

**LABORATORY TESTING PROCEDURE FOR
SOIL & WATER SAMPLE ANALYSIS**

Practical Manual



Manure, Fertilizer & Soil Fertility Management

Dr.V. K. Singh

**Department of Soil Science
School of Agriculture Sciences
Shri Guru Ram Rai University, Dehradun (U.K)**

CONTENTS

Sr. NO.	LABORATORY TESTING PROCEDURE FOR Name of Test	Page No.
	SOIL & WATER SAMPLE ANALYSIS	
A.	Soil Sample Registration & Preparation in the Laboratory for Analysis	3
B.	Soil Sample Analysis	5
I.	Physical Tests	5
1	Determination of Soil Texture – International Pipette Method (Mechanical Analysis)	5
2	Determination of Saturation Moisture Percentage (Water Holding Capacity)	12
3	Determination of Bulk Density - 1) Weighing bottle method 2) Clod method 3) Core method	14
4	Determination of Hydraulic Conductivity of Soil - 1) Constant head method 2) Falling head method	20
5	Determination of Soil Moisture Content 1) Gravimetric method 2) Infrared moisture meter method	27
II.	Chemical Tests	31
1	Soil Reaction (pH)	31
2	Measurement of Electrical Conductivity (EC)	35
3	Determination of Organic Carbon - 1) Walkely & Black Method 2) UV spectrophotometer method	38
4	Determination of Calcium Carbonate (CaCO ₃) Free Lime 1) Acid neutralization method 2) Schrotus apparatus method	45
5	Determination of Nitrogen - Alkaline Permanganate Method	51
6	Determination of Phosphorous – Olsen's Method.	56

Sr. NO.	Name of Test	Page No.
7	Determination of Potassium on Flame Photometer	61
8	Determination of Sodium on Flame Photometer	66
9	Determination of Calcium & Magnesium – EDTA Titrimetric Method	71
10	Determination of Cation Exchange Capacity 1) Ammonium Saturation Method 2) Sodium Saturation Method	76
C.	Reclamation of Problematic Soil	85
1	Determination of Gypsum Requirement of Soil	85
D.	Water Sample Analysis	88
1	Determination of pH	88
2	Determination of Electrical Conductivity (EC)	90
3	Determination of Carbonates & Bicarbonates	93
4	Determination of Calcium & Magnesium - EDTA Titrimetric Method	96
5	Determination of Sodium on Flame Photometer	101
6	Determination of Chloride	104
7	Determination of Sulphate on Spectrophotometer	107
E	Appendices	110

A. SOIL SAMPLE REGISTRATION & PREPARATION IN THE LABORATORY FOR ANALYSIS

Soil Sampling

The method and procedure for obtaining soil samples vary according to the purpose of sampling. Analysis of soil samples may be needed for engineering and agricultural purposes. In this publication, soil sampling for agricultural purpose is described which is done for soil fertility evaluation and fertilizer recommendations for crops.

The results of even very carefully conducted soil analysis are as good as the soil sample itself. Thus, the efficiency of soil testing service depends upon the care and skill with which soil samples are collected. Non-representative samples constitute the largest single source of error in a soil fertility programme. It is to be noted that the most important phase of soil analysis is accomplished not in a laboratory but in the field where soils are sampled.

Soils vary from place to place. In view of this, efforts should be made to take the samples in such a way that it is fully representative of the field. Normally one to ten gram of soil is used for each chemical determination and represents as accurately as possible the entire surface 0-22 cm of soil, weighing about 2 million kg/ha.

Sampling tools and accessories

Depending upon the purpose and precision required, following tools may be needed for taking soil samples.

- Soil auger- it may be a tube auger, post hole or screw type auger or even a spade for taking samples.
- A clean bucket or a tray or a clean cloth for mixing the soil and sub sampling.
- Cloth bags of specific size.
- Copying pencil for markings and tags for tying cloth bags.
- Soil sample information sheet.

Selection of a sampling unit

A visual survey of the field should precede the actual sampling. Note the variation in slope, colour, texture, management and cropping pattern by traversing the field. Demarcate the field into uniform portions, each of which must be sampled separately. If all these conditions are similar, one field can be treated as a single sampling unit. Such unit should not exceed 1 to 2 hectares, and it must be an area to which a farmer is willing to give separate attention. The unit of sampling is a compromise between the expenditure, labour and time on one hand and precision on the other. In view of limited soil testing facilities, it has been suggested to adopt an alternate approach where a sample may be collected from an area of 20-50 ha to be

called as composite area soil sample and analyse the same for making a common recommendation for the whole area.

