used with the appropriate horizon designation designation

Ca =	to indicate accumulation of CaCO_3 (in nodular forms)
Cs =	to indicate accumulation of CaSO_4 (in crystalline forms)
Cn =	to indicate accumulation of sesquioxides nodules.
g =	to indicate glaying
h =	to indicate accumulation of illuvial humus.
P =	to indicate effect of pluoughing
Sa =	to indicate accumulation of soluble salts other than of calcium
t =	to indicate illuvial
c =	to sow concretion or nodules

Note:

1. Eluviation is the process of removal of soil material in suspension or in solution from a layer or layers of a soil (wash out)
2. Illuviation is the process of accumulation of material in soil layer carried out from an overlying layer. The material often precipitated from solution of deposited from suspension.

Materials

Digging tools, 1 N HCl, Munsell colour chart, magnifying lens, etc.

EXAMINATION OF SOIL PROFILE

A field trip is required for this exercise in which the examination of profile of agricultural soil will be made. Profile characteristics and evidence of soil forming processes are to be explained through description of the horizons and other soil features. It is not likely that the profile of any one soil will show all of the horizons that are cited in theoretical mineral profile.

Method

1. Select a suitable site representing the normal conditions prevailing in the area. The site should be representative of a sizable area and should be away from field boundaries, roads or rivers. Extremes of relief are also avoided in the selection of sites.
2. Dig a pit of the dimensions normally of 1.2 M long and 1.5 M deep. While digging, care should be taken to expose the vertical face to sunlight and the soil excavated be thrown away to either side. It is important that the side to be examined is vertical and even.
3. Record any special features observed during the process of digging.
4. Identify different horizons, their relative depth and features like colour, texture, structure, calcareousness etc.
5. Collect soil samples horizon wise for laboratory analysis

Note: In the field, colour of horizon may be identified with the help of Munsell colour chart; texture may be determined by feel method; structure by observation and calcareousness by knowing the extent of effervescence on pouring of 1 N HCl from the top of soil profile to bottom.

Observations (Record of field data)

The data in respect of morphological features from soil profile are recorded in the appropriate observation sheet.

Representation of profile data

Observe the soil profile and point out the horizons present. Record the properties such as texture, structure, colour, calcareousness, permeability, slope and pH etc.

Complete the observation sheet from the examination of profile exposed in the field or from laboratory studies of collected soil samples. Soil properties are expressed in standardized terms. In studying the soil profile and the filling of the observation sheet, use following terms.

Terms used for filling in observation sheet while profile study

1. **Horizons** : These are designated by the standard nomenclature wherever possible as A, E, B, C, etc.
2. **Texture** : Sand, Loamy-sand, sandy-loam, Lam, Silt, Sandy clay loam, Clay loam, Silty clay-loam, Sandy clay, Silty clay and Clay.
3. **Structure**: Granular Blocky, Prismatic, Platy, Single grained, Angular-blocky, Subangular blocky, Massive.
4. **Colour** : Measure with the munsell colour charts, descriptive names and Munsell colour notation are recorded e.g. reddish-brown 2.5 YR 5/4
5. **Free lime** : Present (+) Absent (-) (it indicates calcareousness)

6. **Permeability** : Slow, Moderate and Rapid
7. **Mottling** : Present or Absent
8. **Consistence (When dry)** : Loose, Soft, Slightly-hard, Hard, Very hard and Extremely hard.
9. **Consistence (When wet)** : Nonsticky, Slightly sticky ,sticky, very sticky.

OBSERVATION SHEET

Date

B. Description of the profile					Soil Profile No.				
Horizon Depth (cm)	Texture	Consistence	Structure	Permeability	Colour	Mottling	pH	Free lime	Any other

<p>The nature of the soil forming factors Resulting in the formation of this soil are</p> <p>Climate ----- Native vegetation ----- Parent material ----- Topography ----- Time -----</p>	<p>Soil Classification</p> <p>Soil type ----- Great soil Group ----- Geographical distribution -----</p>
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Soil Samples collected are to be brought in the laboratory for analysis of various physico-chemical characteristics such as mechanical analysis, CaCO₃ pH and EC etc. as per requirement.

Exercise No.: 3

Title : Identification of rocks and minerals
Objective : To identify soil forming rocks and minerals

Rocks consist of an assemblage of minerals. They are classified according to this assemblage, the mineral proportions, and the conditions under which the rock was formed. Rocks are formed in three basic ways resulting in the following classes:

- (A) *Igneous rocks*: Formed by hardening or crystallization of high-temperature, molten material (magma).
- (B) *Sedimentary rocks*: Formed by solidification of material deposited by wind, water, glaciers, or direct chemical precipitation.
- (C) *Metamorphic rocks*: Formed by modification of previously existing rocks by the application of heat and / or pressure within the earth.

Rock Identification Procedure

Identification of rock types involves not only identifying their mineral constituents and their relative amounts, but also a series of characteristics of the rock. The three classes of rocks, igneous, sedimentary, and metamorphic, were defined earlier. Because rocks from different classes can appear quite similar, and rocks within the same class can often appear quite different, separation by class will not be a specific part of the identification process.

I. Distinct mineral grains, easily seen by the naked eye.

A. Entire rock surface easily scratched by a knife. (Make sure that the mineral surfaces are being scratched, and not simply that poorly cemented grains are being dislodged.)

1. Reactivity with dilute HCl.

a. Vigorously, with much effervescence - limestone or marble

NOTE: *If visibly crystalline with interlocking grains, the rock in question may be a marble or a limestone. Without larger samples to reveal the presence or absence of characteristic irregular banding ("marbled" appearance) and other textural features, distinction between the two may not be possible. The presence of noninterlocking mineral grains and undistorted fossils confirm the rock is a limestone.*

b. Slowly on the rock surface, vigorously if the rock is powdered - dolomite or dolomitic marble

c. No reaction, greasy or waxy appearance - serpentinite

B. Entire rock surface cannot be scratched by a knife, (that is, some or all of the minerals present are harder than the knife blade).

1. The minerals are intergrown (contact surfaces between minerals are irregular and interlocking).

a. Minerals arranged in layers or bands (foliated).

(1) Irregular, coarse foliation if poorly developed (or lineation), with zones of light minerals (often quartz or feldspar, or both) alternating with zones of dark minerals (often biotite or hornblende). The foliation is not developed to the extent that planes of weakness are present in the rock - gneiss

(2) Minerals arranged in thinner, more distinct layers which create planes of weakness along which the rock may be split. Mica minerals are plentiful - schist

NOTE: *Some sandstones are color banded. However, examination of the mineral grains will show a lack of intergrowth.*

b. Minerals not in distinct layers. (massive).

(1) Chief minerals are the feldspars, with orthoclase >plagioclase; five percent or more quartz; mica minerals or hornblende, or both in small amounts are common - granite

(2) Like granite, except little or no quartz - syenite

(3) Chief minerals are the feldspars, with plagioclase >orthoclase; five percent or more quartz; mica minerals and / or hornblende in small amounts are common - granodiorite

(4) Like granodiorite, except little or no quartz - monzonite.

(5) Mainly plagioclase, with hornblende and some biotite; no quartz; medium to dark color - diorite.

(6) Like diorite, except pyroxene and possibly olivine present instead of hornblende and biotite; color is usually dark - gabbro

NOTE: *Finer grained gabbros are often referred to as diabase. The rather ambiguous term traprock is also used for this rock type.*

(7) Chief minerals are pyroxenes and olivine; pyroxenes >olivine; dark color - pyroxenite

(8) Chief minerals are pyroxenes and olivine; olivine >pyroxenes; dark color - peridotite .

2. Minerals are in distinct grains that are cemented together rather than intergrown.

a. Sand-sized particles, cemented by silica, clay, calcite, or hematite; chief minerals are usually quartz and feldspar; breaks around rather than through the sand grains - sandstone

b. Similar to sandstone in general appearance; quartz is the chief mineral; grain boundaries range from partial to total intergrowth due to secondary quartz crystallization during metamorphism; due to this intergrowth, the rock breaks through rather than around the mineral grains - quartzite

NOTE: *Under higher levels of metamorphism, quartzite would fall under category B.1 .b., above, due to nearly complete mineral intergrowth. It can easily be separated from the other rocks listed under B.1 .b. because the only mineral present in significant amounts is quartz.*

c. Gravel-sized particles of rocks and minerals, cemented by silica, clay, calcite or hematite - conglomerate

II. Very fine mineral grains, not visible to the naked eye.

A. Glassy.

1. Looks like glass; may have a few inclusions or bubbles; dark brown to black - obsidian.

2. Contains many bubbles, frothy - pumice.

B. Dull, earthy or stony.

1. Can be scratched with a knife.

a. Reacts vigorously with dilute HCl - limestone

b. Reacts slowly when whole or vigorously when powdered with dilute HCl - dolomite

c. Reacts slowly or not at all with dilute HCl, whether whole or powdered.

(1) Tends to break into flaky pieces - shale

(2) Layered; breaks into thin, flat sheets - slate

2. Can't be scratched with a knife.

a. Very hard; fractured surface is smooth (may be conchoidal) with sharp edges; surface may appear waxy; tan to black color - chert.

b. Massive; dull-appearing fractured surface; may have small inclusions of glass or crystals.

(1) Light to medium colors - felsite.

NOTE: Felsite includes the extrusive igneous rock types: rhyolite, trachyte, latite, and andesite which usually can't be distinguished by the naked eye. They differ basically only in the relative amounts of the two feldspars (their primary constituents) and the presence or absence of quartz.

(2) Dark to black color - basalt

Identification of major rocks:

(A) Igneous rocks

S. No.	Rock	Colour	Structure	Relative density	Major minerals	Secondary minerals
1	Graphite	Light	Hard	2.64	Quartz, Orthoclase	Hornblende, Mica, Magnetite
2	Synite	Light	Hard	2.80	Quartz, Albite, Orthoclase	Hornblende, Biotite
3.	Diorite	Dark	Hard	2.85	Plagioclase	Hornblende, Biotite, Magnetite
4.	Gabbro	Black	Hard	3.0	Augite, Olivine, Labrodorite	Hornblende, Ilmenite
5.	Basalt	Black	Hard	3.0	Augite, Olivine, Labrodorite	Hornblende, Ilmenite

(B) Sedimentary rocks

S. No.	Rock	Colour	Structure	Mineral Composition
1	Sand stone	Light to red	Granular and porous	Mainly quartz with Calcium carbonate and Iron oxide
2	Shale	Light to dark	Laminar	Clay minerals, quartz
3.	Lime stone	Light brown	Fine granular, shows effervescence with acid	Mainly calcite or dolomite, certain iron oxide, clay, phosphate

(C) Metamorphic rocks

S. No.	Rock	Colour	Structure	Mineral Composition
1	Gneiss	Light to dark	Foliated granular	Made up from granite, mineral composition like granite
2.	Marble	White (if made up from pure lime stone) light red, light green, light black	Non-foliated, compact fine to coarse granular	Mainly calcite and dolomite, small quantity of iron oxide
3	Schist	Same as parent rock	Foliated	Like basalt or shale
4.	Slate	Brown to black	Foliated compact	Like Shale
5.	Quartzite	Light walnut	Non foliated	Like Sand stone

Mineral Identification

Since minerals are the components of rocks, their identification is an integral part of proper rock identification. For this identification procedure, three characteristics of minerals will be of major importance: hardness, reactivity with dilute hydrochloric acid, and cleavage.