Sampling procedure

Prepare a map of the area to be covered in a survey showing different sampling unit boundaries. A plan of the number of samples and manner of composite sampling is entered on the map, different fields being designated by letters A, B, C etc. Each area is traverse separately. A slice of the plough-layer is cut at intervals of 15 to 20 steps or according to the area to be covered. Collect at least 10 to 15 samples from each sampling unit and place in a bucket or tray. Generally 10 to 20 spots must be taken for one composite sample depending on the size of the field.

Scrap away surface liter; obtain a uniform thick slice of soil from the surface to the plough depth from each place. A "V" shaped cut to a depth of 15 cm in the sampling spot using spade to remove 1 to 2 cm slice of soil. The sample may be collected on the blade of the spade and put in a clean bucket. In this way collect samples from all the spots marked for one sampling unit. In case of hard soil, samples are taken with the help of augur from the plough depth and collected in the bucket.



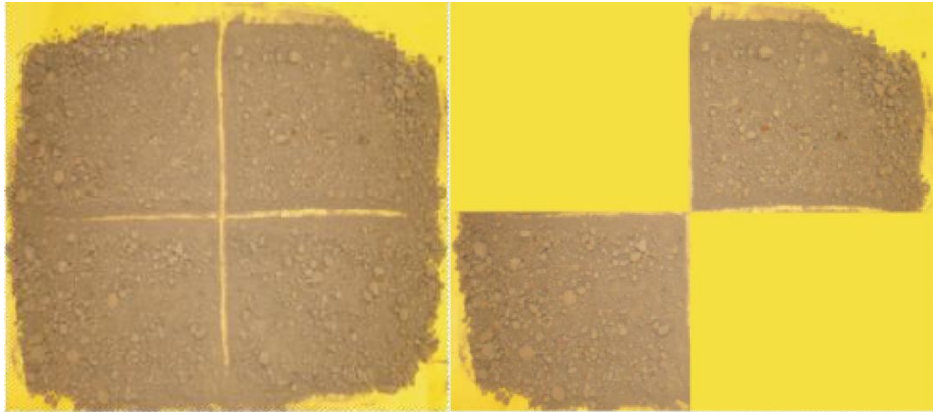
V shaped method of soil

sampling Guidelines for

sampling depth

S.No.	Crop	Soil sampling depth	
		Inches	cm
1	Grasses and grasslands	2	5
2	Rice, finger millet, groundnut, pearl millet, small millets <i>etc.</i> (shallow rooted crops)	6	15
3	Cotton, Redgram, sugarcane, banana, tapioca, vegetables <i>etc.</i> (deep rooted crops)	9	22
4	Perennial crops, plantations and orchard crops	Three soil samples at 12, 24 and 36 inches	Three soil samples at 30, 60 and 90 cm

Pour the soil from the bucket on a piece of clean paper or cloth and mix thoroughly. Spread the soil evenly and divide it into 4 quarters. Reject two opposite quarters and mix the rest of the soil again. Repeat the process till left with about half kg of soil, collect it and put in a clean cloth bag. Each bag should be properly marked to identify the sample.



Quarter method of soil sampling



Collect the sample in a clean cloth or polythene bag



Label with required information

The bag used for sampling must always be clean and free from any contamination. If the same bag is to be used for second time, turn it inside out and remove the soil particles. Write the details of the sample in the information sheet like name of the farmer, location of the farm, survey number, previous crop grown, present crop, crop to be grown in the next season, date of collection, name of the sampler etc., Put a copy of this information sheet in the bag. Tie the mouth of the bag carefully.

Precautions

- Do not sample unusual area like unevenly fertilized, marshy, old path, old channel, old bunds, area near the tree, site of previous compost piles and other unrepresentative sites.
- For a soft and moist soil, the tube auger or spade is considered satisfactory. For harder soil, a screw auger may be more convenient.
- Where crops have been planted in rows, collect samples from the middle of the rows so as to avoid the area where fertilizer has been band placed.
- Avoid any type of contamination at all stages. Soil samples should never be kept in the store along with fertilizer materials and detergents. Contamination is likely when the soil samples are spread out to dry in the vicinity of stored fertilizers or on floor where fertilizers were stored previously.
- Before putting soil samples in bags, they should be examined for cleanliness as well as for strength.
- Information sheet should be clearly written with copying pencil.

Sampling of salt affected soils

Salt affected soils may be sampled in two ways. Surface samples should be taken in the same way as for soil fertility analysis. These samples are used to determine gypsum requirement of the soil. For reclamation purpose, it is necessary to know the characteristics of lower soil depth also. Such soils are, therefore, sampled depth wise upto one meter. The samples may be removed from one to two spots per 0.4 hectare if the soil is uniformly salt affected. If patches are conspicuous then all big patches should be sampled separately. Soil is sampled depth wise separately (about $\frac{1}{2}$ kg from each depth) for 0-15 cm, 15-30 cm, 30-60 cm and 60-100 cm soil depths. If a stony layer is encountered during sampling, such a layer should be sampled separately and its depth noted. This is very important and must not be ignored.

Soil samples can be removed by a spade or if the auger is used then care should be taken to note the depth of 'concretion' (stones) or other impermeable layer (hard pan). If the soil shows evidence of profile development or distinct stratification, samples should be taken horizon wise. If a pit is dug and horizons are absent then mark the vertical side of the pit at 15, 30, 60 and 100 cm depth from the surface and collect about $\frac{1}{2}$ kg. Soil from every layer, cutting uniform slices of soil separately. In addition to the

above sampling, one surface soil sample should be taken as in the case of normal soil sampling for fertilizer recommendation.

Pack the samples and label the bags in the same way as is done for normal soil sampling, giving additional information about the depth of the sample. The sheet accompanying the sample must include the information on nature of soil, hardness and permeability of soil, salinity cause and source, if known, relief, seasonal rainfall, irrigation and frequency of water logging, water table, soil management history, crop species and conditions of plant cover and depth of the hard pan or concretion. As the salt concentration may vary greatly with vertical or horizontal distance and with moisture and time, account must be kept about time of irrigation, amount of irrigation or rain received prior to sampling.

Despatch of Soil Samples to the Laboratory

Before sending soil samples to the testing laboratory by a farmer, it should be ensured that proper identification marks are present on the sample bags as well as labels placed in the bags. It is essential that it should be written by copying pencil and not with ink because the ink will smudge and become illegible. The best way is to get the soil sampling bags from soil testing laboratory with most of the information printed or stencilled on them with indelible ink.

Compare the number and details on the bag with the dispatch list. The serial numbers of different places should be distinguished by putting the identification mark specific for each center. This may be in alphabets, say one for district and another for block/county and third for the village.

Pack the samples properly. Wooden boxes are most suitable for long transport. Sample bags may be packed only in clean bags never used for fertilizer or detergent packing.

Farmers may bring soil samples directly to the laboratory.

Registration of Samples :

1. The soil samples are received in the Sample Receiving Cell, where the condition and quantity of the samples are examined and acknowledgement slip is issued to the person delivering the sample.
2. The entry of soil sample is taken in the Sample Register (office) as per the relevant particulars furnished in the Register. Lab. No. is given to each sample.
3. Test required and expected date of reporting and other details along with the sample is then transferred to analysis.

Soil Sample Preparation for Analysis :

1. The collected soil samples are homogeneously mixed and left to attain equilibrium with air for 2 hours in the trays / paper dishes.
2. If the samples are dry there is no need to keep the samples in the oven. It can be directly

taken for further testing.

3. If the soil samples are wet, samples are dried in the oven at 25°C for 2 hours or more (depending upon the wetness of the sample). If samples found sticky even after drying, then the temperature may be raised by 2 to 5°C, but in any case it should not exceed 35°C.
4. After drying the soil, clods are crushed gently and grounded with the help of wooden pestle and mortar. Gravel, soft chalk, limestone, stones and concretions should be removed from the samples.
5. The soils are passed through 2.0 mm and 0.5 mm stainless steel sieve. The sufficient quantity of sieved soil sample is kept in plastic bag labeled with permanent ink marker (IC No. along with the sieve size 2.0 mm / 0.5 mm).

6. The plant residues, gravel, and other materials retained on the sieve may be discarded.
7. If the gravel content is substantial, the percent of the sample (W/W) may be recorded.
8. After the analysis of samples, the results are reviewed by the Higher Authority and entered in appropriate register.
9. The test reports should be signed by the authorized person.