Hardness. The knife blade is particularly useful in separating the common harder minerals (quartz and the feldspars) from the common softer minerals (calcite and dolomite). To test for hardness with any of these items, two approaches may be used:

- Use the knife blade (or copper penny, etc.) as a tool to attempt to scratch the mineral
- Use the mineral to attempt to scratch the testing material.

Doing it both ways will often give a clearer indication of the relative hardness of the two materials being compared.

HCl Reactivity

This test serves to differentiate the carbonate minerals (which react with HCl) from other mineral types. The acid used is *dilute* HCl. The dilute acid is obtained by mixing water with full-strength acid. By noting the normality of the acid being diluted, an appropriate volume of water can be used to reach the target of 0.1 N. For instance, if the original acid is 1.0N, increasing the volume of water tenfold will result in a 0.1N. When diluting, *always add the acid to the water to avoid splashing full-strength acid.*

Cleavage. If a mineral breaks so it yields definite plane surfaces, the mineral is said to possess cleavage. A mineral can possess one or more directions of cleavage, or none:

- The micas (e.g. muscovite and biotite) are examples of minerals with distinct cleavage in one direction.
- All the feldspars have two cleavage directions, which are at almost right angles.
- Quartz has no cleavage. This fact helps in the distinction between quartz and the feldspars.

When quartz and chert are broken, the resulting surfaces often have a typical concave shape called *conchoidal fracture* because of its shell-like appearance. While not a cleavage, this distinctive fracture habit can be useful in identification.

Other Characteristics. Some minerals have a distinct, definitive color. However, because the color of most minerals can vary significantly, color should normally be used as supportive rather than primary evidence. Another useful characteristic is a mineral's ability to transmit light. Depending on the composition, crystallography, and other factors, a mineral may be *transparent, translucent, or opaque.*

Mineral Identification Procedure:

Is it harder than a knife?

I. If **YES**, what is its overall appearance?

A. Dull and earthy, waxy, or metallic.

1. Magnetic (small fragments stick to the knife blade) - magnetite.

2. Nonmagnetic, how does it break (fracture)?

a. Sharp edges; conchoidal (concave, like the inside of an oyster shell) surface
- chert

b. Rough, uneven surface

red-brown to black - hematite;

brown to dark brown - limonite.

NOTE: *both hematite and limonite can appear softer than a knife if not tested on a fresh, unweathered surface.*

c. Pale to medium brass color, often in cubic crystals - pyrite.

B. Vitreous (glassy), transparent to translucent.

1. No cleavage.

a. Colorless to white to pale pink - quartz

b. Olive green - olivine.

- c. Red-brown - garnet.
- 2. Two cleavage planes, intersecting at approximately a 90-degree angle.
 - a. Good to perfect cleavage surfaces (feldspar group, one surface with parallel striations)
 - Plagioclase; no striations present
 - orthoclase.
 - b. Poor to fair cleavage surfaces - pyroxene.
 - c. Two cleavage planes, intersecting at 120 and 60 degrees - amphibole (includes hornblende)

II. If NO, will it scratch a copper penny?

A. If yes, will it react with dilute HCl?

- 1. Vigorous reaction - calcite
- 2. Minor reaction when whole, vigorous when powdered - dolomite.
- 3. No reaction.
 - a. One plane of perfect cleavage - fluorite.
 - b. plane of poor cleavage - apatite.
 - c. Non-crystalline; waxy to greasy or fibrous appearance - serpentine.

B. If no, does it have perfect cleavage which allows splitting into thin sheets?

- 1. Yes - mica group.
 - a. Pale, light colors, sheets are flexible and elastic - muscovite
 - Usually in very small flakes; sheets are flexible but not elastic - chlorite.
 - b. Dark colors, green to brown to black - biotite

2. No.

- a. Opaque, very fine grained - clay minerals.
- b. Translucent to transparent - gypsum.

Identification of minerals

Mineral	Chemical composition & Name	Relative density	Streak	Hardness (Mohr's Scale)	Cleavage	Lusture	Colour	Structure
Apatite	$\text{Ca}_5(\text{F, Cl OH})(\text{PO}_4)_3$ Calcium Flu phosphate	3.15-3.2	White	5	Bad	Glassy	Greenish walnut red	Solid, granular
Augite	$\text{Ca}(\text{Mg, Fe, Al})(\text{Al, Si})_2\text{O}_6$ Ferro magnesium silicate	3.2-3.4	Greenish-brown	5-6	Complete prism	Glassy	Dark green to black	Crystalline granular, small crystals
Dolomite	$\text{CaMg}(\text{CO}_3)_2$ Calcium Magnesium Carbonate	2.85	Colorless	3.5-4	In three direction at 73°	Glassy and Pearl like	Pinkish like meat, may be white, brown, or green	Tetrahedra crystalline, coarse granular
Calcite	CaCO_3 Calcium carbonate	2.72	Colorless	3	In three direction at 73°	Glassy	Generally white or colourless	Crystalline, Coarse to fine aggregated granular
Gypsum	$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ Calcium Sulphate dihydrate	2.32	Black	2	Good	Glassy, Pearly or silky	Colorless, white or brown full of impurities	Prism like crystal
Biotite	$\text{K}(\text{Mg, Fe})_3 \text{AlSi}_3\text{O}_{10}(\text{OH})_2$ Ferro magnesium silicate	2.8-3.2	Colorless	2.5-3	Thin transparent sheet	Pearly and glassy	Black, dark green	Irregular foliated sometimes crystalline
Orthoclase	$\text{K}(\text{AlSiO}_8)$ Potassic feldspar	2.57	White	6	Fibrous	Glassy	White, brown, meat like pink	Crystalline prism

Muscovite	$KAl_3Si_3O_{10}(OH)_2$ Non-ferro magnisium silicate	2.76-3.1	Colourless	2-2.5	Good	Glassy, Pearly and silky	Light yellow, colourless	Thin flakes
Hematite	Fe_2O_3 Iron oxide	5.26	Light to dark red on heating black	5.5-6.5	Irregular flakes	Metallic	Red to black	Mica like and foliated
Magnetite	Fe_3O_4 Iron oxide	5.18	Black	6	Octahedral	Metallic	Black	Solid granular
Pyrite	FeS_2 Iron Sulfide	5.02	Light green to walnut black	6-6.5	Irregular flakes	Metallic	Yellow	Crystalline solid
Quartz	SiO_2 Silicon oxide	2.65	Colourless	7	Round flakes	Glassy, Oily and Metallic	White	Crystalline prism
Olivine	$(Mg, Fe)_2SiO_4$ Ferro magnesium silicate	3.27-3.37	Yellow, Green, white	6.5-7	Round Flakes	Glassy	Green and brown	Mostly granular
Hornblende	Composed of Ca, Na, Mg, Ti, and Al Ferro-magnesium silicate	3.2	Colorless	5-6	Complete prism at 56° and 124°	Glassy fibrous	Dark green to black	Long crystalline prism, fibrous and granular

Observation sheet: A. Rocks

- (i) Sample No.
- (ii) Colour
- (iii) Relative density
- (iv) Structure-
- (v) Rock identified

Observation sheet: B. Minerals

- (i) Sample No.
- (ii) Colour
- (iii) Relative density
- (iv) Structure-
- (v) Streak
- (vi) Hardness
- (vii) Lusture
- (viii) Cleavage
- Mineral Identified

Exercise No.: 4

Title : Determination of Bulk density and Particle density of soils
Objective : To determine bulk and true density of soil

A. Bulk density determination by core method

Equipment: Surface and depth soil core sampling kit with boxes to hold the brass cylinders containing soil, plain edge 10 cm. blade sharp knife, polyethylene bags, labels, marking pencil and rope.

Procedure

The core sampler consists of a soil core cutter which holds a unit of sectional cylinder to retain the soil. The core cutter is forced into the soil by the use of a hammer. Select a leveled area, clear the soil surface and place the soil core cutter along with collar in position. Drive the core cutter with the help of a hammer in the soil until the edge of the collar comes to rest on the soil surface. Dig around the core cutter with the help of a hand hoe or spade. To remove the sample, place the hand hoe below the core cutter and lift it. Open the collar and remove the sectional brass cylinder with the soil. Add a little water on both sides in case the soil is loose and dry.

Place the sectional cylinder in the polyethylene bag along with a label indicating location and depth. Tie another label recorded with location and depth on the polyethylene bag and place the sample in the box. Similarly collect other surface soil samples.

In Laboratory, take out sectional cylinders from the box at a time. Remove the polyethylene bags and one of the 2 mm sections. Shave off the excess soil slowly to bring it in level with the top of 8 cm ring. Now hold the cylinder carefully and remove the other 2 cm. section and shave off the excess soil slowly, without disturbing the soil in the 8 cm. ring and bring it in level with the edge of 8 cm. ring. Place the 8 cm. ring along with the soil in numbered moisture can. Note the number of moisture can along with depth and location. Dry the soil in oven at 105°C for 24 hour. Then weigh the moisture can along with soil and ring. Remove the soil, wash the ring and moisture can and weigh it. Determine the oven dry weight of soil. Calculate the volume of 8 cm. ring and then determine the bulk density.

$$\text{Bulk density (Mg m}^{-3}\text{)} = \frac{\text{Wt. of oven dry soil in 8 cm ring (g)}}{\text{Volume of 8 cm ring (cm}^3\text{)}}$$

Observations

Weight of oven dry soil & ring + moisture can = X g

Weight of ring + Moisture can = Y g

Weight of oven dry soil = (X-Y) g

Radius of Ring = r cm

Length of Ring = L cm

Volume of Ring = $V = \pi r^2 L$ (cm³)

Calculations

$$\text{Bulk density} = \frac{(X-Y) \text{ g}}{V \text{ cm}^3}$$

Observation sheet

Weight of oven dry soil & ring + moisture can =g
Weight of ring + Moisture can = g
Weight of oven dry soil = g
Radius of Ring (r) = cm
Length of Ring (L) = cm
Volume of Ring (V) =cm³

$$\text{Bulk density} = \frac{(X-Y) \text{ g}}{V \text{ cm}^3} = \text{-----Mg/m}^3$$

B. Particle density determination by pycnometer

A ratio of the total mass of soil solids to their total volume excluding pore spaces between particles is known as particle density of soil. It is expressed as Mg/m³ (Mega grams per cubic meter). Interrelationships of porosity, bulk density and air space and rates of sedimentation of particles in fluids depends on particle density.

Principle

Particle density (Pd) determination requires the measurement of mass of the solid and its volume. The mass is obtained by weighing the oven dry sample and volume by calculating from the mass and density of water displaced by sample in a pycnometer or relative density bottle. (See figure). The pycnometer is a glass flask with a ground glass stopper having capillary opening.

Materials/Equipments required

1. Pycnometer (Specific gravity bottle)
2. Weighing balance
3. Distilled water

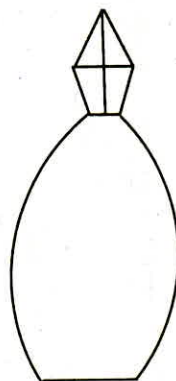


Fig: A 100 ml pycnometer for determination of particle density of soils

Procedure

1. Weigh a clean, dry pycnometer with stopper (W_p).
2. Weigh 10 g of soil and fill it in to the empty pycnometer
3. Carefully clean the outside and neck of the pycnometer so that any soil that may adhered should removed.
4. Weigh the pycnometer including stopper with soil (W_s).

5. Fill the pycnometer about half with distilled water carefully washing inside of the neck to bring down soil adhered on inside of the neck of pycnometer.
6. Gently boil the water (using gas stove) inside pycnometer for several minutes to remove entrapped air.
(Alternatively water can be boiled separately and that boiled water is filled to half in the pycnometer).
7. Cool the pycnometer and its contents at room temperature.
8. Now fill the pycnometer with cooled boiled distilled water.
9. Insert the stopper carefully.
10. Using a clean dry cloth wipe out outer side of the pycnometer and weigh it (W_{sw}).
11. Remove the contents from the pycnometer.
12. Fill cooled distilled water and wipe out outer side of the pycnometer with a clean dry cloth.
13. Now again weigh the pycnometer with water.

Observation sheet

- | | | |
|----|--|---|
| A. | Weight of pycnometer (W_p) g | = |
| B. | Weight of pycnometer + 20 g soil (W_s) g | = |
| C. | Weight of pycnometer + soil + water (W_{sw}) g | = |
| D. | Weight of pycnometer + water (W_w) g | = |

Calculation

$$P_d = \rho_w (W_s - W_p) / (W_s - W_p) - (W_{sw} - W_w)$$

Where, ρ_w = density of water

Exercise No: 5

Title : Soil aggregate analysis by wet sieving method

Objective : To determine water stable aggregates

An aggregate can be defined as a group of two or more primary particles which cohere together more strongly than the surrounding mass. This coherence together more strongly than the surrounding mass. This coherence of primary particle is due to the lowering of electrokinetic (Zeta) potential, resulting in the formation of a floccules. Cementation or binding together of these flocculated mass by certain agent, ultimately results in the formation of aggregates. These agents of cementation include clay to clay cementation. Pre-humic constituents like gums and polysaccharides, resistant and stable humus, fungal mycelia, inorganic polyvalent captions. The type and extent of cementing material determines the stability of an aggregate. Apart from these cementing agents, some physical process like shrinkage and swelling (wetting and drying), freezing and thawing etc. also affect the aggregate formation.

Significance of soil aggregation

The distribution and arrangement of aggregates in a given soil is the cause of its porous nature. Though soil structure as such is not an essential factor for plant growth, but it modifies soil physical environment for plant growth in various ways. Total porosity and pore size distribution are closely associated with the aggregate size distribution which in turn affects the entry and movement of fluids (air and water) in soil. A uniform distribution of both macro and micropores allows an even air water relationship giving required physical environment for the crop roots to grow. It is the size distribution of aggregates that determines the amount of macropores persisting in the soil.

Aggregate stability which refers to the resistance of the soil aggregates towards the disintegration influences of water and mechanical manipulations. The severity of soil crusting by rainfall drops depends on how stable aggregates are. Stability of aggregates can be characterized against wetting, impact of falling raindrops, disintegration during leaching with sodium salt. Aggregate stability is significantly correlated with crop yields when other conditions are equal. It is, therefore, one of the better tests for evaluating the physical condition of the soil.

The size and stability of aggregates also determines the erodibility of a soils i.e. susceptibility of soil particles to detachment and their subsequent transportation by wind and water. More stable and large the aggregates more will be the resistance of these towards the detachment and transportation of soil particles by wind and water.

Aggregate analysis

An aggregate analysis aims to measure the percentage of water stable secondary particles in the soil and the extent to which the finer mechanical separates are aggregated into coarse fractions. The choice of conditions under which the aggregate size distributions are to be measured is largely determined by the purpose of the analysis. Since the purposes of analysis have often been different, a variety of procedures have been used. In most cases a measure is made of the amount of a given sample that resists breakdown in an arbitrary water treatment. The treatment may be stirring, shaking or impact of water drops on the sample. All measures are relative, but none meets the requirement of all investigators,. A few basic and most commonly used methods of aggregate analysis are described below.

- (a) **Measurement of dry aggregate:** Mechanical disruption during the course of preparing and sieving the sample is used to break the soil mass into aggregates. The various size fractions are collected on screens of successively smaller size, and the amount in each fraction is determined by weighing.

Apparatus : A rotary sieve machine, concentric sieves of various sizes of pores to fit properly into the machine, balance.

Procedure

- Arrange a nest of sieves of various sizes (2,1,0.5,0.25 and 0.1 mm) in descending order with the bottom sieve fitted with a pan of same size.
- Keep 50 g of soil sample on the top sieve and cover the sieve with a pan of same size.
- Fit the whole nests of sieves on the rotary-sieve shaker and bolt it. Turn on the machine for 5-10 minutes.
- Collect dry aggregates on various sieves separately and weigh these.
- Separate the stones on each sieve by thoroughly grounding the aggregates in water and pass through the same sieve. The stones of that size will be caught on that sieve itself.
- Dry the remaining soil water suspension in an oven at 105⁰C to 110⁰C for 24 h and weigh it.
- Determine the moisture content of the composite sample used by keeping a part of it in an oven for 24 h at 105⁰C. Thus the whole soil sample taken for sieving can be converted on oven dry weight basis.
- Subtract total amount of stones from weight of aggregates on each sieve to get actual amount of different aggregate fractions.

Precautions

- Samples for analysis should be taken when the soil is reasonably dry to avoid breakdown or change in structure.
- Air over dry samples should be used for analysis.
- Same depth of sampling must be used for the sake of comparison of results.

(b) **Measurement of wet aggregates (wet sieving) :** The stability of aggregates is of more importance than simple their size. Soil aggregates under natural conditions have to undergo considerable disruption by irrigation and rain water. The size of aggregates after this disruption is apparently an important parameter. Aggregates that do not break down in very small units on wetting are said to be water stable. Thus water stability aggregates will determine the stability of soil structure even after being wetted by rain/irrigation water.

This method therefore, combines an arbitrary water treatment with a separation of the stable aggregates into size by the use of a nest of sieves. Aggregate size distribution is often measured to gain information on the size of the aggregates as they exist in the mass of soil.

Apparatus

- Nest of sieves of sizes 8.0,4.0,2.0,1.0,0.5,0.2 and 0.10 mm.
- A Yoder type sieving machine which raises and lowers the nest of sieves through water approximately 30 times per minutes.
- Mechanical stirrer.
- Balance.

Procedure

- Take the sample when the soil is moist and friable. Sieve it through 8 mm sieve and collect the aggregate on 4mm sieve.
- Air dry the sample retained on 4mm sieve at room temperature.
- Fill the Yoder container in which sieving is to take places with salt free water to a level somewhat below that of the screen in the top sieve.
- Immediately prior to sieving, raise the water level rapidly to a point where it barely covers the sample when the sieves are in their highest position. Allow <3 seconds to elapse between the time the water first touches the sample and the time it completely covers the sample.
- After 10 minutes of wetting, sieve the sample for 10 minutes on a nest of sieves of sizes 2, 1, 0.5, 0.2, 0.1mm.
- Remove the sieves from the water and determine the over dry weight of the material on each sieve.
- Determine the amount of sand in each sieve by dispersing agent and washing the material through the sieve, which will retain the sand larger than the sieve holes.
- Oven dry the sand weight it.
- Determine the weight of aggregates in each sieve by subtracting the weight of the sand from the weight of the oven dry material retained after the first sieving.
- Calculate the quantity of the material smaller than 0.2mm by subtracting the sum of the oven dry weight of material retained on each from the oven dry weight of the original sample.

Precautions

- Sample should be dried at room temperature as drying the soil at high temperatures sometimes causes irreversible or slowly reversible dehydration of the bonding materials and clay particles.
 - Compression of samples when they are taken from the soil causes some variation in aggregate stability especially when soil is moist. Proper care should be thus taken to avoid such compression.
 - Analysis should be dried within 24 h by spreading these out in a layer <3mm thick. Soil dried slowly develop are strength.
 - The temperature of water used for wet sieving should be within 22-25⁰C range because at higher temperature the aggregate stability decreases.
 - Salt content of water should be low enough having electrical conductivity of <0.01 dSm⁻¹.
- (c) **Aggregate stability by water drop method** : Falling rain drops have kinetic energy which destroys the aggregates by exerting a significant dispersive action on the aggregates. The dispersed particles are then carried into the soil pores causing increased compaction and decreased porosity. Intense rains destroy the granulation and open structure of the top soil to from a dense impervious surface called crust. Insufficient emphasis has been placed upon the deterioration action of falling rain drops. Their immediate influence is confined to a shallow layer on the surface, but the structure of the layer may be broken down to limit the air-moisture relations of the entire profile.

Apparatus

- Rain drop simulator
- Vacuum desiccators
- Weighing balance

Procedure

- Air dry the soil sample and break it manually into peds.
- Equilibrate the peds to a constant weight at a relative humidity of 98% in 1N sulphuric acid in a vacuum desiccators at 25°C.
- Take a ped weighing between 0.75 to 3.75 g and weight it exactly.
- Place this ped on a 2 mm sieve kept on the top of a beaker.
- Allow the distilled water drops of 4.7 mm diameter to fall from the rainfall simulator kept at a height of 30cm above the soil aggregate, till it is completely destroyed.
- Count the number of drops used in breaking the soil ped completely and designate it as N.

Find the 1/N value and classify the stability as follows :

(1/N value)	Stability
0.0-0.1	Very stable
0.01-0.04	Stable
0.04-0.06	Slightly unstable
0.06-0.08	Moderately unstable
>0.08	Highly unstable

Characterization of soil structure

It is generally considered that a unit weight of large aggregates is more indicative of good structure for most agricultural purpose than is an equal weight of small aggregates and this concept is incorporated in certain mathematical techniques for expressing aggregation data in the form of a single parameter.

- (a) **Mean weight diameter (MWD)** : It is equal to the sum of products of the mean diameter d_i of each size fraction and the proportion of the total sample weight W_i occurring in the corresponding size fraction, where the summation is carried out over all 'n' size fractions, including the one that passes through the finest sieve.

$$MWD = \frac{\sum_{i=1}^n (d_i \times W_i)}{\sum_{i=1}^n W_i}$$

It has been shown that the change in MWD when dry soil sample are wetted is correlated with crop yields under some conditions. An average correlation coefficient of about 0.9 has been obtained.