Reviewing of Soil Samples :

1. After the completion of the analysis, the remaining samples are stored in the sampling room.
2. The advisory soil samples having test results not in normal range are retained for one month after sending the test results.
3. The samples are retained to recheck the test results, if any query is raised.
4. The soil samples of research studies are retained depending on the objective of the investigation of the project.

Reference Documents :

Jackson, M. L., 1967, Handling Soil Samples in the Laboratory, in soil "Chemical Analysis", Prentice Hall of India Pvt. Ltd., New Delhi, 2, 30-37.

Singh, Dhyan, Chhonkar, P. K. and Pande, R. N., 1999, Soil Testing in Soil, Plant, Water Analysis, Methods Manual IARI, ICAR, New Delhi, 1, 1-6.

B. SOIL SAMPLE ANALYSIS

I. PHYSICAL TESTS

1. DETERMINATION OF SOIL TEXTURE - INTERNATIONAL PIPETTE METHOD (MECHANICAL ANALYSIS)

Purpose :

The process of determining the amount of individual size separates of soil below 2 mm in diameter i.e. sand, silt and clay called particle size analysis. Particle size distribution has an important influence on soil permeability or water intake rate, water storage capacity ability to aggregate, crushing and the chemical makeup of the soil. The value of land, land use capability and soil management practices are largely determined by the texture.

The size limits for different fractions according to International system of classification is as follows – ISSS.

- | | | |
|----|-------------|-----------------------------|
| a) | Gravel | Greater than 2 mm diameter |
| b) | Coarse Sand | 2.0 to 0.2 mm diameter |
| c) | Fine Sand | 0.2 to 0.02 mm diameter |
| d) | Silt | 0.02 to 0.002 mm diameter |
| e) | Clay | Less than 0.002 mm diameter |

The size limits for different fractions are also classified according to CSSC, USDA.