- (h) **Geometric mean diameter (GMD)** : It is calculated approximately by the equation.

$$GMD = \exp \left(\frac{\sum_{i=1}^n W_i \log d_i}{\sum_{i=1}^n W_i} \right)$$

Where W_i is the weight of aggregates in a size class with an average diameter d_i and W is total weight of the sample. It is based on the finding that aggregate size distribution in most soil is approximately log normal rather than normal. This log normal distribution provides the opportunity to describe the actual aggregate size distribution of most soils with two parameters, the geometric mean diameter and the log standard deviation. If data are summarized in the form of these two parameters, the MWD and other aggregate size indices may be calculated from of these parameters. The main disadvantage of expressing data in terms of the GMW and log standard deviation is the extensive work involved in obtaining them. The log standard deviation must be obtained by either graphical or differential interpolation from the data.

(b) Stability Coefficient (SC) :

$$SC = \frac{D - S}{D}$$

Where D is percentage of primary particles <0.25 mm in diameter obtained from particle size distribution analysis. S is percentage of soil particles <0.25mm in diameter determined by aggregate analysis (wet sieving)

(c) Aggregate index : It is defined as the area between the two curves obtained by plotting the percentage of soil particles below a size range against that size class and other by plotting the percentage of primary particles below the size range against the respective size on the same graph.

Its Value should always be <1.0

Observations and Calculations sheet

Aggregate size distribution

S. No.	Mean size of sieve(mm)	Size of sieve(mm)	Aggregate size (mm)	Sand size (mm)	Aggregate size - Sand size (mm)	WSA (%)
1.						
2.						
3.						
4.						
5.						
6.						

$$MWD = \{ \text{-----} X \text{---} \} + \{ \text{-----} X \text{---} \} + \{ \text{-----} X \text{---} \} + \{ \text{-----} X \text{---} \} + \{ \text{-----} X \text{---} \}$$

MWD = -----

GMD =

SC =

Exercise No: 6

Title : Determination of soil strength by cone penetrometer.

Objective : To determine hardness and resistance of soil to mechanical manipulation

Any device forced to measure its resistance to vertical penetration may be called a "Penetrometer". It is a device used in evaluating the physical condition of soil. Penetrometer consists of a probe which penetrates the soil with a means of measuring the force required to thrust it through a soil mass. Usually the force required to thrust the penetrometer probe to a certain depth is recorded in the measuring device attached to the penetrometer. This reflects hardness of the surface crust, soil moisture condition and compactness of the soil. If properly correlated under similar and controlled conditions, the penetrometer measurements will reflect any variation in these characteristics of the soil. The greatest use of penetrometer lies in tillage investigations involving crusts, ploughpans and other compaction phenomena. Its use is limited, however, since the force required for penetration depends not only on the amount of compaction, but also upon the soil consistency. In practice the penetrometer must be looked upon as a tool for measuring qualitatively the soil physical conditions.

Procedure

Check the black pointer at the manometer is at zero. Because of internal friction the pointer may sometimes not return to zero. By turning the plunger and pulling it out a little the pointer must return to zero. The red maximum pointer can be turned to zero with the help of adjusting screw. If both the pointers are at zero select a cone appropriate for the expected density of the soil that is to be sounded. Measurements in the range 0 to 0.1 kN must be avoided because the working pressure in apparatus will be too low. Fit the cone to the rod and connect the rod via an extension rod and the plunger to the measuring instrument. Applying uniform pressure to the hand grips push the cone to the ground at a constant rate. During the measurement the resistance can be read under the black pointer at the manometer. The maximum resistance while measuring that section, is indicated by the red maximum pointer. The resistance is read in kN (kilo Newton) and noted for appropriate depth. The base area of the cone should also be noted because the cone resistance is expressed in kN/cm^2 .

$$\text{Cone resistance} = \frac{\text{Manometer reading}}{\text{Base area of cone}}$$

Observation Sheet

1. Manometer reading=-----
2. Base area of cone=-----

Calculation

Cone resistance

Exercise No: 7

Title : Determination of soil moisture by Gravimetric and Volumetric method.

Objective : To determine soil moisture content.

A knowledge of soil water content and other soil properties allows one to know :

- (a) To infer the influence of water on plant growth
- (b) To compute the amount of irrigation or rainfall need or the depth of penetration of given amount of water.
- (c) To compute the evapotranspiration.

A. Gravimetric method

The gravimetric method is most commonly used and it is a most basic method.

Principle

Soil water content is determined by drying a known mass of moist soil sample in an oven at 105°C. This method involves three independent measurements, viz. the mass of the wet soil, the mass of dry soil and mass of the container. The dry mass of the soil is taken after drying the soil in an oven at a temperature of 105°C for 24 hours or till the mass becomes constant. At this temperature, all water except that chemically bound is driven out of soil. The oxidation of soil organic matter is low at this temperature and thus loss is negligible.

Apparatus

1. Sampling auger/soil tube
2. Moisture cans (Numbered)
3. Oven for drying the samples
4. Balance
5. Desiccators

Procedure

Take the soil sample with the help of an auger or sampling tube. Place the moist soil samples in moisture boxes. Weigh the samples after taking them to laboratory. Place the boxes with lids off in an oven at a temperature of 105°C to dry the soil to a constant weight. Remove samples from the oven. Replace the lids and place the boxes in the desiccators. Upon cooling, determine the mass of dry soil and moisture boxes.

Observations and calculations

1. Mass of moisture can + moist soil, g = X_1
2. Mass of moisture box + dry soil, g = X_2
3. Mass of empty moisture box, g = X_3

$$\text{Soil moisture content on weight basis } (\Theta_w \%) = \frac{\text{Weight of wet soil} - \text{weight of dry soil}}{\text{Weight of dry soil}} \times 100$$

$$\text{Soil moisture content on weight basis } (\Theta_w \%) = \frac{X_1 - X_2}{X_2 - X_3} \times 100$$

$$\text{Soil moisture content on volume basis } (\Theta_v \%) = \text{Percentage of moisture on wt. basis} \times \text{bulk density}$$

$$\Theta_v = \Theta_w \times \text{BD}$$

Precautions

1. Weight moist and dry soil samples accurately and carefully.
2. Place the moisture boxes with their lids removed in the oven for soil drying.

Observations sheet and calculations

1. Mass of moisture can + moist soil, g = -----
2. Mass of moisture box + dry soil, g = -----
3. Mass of empty moisture box, g = -----

$$\text{Soil moisture content on weight basis } (\Theta_w \%) = \dots \times 100$$

$$\text{Soil moisture content on volume basis } (\Theta_v \%) = \dots \times \dots$$

B. Volumetric method for determination of Volumetric moisture content

It is based on volume weight relationship and need not require drying of sample

Principle

When known amount moist soil is added and shaken with a liquid contained in a vessel the soil air is escapes and the increase (V_i) in the original volume of the liquid is equal to the volume of soil water (V_w) plus the volume of soil solids (V_s) i. e.

$$V_i = V_s + V_w$$

But $V_s = W_s / D_s$

Where,

W_s is the weight of dry soil
 D_s is the particle density
 and $V_w = W_w / D_w$

Therefore, representing weight of moist soil by W_{sm} we get,

$$V_i = (W_{sm} - W_w) / D_w + W_w / D_w$$

on solving above equation we get,

$$W_w = D_w (D_s V_i - W_{sm}) / (D_s - D_w)$$

It is thus possible to compute the quantity of moisture present in a given moist soil sample if only two quantities, viz. V_i and D_s are known. D_w is taken one $M\text{ gm}^{-3}$.

Apparatus

1. Moisture gauge with matching 50 ml pipette
2. Balance
3. A piece of glazed paper

Procedure

Weight 20 g of the moist soil sample on a piece of glazed paper and transfer it to the basal flask. Add 50 ml of water to the flask with pipette while washing down any soil particles sticking to the neck of the flask with a jet of water from the pipette. Fit the tubular stopper into the neck of the flask with a gentle rotation. Gently shake the contents of the flask and make sure that the air is expelled from the soil. Add another 50 ml of water with the pipette through the stopper and allow the contents to stand for two minutes. Note down the reading on tubular stopper as final reading. The difference of the final and initial reading is increase in volume.

Observation and calculation

Mass of the moist soil = 20g

Initial reading of the gauge with 100 ml water only = V_w

Final reading of the gauge with 100 ml water + moist soil = V_f

Increase in volume of water, V_i in Cm^3 = $V_f - V_w$

Particle density of soil = $D_s\text{ Mg m}^{-3}$

Thus,

$$W_w = D_w [D_s (V_f - V_w) - V_{sm}] / (D_s - D_w)$$

$$W_w = 1 (D_s V_i - 20) / (D_s - 1)$$

$$W_w = D_s V_i - 20 / D_s$$

Observations and calculations sheet

Mass of the moist soil = -----

Initial reading of the gauge with 100 ml water only = -----

Final reading of the gauge with 100 ml water + moist soil = -----

Increase in volume of water, V_i in Cm^3 = -----

Particle density of soil = -----

Thus,

$$W_w = \text{-----}$$

$$W_w = \text{-----}$$

$$W_w = \text{-----}$$

Exercise No.: 8

Title : Determination of soil moisture constants - field capacity, water holding capacity and wilting point

Objective : To quantify soil moisture parameters.

General Principles

The relation between the soil water content and the soil water suction is a fundamental part of the characterization of the hydraulic properties of soil. The relationship is defined as moisture characteristics. The function relates a capacity factor, the water content, to an intensity factor, the energy state of the soil water. The term soil water is commonly used for the solution or liquid phase of the soil. One of the various terms synonymous to suction, such as capillary potential, capillary pressure head, matric pressure head, tension, matric potential, or pressure potential may be used instead of suction. Matric pressure head values for unsaturated soil are negative. Suction and tension are the negative of the pressure head, and hence are positive.

The potential of the soil water may be expressed in units of energy per unit mass, energy per unit volume, or energy per unit weight of the soil water. Energy per unit volume is essentially equivalent to force per unit area or pressure. Energy per unit weight has dimensions of length.

The water content, solution phase content or wetness of the soil may be expressed on a weight, volume, or degree of saturation basis. For analysis of water flow in soil profiles, the volume basis is the most useful.

Mechanism of water retention

In order to clearly comprehend the mechanism of water retention by soil one should understand the phenomenon of capillarity. When a capillary is placed on the surface of a free water body, the water is attracted by the capillary to certain height depending on the radius of the capillary. This rise is because of (a) the attraction of solid surface (glass) for water molecules (adhesion) and (b) the attraction of water molecules for each other (cohesion). By adhesion the water molecules are held rigidly at the solid surface. These water molecules in turn, hold water molecules further away from the solid surface by cohesive force. The height of rise of water column in a capillary of radius r is given by :

$$h = 2 \sigma \cos \theta / pgr$$

where, h = height of rise in a capillary tube (cm)

r = radius of capillary tube (cm)

p = density of water (g cm^{-3})

σ = surface tension of water (dyne cm^{-1})

θ = contact angle between glass surface and liquid water and is 0° .

The rise of water, h , is inversely proportional to the radius of capillary tube.

Soil, in essence, contains numerous capillaries of various shape and size. A soil column when brought in contact with a free water surface, therefore, attracts water and the water rise in to the column through the interconnected pores (visualized as capillaries) till the capillary force is counter balanced by the weight of rising water column or gravitational force. Thus a soil with greater proportion of smaller pores will retain more water at a given externally applied. Suction. Numerous forces act upon water in a soil. They include earth's gravitational fore matric force due to surface tension, osmotic force due to presence of solutes and electric double layer at micellar surface, force due to weight of water and also due to weight of soil particles where soil matrix lacks rigidity and adsorptive forces. All these forces influence energy status of soil water, retention and movement in soil. Matric forces however, are the most important form water retention and release point of view in normal soils.

Hysteresis in the water function

The retention function is hysteretic, i.e. the water content at a given pressure head for a wetting soil is less than that for a draining soil). The drainage curve that starts at complete saturation of the porous medium is the initial drainage curve (IDC). In many porous media, as water is removed through a porous plate, the matric pressure head decrease (i.e. becomes more negative), and the water content approaches a limit called the residual water content. The main wetting curve (MWC) is obtained by wetting the soil from low water content. The initial water content of the sample that is used to determine the MWC will effect the relationship found between θ and μm . In many instances, the MWC is determined starting at the residual water content, or. As the soil is wetted along the MWC and the pressure head approaches zero, the water content approaches a value, θ_0 , that is less than the total porosity, θ_s , due to the presence of entrapped air. Usually θ_0 is about 0.8 θ_s to 0.9 θ_s . The water content, θ_0 is called the natural saturation or the satiated water content, The drainage curve obtained beginning at θ_0 is called the main drainage curve (MDC) . The MDC merges asymptotically with the IDC as the pressure head decreases.

In some cases, only the drainage curve (either the IDC or the MDC) is required for the analysis of the water flow. In other cases only a wetting curve (i.e. the MWC) is required. Most "natural" fine-dependent boundary conditions on water flow in soils will insure that the flow is hysteretic. Recent numerical methods of solution of the flow equation for soil water are beginning to incorporate hysteresis information.

Methods for laboratory determination of water retention

Apparatus

The nature of the apparatus required will depend upon the range of matric pressure had in which the retention measurement are to be made. In general, the lower the pressure head, the higher the bubbling pressure requirement of the porous plate, and the greater the strength requirement of the pressure chamber.

Low range system

This system is especially suited to measurements in the matric pressure head range 0 approximately-200 cm of water. The major components of the system are (i) the sample chamber, (ii) the cell pressure control system and (iii) the suction or in the combined pressure suction mode.

Mid-range system

The system is suited to the measurements in the matric pressure head range-200 to-1000 cm of water. The chamber is standard pressure cooker, which is available at outlets for kitchen utensils, The cell gas phase pressure regulation system utilizes a pressure regulator, which is capable of regulation in the output pressure range 0 to 15 kPa. The cell pressure regulator requires a drive pressure at about 35 to 5Pa.

which can be obtained from another pressure regulator, operating from a compressed air source at a higher pressure.

The out flow system consists of the connection from the outlet from the plate, and the reference bottle. The porous plate is ceramic with a bubbling pressure of at least 1000 cm of water (1 bar). The range of measurement of the mid range system may be extended to matric pressure heads of -30 m of water (-3 bars) by using a ceramic plate with a bubbling pressure of 30 cm of water (300 kPa), a suitable higher output range pressure regulation, and a 500 kPa (5 bar) chamber.

High range system

The measurement range of this system is from 10 to 1500 kPa (10-15 bar) suction. The essential components of this system are (i) a pressure chamber (ii) a ceramic plate with a bubbling pressure of at least 1500kPa and (iii) a gas pressure supply and regulation system capable of pressure regulation to 1500kPa. If frequent measurements are to be made, it is better to use a special high pressure, low capacity compressor, which can supply compressed air at approximately 2MPa (300 psi) The pressure regulator must be capable of control in the range from 0.1-1.5 MPa.

Samples

The soil samples for determining the retention curve may be either repacked samples or samples of natural structure. Since the structure of the sample affects the water retention, especially in the low suction large, it is generally best to use sample of natural structure. More samples may be used to obtain soil samples of relatively undisturbed structure. Thin walled metal cylinders with sharpened edge may be pressed to obtain undisturbed soil samples. McIntyre (1974) discussed methods of procuring undisturbed samples for physical measurements. Water retention measurements on sub-soils especially those in which there is a significant degree of fragmented and repacked samples for such materials can give results that are not representative of the soil *in situ*, even if the fragmented soil is repacked to the same bulk density as the soil *in situ*, Crushing, drying and sieving of the soil destroys or severely modifies any structural units the soil may have had *in situ*, and repacking, even to the original bulk density, cannot reproduce the field structure.

The practice of placing fragmented samples on the porous plate with no control and knowledge of the bulk density of the packing is not recommended. The volumetric water content obtained by such a procedure is greatly in error at low suctions. However at higher suctions, where the retention of water is primarily by adsorption forces and is proportional to the specific surface of the soil, this practice may yield more valid results.

Sample size

The sample dimensions will generally be in the range of 5 to 15 cm diameter and 1 to 5 cm high. The time for reaching equilibrium is proportional to the square of the height of the sample, and the height should be small to reduce the equilibration time. On the other hand core sample with lengths of 1 cm or less are difficult to handle. A practical length is 2 to 3 cm. Generally, samples with a diameter of 5 to 8 cm and a height of 2 to 3 cm are used. However, the consideration with respect to sample size in relation to the structural elements of the soil should be kept in mind when making and interpreting measurement.

Wetting of samples

The method of wetting depends on whether points on the IDC or the MDC are to be determined. If points on the MDC are to be determined, place the replicate samples for a given suction on a prewetted plate or membrane that has an appropriate bubbling pressure. Wet the samples on the plate by immersing the plate and the samples in water to a level just below the top of the samples. It is best to do this in the

chamber in which the samples are to be drained to equilibrium. If points on the IDC are desired, a vacuum wetting procedure to attain complete saturation must be used.

Equilibration

Remove any excess water from the porous plate with a syringe or siphon, connect the out flow tube to the pass through connector in the wall of the chamber, and connect it in turn to the out flow reservoir close the chamber. Apply the desired all gas pressure and/or the desired suction to the outflow system. Allow the samples to come to equilibrium. For core samples that the about 2 to 3 cm high, an equilibration time of 2 to 3 days has been found sufficient.

Sample removal

Weight and number the set of moisture cans, each of sufficient capacity to hold the soil from a core. When the samples are judged to have come to equilibrium, place a clamp on the outflow tube to prevent backflow of water and release the cell air pressure, quickly transfer the soil from each sample ring to a moisture can and replace the lid of the can. Determine the wet weight of the soil plus can W_w . Dry the samples at 105°C and determine the oven dry weight of the soil plus can, W_d .

Calculations

Calculate the volumetric water content θ_v and bulk density P_b of each sample from.

$$\theta_v = (W_w - W_d) / d V_s \times P_b$$

$$P_b = (W_d - W_c) / V_s$$

Where, d is the density of water
 V_s is the dry wt. of soil
 W_c is the weight of empty can.

Observations and Calculations sheet

Gravimetric moisture content θ_w at different pressure head

S. No.	Depth	0 bar	0.33 bar	5.0 bar	15 bar

Volumetric water content $\theta_v = \theta_w \times P_b$

$$\theta_v = \text{-----} \times \text{-----}$$

1. The wet weight of the soil plus can W_w (g) = -----

2. Oven dry weight of the soil plus can, W_d (g) = -----

3. The weight of empty can W_c (g) = _____

4. The dry weight of soil V_s (g) = _____

5. The density of water d (g cm^3) = _____

Calculate the volumetric water content θ_v and bulk density P_b of each sample from

$$\theta_v = (\quad - \quad) /$$

$$P_b = (\quad - \quad) /$$

Exercise No.: 9

Title : Determination of infiltration rate by Double Ring Infiltrometer

Objective : To determine water entry rate in to the soil.

Principle

The rate at which water penetrates into a soil , depends upon the distribution and continuity of both capillary and non capillary pores in a particular soil in addition to its dependence on the moisture content and condition of the surface soil.

Equipments

- (i) Two rimless mild steel metal rings, 20cm in length with one edge sharp and 30 cm in diameter and the other is 50 cm in diameter.
- (ii) Stop watch
- (iii) Hammer (10Kg)
- (iv) Twenty litter plastic bottles
- (v) Driving plate
- (vi) Steel plate $\frac{1}{2}$ inch thick, 60 cm in diameter with lags to keep the plate on the ring
- (vii) Heavy paper or cloth to prevent puddling

Procedure

Select a leveled and clean soil surface area and drive the 30 cm and 50 cm ring on the ground by hammering on the driving plate placed on the rings until 10 cm of each ring goes into the soil. Place the cloth or heavy paper on the soil surface of the inner ring. Fill the outer ring with water and then add water in the inner ring. Before filling the water in the inner ring plastic scale must be inserted on the soil surface. Record the fall of water level on the scale and continue recording after 1, 3, 5, 10, 20, 45, 60, 90, and 120 minutes and then after each 30 minutes until the amount of water going into the soil per unit time becomes essentially constant. Refill the rings as soon as the water level drops to the middle.

Observation and calculation

S. No.	Time	I	II	III	Mean	Infiltration rate (mm/hr)	Cumulative infiltratic rate (mm)
1.	5						
2.	10						
3.	15						
4.	30						
5.	60						
6.	90						

Exercise No.: 10

Title : Determination of soil texture by International Pipette Method
Objective : To determine relative proportion of sand, silt and clay and classify texture of soil.

Several soil physical properties depend upon particle size distribution of soils. In fact, particle size distribution is the basic determinant of almost all the physical, chemical and biological phenomena. Particle size distribution is a basic property of soil and determines its texture. Soil texture is defined as the relative proportion of various sized soil particles (sand, silt and clay) in a soil sample.

The classifying and naming of the different soil fractions is arbitrary and numbers of systems are used. It has been possible, however, in all these systems to classify the soil particles by separating them into groups based on diameter size. Each size group is known as a “soil separate”. The systems adopted by British, American and International Soc. Soil Sci. (1929) are shown in the following table:

Table: Size limits from three systems of analysis

British system		U.S.D.A. system		International system	
Separate	Diameter (mm)	Separate	Diameter (mm)	Separate	Diameter (mm)
Fine gravel	2.0-1.0	Fine gravel or very coarse sand	2.0-1.0	Coarse sand	2.0-0.2
Coarse sand	1.0-0.2	Coarse sand	1.0-0.5	Fine sand	0.2-0.02
Fine sand	0.2-0.04	Medium sand	0.5-0.25	Silt	0.02-0.002
Silt	0.04-0.01	Fine sand	0.25-0.10	Clay	<0.002
Fine silt	0.01-0.002	Very fine sand	0.10-0.05		
Clay	<0.002	Silt	0.05-0.002		
		Clay	<0.002		

The determination of relative proportion of the size groups of ultimate soil particles (sand, silt and clay) is called Mechanical Analysis. The older and somewhat ambiguous term “mechanical analysis has now been replaced by particle size analysis.