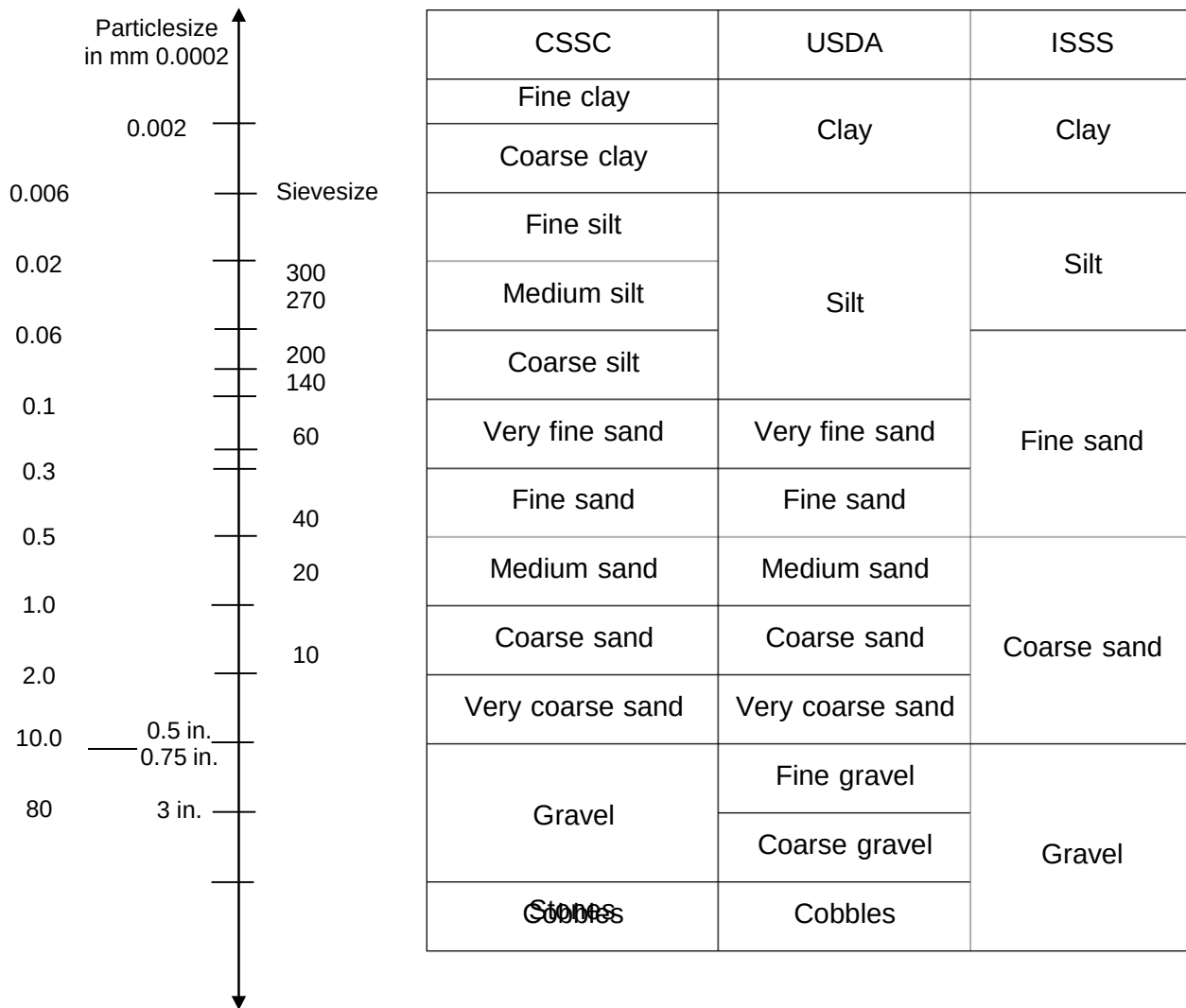


Fig. 1 : A comparison of particle size limits in 3 systems of particle size.

Principle :

Mechanical analysis of soils consists of essentially two distinct operations.

- i) Dispersion of the soil
- ii) Determination of particle size distribution by sedimentation (by Stokes Law)

Apparatus :

- i) Shaking machine
- ii) 1000 ml measuring stoppered glass cylinder
- iii) Thermometer
- iv) Pipette
- v) Stirrer
- vi) Stop watch
- vii) Oven
- viii) Physical balance
- ix) 500 ml plastic bottle.
- x) Sieve (2, 0.2, and 0.02 mm)
- xi) China dishes

Reagents :

- i) Sodium hexa-meta phosphate $[\text{NaPO}_3]_6$
- ii) Sodium carbonate (Na_2CO_3)
- iii) Dispersing reagent - Take 33 gm of Sodium Hexameta- phosphate and add 7 gm of Sodium carbonate and make the volume 1 litre with distilled water.

Procedure :

- i) For determination of soil texture, take 50 gm of air dry soil (passed through 2 mm sieve) in 500 ml bottle.

- ii) Add 100 ml above dispersion solution in 50 gm soil in 500 ml plastic bottle.
- iii) Shake a set of sample bottles at regular intervals for half an hour on shaking machine for preparing homogeneous solution.
- iv) Transfer above soil sample solution transferred to 1000 ml glass measuring cylinder and make solution 1000 ml by adding water.
- v) As per International approved system, shake the sample solution for 30 sec. Depending on the solution temperature and sedimentation chart, first pipetting is done with 50 ml pipette at 10 cm depth. In first pipetting, 50 ml solution sucked and transferred in 60 ml china dish. This sample solution contains mixed of clay and silt particles.
- vi) Depending on the solution temperature and sedimentation chart, second pipetting is done with 50 ml pipette at 10 cm depth. In second pipetting 50 ml solution sucked and transferred in 60 ml china dish. This solution contains clay particles in soil sample.
- vii) Transfer remaining soil solution in 1 lit. measuring cylinder by using 0.02 mm sieve and wash the material through the sieve using jet of water. Sand particles on sieve are collected in china dish.
- viii) Transfer pipetted solution in 3 dishes and dry overnight in an oven at 105°C, Cool in a desiccators and weigh quickly.
- ix) The weight of fine sand determined by deducting the weight of clay, silt and coarse sand particle from 100.

Setting time for Silt and Clay, 10 cm depth
At different temperatures (Sedimentation Time Chart)

Temperature °C	I – Pipetting Time Upper limit of silt (0.02 mm Dia.) min-Sec.	II – Pipetting Time Upper limit of Clay (0.002 mm Dia.) Hrs.-Min.
15	5-30	9-05
16	5-20	8-50
17	5-10	8-35
18	5-00	8-25
19	5-00	8-10
20	4-48	8-00
21	4-40	7-50
22	4-30	7-40
23	4-30	7-25
24	4-20	7-15
25	4-15	7-07
26	4-10	6-55
27	4-05	6-45
28	4-00	6-40
29	3-55	6-30
30	3-50	6-20
31	3-45	6-15
32	3-40	6-10
33	3-35	5-55

Calculations :