In this method two important steps are necessary.

1. Dispersion: Separation of all particles from each other ie. complete dispersion into ultimate particles.
2. Fractionation: Measuring the amounts of each size group in the sample.

A. Dispersion

Under natural soil conditions soil particles generally exist as aggregates or floccules in which soil particles are grouped and cemented together by various cementing materials e. g. organic matter, calcium carbonate, colloidal oxides of Fe and Al, low electrokinetic potential and surface tension etc. present in soils. To determine particle size distribution, it is absolutely necessary to disperse the soil particles completely. The dispersion of soils is accomplished by inactivating or removing cementing and flocculating agents as described below:

Removal of cementing agents:

Organic matter: It is removed by treating the soil with hydrogen peroxide (30%) which oxidizes the organic matter to carbon dioxide and water.

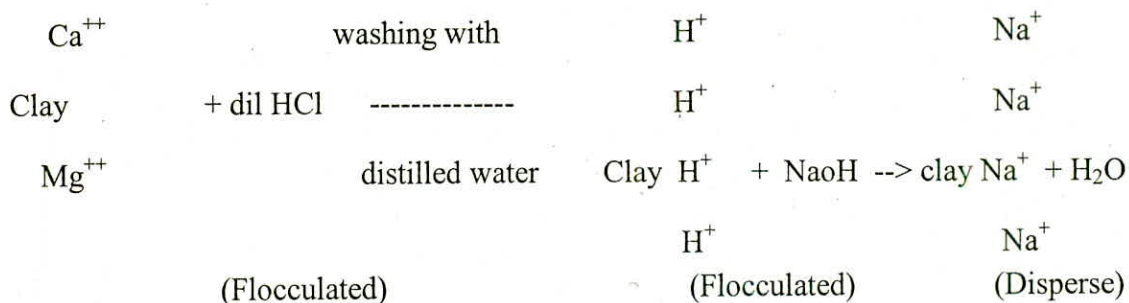
Oxides of iron and aluminum: The cementing agent is present in lateritic soils. The removal of the cementing influences of the oxides of Fe and Al is successfully done by sodium dithionite citrate procedure. This removes free Fe and Al oxides with a minimum of destruction of the clay minerals. Sodium citrate serves as the chelating agent for ferric forms of iron.

Clay to clay cementation: Clay particles are strongly bonded with one another upon dehydration. Thus these bonds can be broken by rehydration. The physical methods of rehydration are shaking and stirring with an electric shaker.

Calcium carbonate: Cementation due to calcium carbonate is removed by treating the soil sample with dilute HCl.

Removal of flocculating agents (soluble salts and ions): To deflocculate the soil particles the soil is repeatedly washed with distilled water till it is free from soluble salts:

The HCl treatment saturates the exchange complex with hydrogen ions (H⁺) which also keeps the soil flocculated. They disperse the soil the H⁺ on the soil exchange complex is replaced by a peptizing action like Na⁺ by treating the sample with sodium hydroxide (NaOH). The above steps are summarized in the following reactions.



When sodium ion replace other cations on the clay a strong electrical repulsive force develops between soil particles which keeps the clay particles dispersed. Other dispersing reagents that can be used are sodium oxalate, sodium phosphate, sodium metaphosphate, sodium pyro-phosphate and sodium hexameta phosphohate or their mixture in dilute concentration. Finally, particles are dispersed by vigorous stirring of the soil water suspension with the electric shaker.

B. Fractionation

Once the soil sample is dispersed into its ultimate particles of each particle size fraction is measured coarse sand particles are separated by sieving the dispersed soil suspension. Fine sand particle are separated by decantation. Fraction of the finer sized particles (clay silt) is based on their settling velocities in a liquid which is governed by stock's law of sedimentation given below:

A rigid particle falling freely through a liquid of a lower density will attain a constant velocity (called terminal velocity) when the frictional force of the liquid (F upwards) opposing downward movement is equal and opposite to the force of gravity (F downward) acting on the particle.

The drag force (downward force) acting on the spherical particle moving in viscous medium is given by:

$$F_D = 3 \pi d \eta v$$

Where,

F_D = drag force acting on particle (dynes)

d = diameter of the particle, cm

η = viscosity, $\text{g cm}^{-1}\text{sec}^{-1}$

v = velocity of particle relative to fluid, cm sec^{-1}

The gravity force F_g , acting downward on the particle of diameter d is

$$F_g - Mg = \rho_p \pi d^3 / 6g$$

Where, m =mass of particle, g = acceleration due to gravity and ρ_p = density of particle.

The buoyant force (upward force) due to displacement of liquid by particle also acts upward resisting the motion of the particle.

$$F_b = W = \rho_l \pi d^3 / 6g$$

W = weight of displaced liquid

ρ_l = density of liquid, Mg m^{-3}

The resultant downward force on the particle is

$$F_g - F_b = \rho_p \pi d^3/6g - \rho_l \pi d^3/6g = \pi d^3/6g (\rho_p - \rho_l)$$

At equilibrium

$$F_D = F_g - F_b$$

$$3 \pi d \eta v = \pi d^3/6g (\rho_p - \rho_l)$$

$$v = d^2 (\rho_p - \rho_l) g / 18 \eta$$

$$v = (2r)^2 (\rho_p - \rho_l) g / 18 \eta$$

$$v = 2/9 r^2 (\rho_p - \rho_l) g / \eta = h / t \text{ ----- (1)}$$

where, h is depth of settling and t is time

Equation (1) is called Stock's law.

Thus in a time t all particles having settling velocities greater than h/t would have traveled beyond depth h and the particles having settling velocities less than h/t would be as yet present above this depth. The taking of a small volume element by a pipette at a depth "h" at time "t" furnishes a sample from which all particles coarse than a particular size have been eliminated and in which all particles finer than that size are present in the same amount as initially.

There are certain assumptions on which the Stock's law is based such as :

1. Soil particle must be spherical and rigid.
2. Particles must be large in comparison with the molecules of the liquid (>0.0002 μm).
3. Fall must be unhindered so that they do not cause turbulence during fall.
4. Particle must be of uniform density.
5. The temperature fluctuations do not set up convection currents in the suspension.

In spite of these assumptions, the law is fairly accurate in its application in particle size analysis of soils. For different temperatures the time of settling or sedimentation of different particles calculated using equation (1) and assuming particle density of 2.6 Mgm⁻³ are given in the following table :

Table : Sedimentation times for silt and clay particles in water to reach at 10 cm depth

Temperature °C	Settling time			
	Clay (<0.002 mm)		Silt + clay (<0.02 mm)	
	Hours.	Minutes	Minutes	Seconds
10	10	25	6	20
11	10	10	6	10
12	9	60	6	0
13	9	35	5	50
14	9	20	5	40
15	9	5	5	30
16	8	50	5	20
17	8	35	5	10
18	8	25	5	0
19	8	10	4	50
20	8	0	4	45
21	7	50	4	40
22	7	35	4	35
23	7	25	4	30
24	7	15	4	20
25	7	0	4	15
26	6	55	4	10
27	6	45	4	5
28	6	40	4	0
29	6	30	3	55
30	6	20	3	50
31	6	15	3	45
32	6	0	3	40
33	5	55	3	35

Apparatus

1. A 500 ml beaker
2. A hot plate
3. A glass rod
4. A wash bottle
5. Whatman No. 50 filter paper
6. An electric stirrer
7. A to mesh sieve
8. A sedimentation cylinder one litre capacity
9. A glass funnel
10. A thermometer
11. A plunger – an iron rod attached to a circular metallic disc of 5-6 cm in diameter and 0-15 cm. in thickness
12. A sampling pipette

13. Beaker (100 ml capacity)
14. A balance

Reagents

1. Hydrogen peroxides, H_2O_2 (30 %)
2. HCl (2N)
3. $AgNO_3$ (N/10)
4. NaOH (N/10)
5. Phenolphthalein indicator

Procedure

Take 20g oven dry soil (ground and sieved through a 2 mm sieve) in a 500 ml beaker. Put 20 ml of H_2O_2 and swirl the contents well. Allow the reaction to take place for 5-10 minutes. Place the beaker on a hot plate. Continue heating, stirring the contents all the time with a glass rod to minimize frothing, till the completion of the reaction. If frothing and reaction persists for a long period another lot of H_2O_2 should be added. Cool the contents and remove the soil particles from the inner sides of the beaker by rubbing with the glass rod and using a jet of distilled water. Add 25 ml of 2 N HCl. If more than 2% $CaCO_3$ is present, more HCl should be added at the rate of 7.5 ml for each per cent of $CaCO_3$. This step can be omitted if soil is free from $CaCO_3$. Let the reaction proceed for about an hour with intermittent shaking. Filter the contents through Whatman No. 50 filter paper. Discard the filtrate. Wash the soil retained on the filter paper with distilled water till the filtrate is free from chlorides (This can be tested by adding a small amount of $AgNO_3$ to the filtrate. If a white precipitate appears, chloride is still present and sample needs more washing). Transfer the soil sample from filter paper to a 500 ml beaker with a jet of distilled water. Make the volume to about 300 ml with distilled water. Add a few drops (5-6) of phenolphthalein indicator. Add N/10 NaOH till the whole suspension shows pink colour indicating its alkaline reaction. Avoid over alkalization. Shake the contents of the beaker with an electric shaker for 10 minutes. Transfer the contents on to a 70 mesh sieve placed in the funnel kept over the top of one liter cylinder. Wash the soil particles on the sieve with a jet of distilled water till only the coarse particles are left on the sieve. Transfer these particles to a weighed 100 ml beaker oven dry it at $105^\circ C$ to a constant weight. Record this weight as the weight of coarse sand. Make the volume of the suspension in the cylinder to one liter by adding more distilled water. Note down the temperature of the suspension. Against this temperature, read the required time for sampling silt + clay and clay alone from above table. Stir the contents with plunger by moving it up and down gently for about 20-25 times in one minute so that no soil remains settled at the bottom of the cylinder. Remove the plunger carefully and note down time at which the plunger is taken out. This is the beginning time for settling of finer particles. Insert the sampling pipette gently into the suspension and dip it to 10 cm depth from the surface of suspension about 20 seconds before the expiry of the sampling the time (time is decided by knowing the temperature of the suspension and using the above table). Pipette out 25 ml of the suspension at each requisite time (for silt + clay and clay) at a moderate speed. Take out the pipette and transfer the sample collected to a weighed 100 ml beaker. Oven dries the sample at $105^\circ C$ to a constant weight. Weight the beakers with silt + clay and clay upto milligrams. Note down three weights. Calculate the weight of silt by subtracting weight of clay from that of silt + clay. Decant the remaining suspension by washing repeatedly with distilled water. Remove any particles left in the cylinder

with a jet of distilled water into a weighed 100 ml beaker. Dry the contents of beaker in an oven and weigh them. This is the fine sand in the soil sample.

Observation and Calculation

Mass of soil taken (g)	=	20
Mass of beaker (g)	=	M1
Mass of beaker + coarse sand,(g)	=	M2
Per cent coarse sand, P_{cs}	=	$(M2-M1)/20 \times 100$
Temperature of the suspension, ° C	=	T
Time at which plunger is taken out of the cylinder	=	t_o per min.
Time at which sample for silt + clay is to be collected	=	$t_o + t_{sic}$
Time at which sample for clay is to be collected	=	$t_o + t_c$ hr min.
At time $t_o + t_{sic}$		
Mass of beaker, (g)	=	M3
Mass of silt + clay, (g)	=	M4 - M3
Per cent silt + clay, P_{sic}	=	$[(M4-M3)/20] \times (1000/25) \times 100$
At time $t_o + t_c$		
Mass of beaker, (g)	=	M5
Mass of beaker + clay, (g)	=	M6
Mass of clay, (g)	=	M6 - M5
Per cent clay, P_c	=	$[(M6-M5)/20] \times (1000/25) \times 100$
Per cent fine sand	=	$100 - (P_{sic} + P_{cs})$

Observation and Calculation sheet

Mass of soil taken (g)	=	-----
Mass of beaker (g)	=	-----
Mass of beaker + coarse sand,(g)	=	-----
Per cent coarse sand, P_{cs}	=	-----
Temperature of the suspension, ° C	=	-----
Time at which plunger is taken out of the cylinder	=	-----

Time at which sample for silt + clay is to be collected = -----

Time at which sample for clay is to be collected = -----

At time $t_0 + t_{sic}$

Mass of beaker, (g) = -----

Mass of silt + clay, (g) = -----

Per cent silt + clay, P_{sic} = -----

At time $t_0 + t_c$

Mass of beaker, (g) = -----

Mass of beaker + clay, (g) = -----

Mass of clay, (g) = -----

Per cent clay, P_c = -----

Per cent fine sand = -----

Exercise No.: 11

Title : Preparation of primary and secondary standard solutions.

Objective : To acquaint with the procedure of preparation of primary and secondary standard solution for their use in analysis process.

Standard solutions: The solutions of accurately known strength (or concentration) are called standard solution. It contains a definite number of gram equivalent or gram mole per litre of the solution.

Strength of a solution: Strength of a solution refers to the weight of a solute dissolved in a unit volume of the solution. It can be expressed in many ways as follows

(A) **Normality (N) and normal solution:** Normality is the number of gram equivalent of substance dissolved in one litre of the solution, thus a normal solution is one which contains 1 gram equivalent (equivalent weight in grams) of the active reagent, dissolved in one litre of the solution.

If the number of gram equivalent is one it is expressed as 1N. If the number is 1/10, 1/100 or 1/1000 then it is designated respectively as 0.1N (deci normal), 0.01N (centi normal) or 0.001 (mill normal) solution.

(1) **Gram equivalent:** The gram equivalent of a substance is the number of grams of this substance chemically equivalent to one gram atom or ion of hydrogen in a given solution.

(i) **Gram equivalent of an acid:** The gram equivalent of acids is the weight of it contains one gram atom (or ion = 1.0078 gram) of a replaceable hydrogen.

Thus, Equivalent weight of an acid = Molecular weight of acid \div Number of replaceable hydrogen atoms in a molecule or

Equivalent weight of an acid = Molecular weight of acid \div Basicity of the acid

(ii) **Gram equivalent of a base:** The gram equivalent of bases is the weights of bases which contains one replaceable hydroxyl group or which react with one hydrogen ion.

Thus, Equivalent weight of base = Molecular weight of the base \div Number of replaceable hydroxyl groups in a molecule, or

Equivalent weight of a base = Molecular weight of the base \div Acidity of the base

(B) **Molarity (M) and molar solution:** Molarity is the number of moles of a compound dissolved in one litre of its solution, i.e., A molar solution is the one which contains one mole or one molecular weight in grams of a substance in each litre of the solution, whether the substance is in the form of molecules, ions or any other species.

(C) **Molality (m) and molal solution:** The molality of a solution is the number of moles of the solute per 1000 gram of the solvent. Thus a molal solution is one which contains a gram molecule of the solute dissolved in 1000 gram of solvent.

(D) **Formality (F) and formal solution:** Formality is the formula weight of a solute dissolved in a litre of solution. Thus, a formal solution is one which contains a formula weight of a solute in a litre of solution. In most cases formula weight and molecular weight are identical but sometimes the true molecular weight of a compound is a multiple of the weight expressed by its formula as as ordinarily written in a chemical reaction.

There are two types of standard solutions, viz., primary standard solution and secondary standard solution.

Primary standard solution: The substances which are available in pure state and are not hygroscopic can be weighed accurately a definite fraction in an analytical balance, dissolved in distilled water and diluted to accurately known volume. These solutions are called primary standard solutions. Examples of substances which can be obtained in a state of high purity and are therefore suitable for preparation of standard solution are

Oxalic acid ($H_2C_2O_4 \cdot 2H_2O$), Sodium carbonate (Na_2CO_3), Sodium borate ($Na_2B_4O_7 \cdot 10H_2O$), Silver nitrate ($AgNO_3$), Potassium hydrogen phthalate (KHP), etc.

Secondary standard solution: The accurate primary standard solutions of those substances which are not practically obtained in pure state and are hygroscopic in nature or easily affected by exposure to air are not being possible. e. g., Sodium hydroxide ($NaOH$), Potassium hydroxide (KOH), Hydrochloric acid (HCl), Sulphuric acid (H_2SO_4).

The approximate solution of desired strength of secondary standard substances are standardized with primary standard solution and diluted to desired normality.

Exercise: Preparation and standardization of 0.1 N HCl using Na_2CO_3

Procedure: Fill the burette with decinormal solution of HCl . Pipette out 10 ml of 0.1 N Na_2CO_3 in a conical flask, add 2 drops of methyl orange indicator and titrate with HCl till the yellow colour changes to red. Repeat the experiment to get at least three concordant readings.

Calculation:

Calculate the normality of unknown HCl solution by the equation $N_1V_1=N_2V_2$

Where, N_1 = Normality of standard Na_2CO_3 =0.1
 V_1 = Volume of standard Na_2CO_3 pipetted (volume of pipette)=10 ml
 N_2 = Normality of HCl (?)
 V_2 =Volume of Na_2CO_3 used in titration (burette reading)

Dilution of HCl to exact decinormal solution: The standardized HCl can further be diluted to desired accurate normality using the above expression $N_1V_1=N_2V_2$ again

Where, N_1 = desired normality =0.1
 V_1 =desired volume= 100 ml (say)
 N_2 = normality of HCl found (on the above procedure)
 V_2 = volume of HCl required (?)

Procedure: The value of V_2 (i. e., volume of HCl required in ml) if diluted to 100 ml final solution in a volumetric flask then we get theoretically 0.1 N HCl .

Observation:

S. No	Volume of 0.1 N Na_2CO_3 taken conical flask (ml)	Volume of HCl used in titration (Burette reading)		Exact volume of HCl used titration (B-A) ml
		Initial (ml) (A)	Final (ml) (B)	
1.				
2.				
3.				

Calculation:

- Normality of HCl =.....N
Using $N_1V_1=N_2V_2$
- Dilution of HCl to exact decinormal solution (volume required)=.....ml
Using $N_1V_1=N_2V_2$

Exercise No: 12

Title : Determination of soil organic Carbon (Walkley and Black's (1934) rapid titration method).

Objective : To determine soil organic carbon and matter content of soil.

Principle: The organic matter (humus) in the soil gets oxidized by chromic acid (potassium dichromate plus conc. sulphuric acid) utilizing the heat of dilution of sulphuric acid. The unreacted dichromate is determined by back titration with ferrous ammonium sulphate (redox titration).

Reagents:

- (i) 1 N potassium dichromate (49.04 g of AR grade $K_2Cr_2O_7$ per liter of solution)
- (ii) 0.5 N (aprox.) ferrous ammonium sulphate (196 g of hydrated crystalline salt per liter containing 20 ml of conc. H_2SO_4)
(This solution is relatively more stable and convenient to work than that of ferrous sulphate).
- (iii) Diphenylamine indicator: Dissolve 0.5 g diphenyl amine in a mixture of 20 ml of water and 100 ml of conc. H_2SO_4
- (iv) Concentrated sulphuric acid (sp.gr.1.84)
- (v) Ortho-phosphoric acid (85%) (chemically pure).

Procedure: The soil is ground and passed through 0.2 mm sieve. Place 1.00 g. soil at the bottom of dry 500 ml conical flask (Corning /Pyrex) and add 10 ml of 1N $K_2Cr_2O_7$ and swirl a little. The flask is kept on asbestos sheet. Then add 20 ml of concentrated H_2SO_4 and swirl again two or three times. The flask is allowed to stand for 30 minutes preferably in darkness. Add 200 ml of distilled water, 10 ml of ortho-phosphoric acid and titrate the contents with ferrous ammonium sulphate solution till the colour changes from blue-violet to green. Simultaneously, a blank is run without soil. If more than 7 ml of the dichromate solution is consumed, the determination must be repeated with a smaller quantity (0.25-0.50 g) of soil.

Calculation

$$\text{Organic carbon (\%)} = \frac{10 (B-T)}{B} \times 0.003 \times \frac{100}{\text{wt. of soil}}$$

Where, B = Volume (in ml) of ferrous ammonium sulphate solution required for blank titration;
and

T = Volume of ferrous ammonium sulphate needed for soil sample

Observation sheet:

Volume of 0.5 N Ferrous ammonium sulphate consumed in blank titration =.....ml

Volume of 0.5 N Ferrous ammonium sulphate consumed in sample titration =.....ml

Weight of soil taken =.....g

Calculations

Organic carbon (%) =

Exercise No. :13

Title : Determination of soil reaction (pH) and Electrical Conductivity (EC)
Objective : To determine soil reaction (acid/alkaline) and salt content.

Reagents

Standard buffer solutions: These may be of pH 4.0, 7.0 or 9.2 in pure water or in other ranges of expected pH value. In case of buffer tablets (available commercially), a single piece is to be dissolved in freshly prepared double distilled water and made up to 100 ml. It is necessary to prepare a fresh buffer after every few days, as the solutions do not keep for long, even when stoppered.

Soil water suspension (1:2.5):

Take 20 g of soil in a 100 ml beaker and add 50 ml of distilled water. Stir the suspension at regular intervals for 30 minutes and record the pH with pH meter. The suspension must be stirred well just before the electrodes are immersed and readings are taken.

Standard potassium chloride solution: Dissolve 0.7456 g of dry reagent grade potassium chloride in freshly prepared double distilled water and make up to one litre. At 25⁰C, it gives an electrical conductivity of 1411.8×10^{-6} (0.0014118) mhos/cm or 1.41 dSm⁻¹. Calibrate the conductivity bridge and determine the cell constant with the help of this solution. Even if the scale is marked directly in dSm⁻¹ (as in solu bridge), it is necessary to check and calibrate the instrument with the KCl solution. Calibration of the instrument should be done once in a week.

Procedure

Shake 20 g of soil intermittently with 50 ml of distilled water in a 150 ml conical flask for one hour and allow it to stand. Alternatively, the clear extract after pH determination, can be used for electrical conductivity measurement. Determine the conductivity of the supernatant liquid with the help of the salt (conductivity) bridge. The measurement of EC (expressed in dSm⁻¹) is to be adjusted for a known temperature (usually 25⁰C) of the solution by setting the knob provided for this purpose.

Observation Sheet

1. pH of given soil sample (in 1:2.5 soil water suspension) =-----
2. EC of given soil sample (in 1:2.5 soil water suspension) =-----

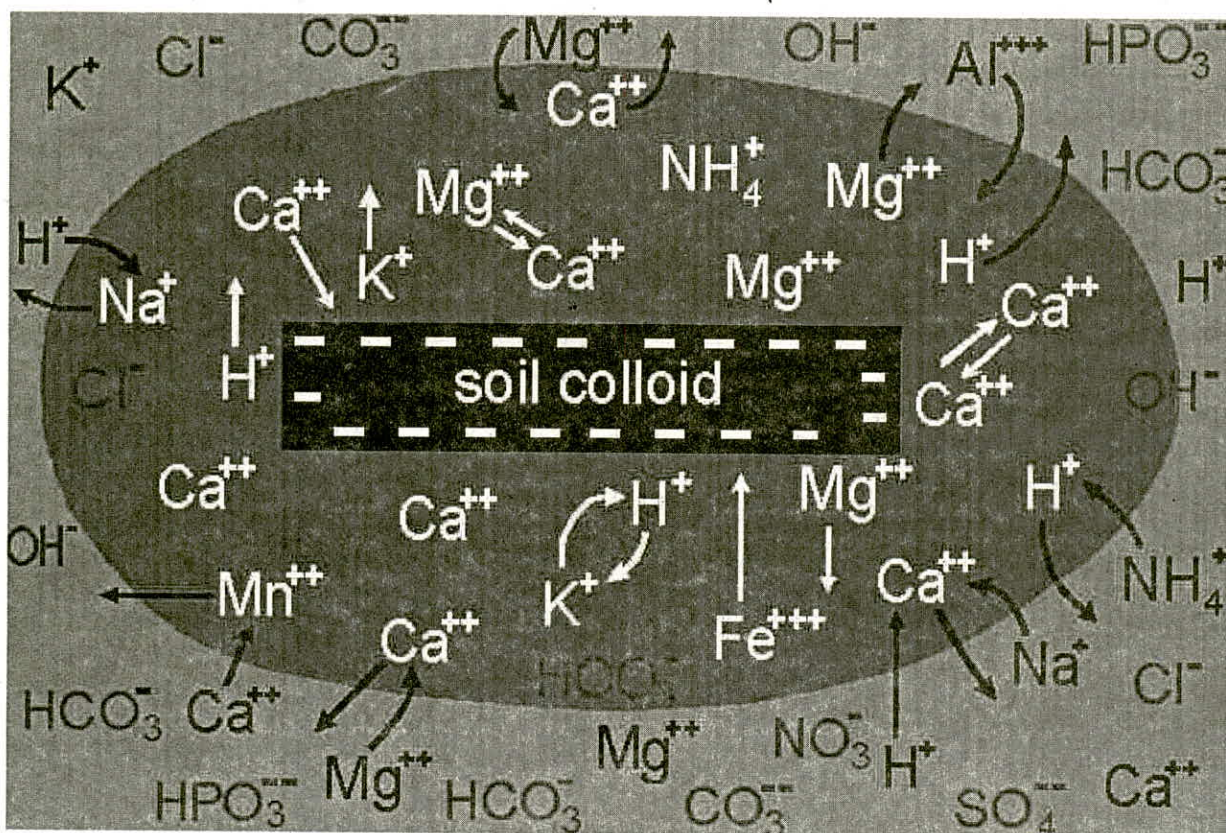
Exercise No: 14

Title : Determination of soil CEC, soluble cations and anions

Objective : Determination of cation and anion exchange capacity of soil

Determination of Cation Exchange Capacity in given Soil sample.

Principle: Cation Exchange capacity in calcarious or noncalcarious soil is preferably determined in 1 n ammonium acetate extract. CEC determined by saturation of soil with cation index (NH_4^+), removal by washing of excess cation, and subsequent replacement of the adsorbed cation by another cation (Na^+) and measurement of the index cation (NH_4^+) in the final extract (Rechards, 1954).



The exchange of one cation for another on the soil particles is called "cation exchange", and the number of exchange sites in a given mass of soil is called the cation exchange capacity (C.E.C.). Cation exchange capacity is an important soil property, because it influences a soil's ability to hold nutrients, remove contaminants from infiltrating water and buffer against changes in soil pH.

This C.E.C. is expressed in milli-equivalents per 100 grams of oven dry soil (me/100 g). A milli-equivalent is the mass of a cation which will exchange with one milligram of H^+ ions. A mg of H^+ will occupy 6×10^{20} negative charges on soil colloids (that's 600,000,000,000,000,000,000).

exchange sites). So, for example, a soil with a C.E.C. of 10 me/100 g soil would have 6×10^{21} negative charges and would accommodate 6×10^{21} positive charges in the form of cations adsorbed onto the surfaces of its colloidal particles.

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

Apparatus:

1. Flame photometer

A traditional and simple method for determining **sodium** and **potassium** in biological fluids involves the technique of emission **flame photometry**. This relies on the principle that an alkali metal salt drawn into a non-luminous flame will ionize, absorb energy from the flame and then emit light of a characteristic wavelength as the excited atoms decay to the unexcited ground state. The intensity of emission is proportional to the concentration of the element in the solution. You are probably familiar with the fact that if you sprinkle table salt (**NaCl**) into a gas flame then it glows bright orange (**KCl** gives a purple colour). This is the basic principle of flame photometry. A photocell detects the emitted light and converts it to a voltage, which can be recorded. Since Na^+ and K^+ emit light of different wavelengths (colours), by using appropriate coloured filters the emission due to Na^+ and K^+ (and hence their concentrations) can be specifically measured in the same sample. One drawback of flame photometers, however, is that they respond linearly to ion concentrations over a rather narrow concentration range so suitable dilutions usually have to be prepared.

2. Mechanical shaker 3. Centrifuge and centrifuge tube (50ml)

Reagents:

1. **Sodium acetate solution (1.0N):** dissolve 136g. Sodium acetate trihydrate in 950 ml distilled water and adjust pH by adding acetic acid or sodium hydroxide dilute to a volume of 1 L. (the pH of this solution should be approximately 8.2)..

2. Etanol 95%

3. **Ammonium acetate solution (1.0N):** Dissolve 77g ammonium acetate in 950 ml distilled water and adjust pH by adding acetic acid or liquid ammonium hydroxide to a volume of 1 L. (the pH of this solution should be approximately 7.0).

4. Standard stock solution:

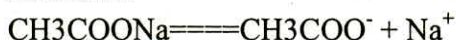
- Dry about 10g sodium chloride (NaCl) in an oven at 105°C for 3 hours, cool in a desiccators, and store in a tightly stoppered bottle.
- Dissolve 5.844g dried sodium chloride (NaCl) in 1.0N neutral ammonium acetate solution and make up the volume 1L. This solution contains 100me/L of Na (stock solution)

- Prepare a series of standard solutions from the stock solution as follows: take 0, 1.0, 2.5, 5.0, 7.5 and 10ml in 100ml volumetric flask and make up the volume by using 1.0N neutral ammonium acetate solution. This will give i.e. 0, 1.0, 2.5, 5.0, 7.5 and 10.0 me/L Na.

Method:

1. Take 5g. of soil in 50 ml centrifuge tube
2. Add 25ml Sodium acetate solution (1.0N), stopper tube and shake for 5 minutes.
3. Remove stopper from tube and centrifuge at 2000rpm approximately 10 minutes or until supernatant liquid is clear. Decant the supernatant as completely as possible and discard.
4. Repeat the sample in this manner with 25 ml of sodium acetate 1.0N four times discard the supernatant each time
5. Add 25 ml 95% ethanol to the tube, stopper, shake for 5 minutes remove the stopper and centrifuge until the supernatant clear (5 minutes). Decant and discard the supernatant liquid.
6. Wash the sample with 25 ml ethanol 3 times.
7. Add 25 ml of 1.0N ammonium acetate to the centrifuge tube, stopper and shake for 5 minutes. Remove stopper from tube and centrifuge at 2000rpm approximately 10 minutes or until supernatant liquid is clear. Decant the supernatant liquid as possible as in 100 ml volumetric flask. Repeat this extraction three times decanting into the same flask. This way ammonium ion will replace sodium ions which will come in the supernatant liquid.
8. Dilute the liquid of volumetric flask up to volume and determine sodium concentration by flame photometer after setting and preparation of standard curve of known concentration sodium (As given below)

Reaction:



Observation:

Record reading for standard curve and unknown solution as given exchangeable sodium determination.

Calculations:

$$\text{CEC in me/100g} = \frac{\text{Na conc. of extract in me/L} \times 100}{\text{Wt of soil in gram}} \times \frac{\text{volume of extract (ml)}}{1000}$$

$$= \frac{\text{Na conc. of extract in me/L} \times 10}{\text{Wt. of soil in gram}}$$

Here volume of extract = 100ml

Explanation of formula:

$$\text{CEC per ml of extract} = \frac{\text{Na conc.of extract in me/L} \times \text{volume of extract (ml)}}{1000}$$

$$\text{CEC per ml of extract} = \frac{X \times 100}{1000}$$

$$\text{CEC per me/g of soil} = \frac{\text{Na conc.of extract in me/L} \times \text{volume of extract (ml)}}{\text{Wt. of soil in gram} \times 1000}$$

$$\text{CEC per me/100g of soil} = \frac{\text{Na conc.of extract in me/L} \times \text{volume of extract (ml)} \times 100}{\text{Wt. of soil in gram} \times 1000}$$

$$\text{CEC per me/100g of soil} = \frac{X \times 10}{\text{Wt. of soil}}$$

X= Sodium concentration of extract in me/Litre (obtain from curve).

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

Atomic Absorption Spectrometer Volumetric flasks, 50 mL Adjustable micro-pipet (10-100 uL) and disposable tips

REAGENTS

Lanthanum oxide solution (La₂O₃), 5% - dissolve 50 g of La₂O₃ in 100 mL of concentrated Hydrochloric 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 interferences and simultaneously control a slight ionization interference which occurs when Ca and Mg are determined in an air acetylene flame.

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% La₂O₃ 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.

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

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

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

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

d.f. = dilution factor of sample

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कृषि मन्त्रालय

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