Coarse Sand (A gm) x 2 =% Coarse sand

In 50 gm soil sample A gm → Coarse sand

∴ 100 gm soil sample A gm x 2 Coarse sand (I)

In 1000 ml sample solution 50 gm soil

In 50 ml sample solution 2.5 gm soil

In 2.5 gm sample B gm → (Clay + Silt)

∴ In 100 gm sample (B x 100) / 2.5 = B x 40 gm ... (Clay + Silt)

In 2.5 gm sample C gm → (clay)

∴ 100 gm sample C x 40 gm (clay) (II)

∴ % Silt = (B x 40) – (C x 40) (III)

% Coarse sand = ----- (1)

% Clay = ----- (2)

% Silt = ----- (3)

∴ % Fine sand = 100 – (1) + (2) + (3)

Reference Documents :

Singh, Dhyan, Chhonkar, P. K. and Pande, R. N., 1999, Soil Testing in Soil, Plant, Water Analysis, Methods Manual IARI, ICAR, New Delhi, 1, 6-11.

Kadam, J. R., Shinde P. B., 2005, Practical Manual on Soil Physics
– A method manual, Department of Agricultural Chemistry and Soil Science, P.G.I., Rahuri, P-8

2. DETERMINATION OF SATURATION MOISTURE PERCENTAGE (WATER HOLDING CAPACITY)

Purpose :

The determination of water holding capacity in soils is important as it gives an idea of the capacity of soil to hold water for the use by crops. The light soils which do not hold such water require more frequent irrigations than heavy clay soils, well decomposed organic matter increases the water holding capacity. Exchangeable sodium and type of clay mineral also have a marked effect on water holding capacity.

Water holding capacity of soils is useful for selection of soils for irrigability classification. It also helps for comparing other properties of soils.

- Apparatus :**
- 1) Physical balance
 - 2) Perforated dish or circular brass boxes
 - 3) Enamel tray.
 - 4) Sieve (2 mm)
 - 5) Filter paper

Procedure :

- 1) Crush air-dry soil and pass through 2 mm sieve.
- 2) Place round filter paper and fix it to the internal perforated floor of the dish. The weight of the dish and filter paper is noted. The dish is then filled with soil by tapping the dish briskly & making plane the top of soil and find out its weight.
- 3) Place the set of perforated dishes in enamel tray. Pour the water in enamel tray at half of height of dish. Water may rise in dish through perforated bottom and moist the soil to its capacity. Keep it for 5 to 6 hours in water.
- 4) Take the dishes and place it on a filter paper sheet, so that the excess of water may drain away from the pores within half an hour. The dish containing moist soil is weighted and the weight is noted.

Observations :

- | | | | |
|----|---|---|--------|
| 1) | Weight of empty dish + filter paper | - | a gms. |
| 2) | Weight of empty dish + filter paper
+ air dry soil | - | b gms. |
| 3) | Weight of empty dish + filter paper
+ wet soil | - | c gms. |

Calculations :

$$\begin{aligned}\text{Saturation Moisture \%} &= \frac{(c - a) - (b - a)}{(b - a)} \times 100 \\ &= \frac{(c - a) - (b - a)}{(b - a)} \times 100\end{aligned}$$

Reference Documents :

Soil Testing Procedure Manual 2008, Marathwada Agricultural University,
Parbhani, P-22.

3. DETERMINATION OF BULK DENSITY -

Purpose :

The bulk density varies indirectly with total pore space present in the soil and gives a good estimate of porosity of soil. Bulk density is of great importance than particle density in understanding the physical condition of soil.

Soil bulk density is defined as the ratio of the mass of the oven dry soil to its bulk volume.

Method of determination :

Different methods are available for bulk density determination which differ how the soil sample is obtained and its volume is determined such as core, clod and excavation methods. There are other methods for the determination of bulk density where different principles are employed, for example, radiation method. Commonly used methods are weighing bottle, core and clod method.

1. Weighing bottle method :Principle :

The mass of the soil is determined by weighing the oven dry soil sample. The soil in small amounts, say 5-6 gm., is placed in a container which is tapped 15-20 times on a table by letting it fall from a height of about 2-3 cm. This tapping is assumed to produce the same packing as occurring naturally in the field, even though this assumption is not strictly correct. The volume of this packed soil will be equal to the volume of the container. Bulk density is calculated from the mass and volume of the soil.

Apparatus :

A weighing bottle (50 ml), balance and a burette of 50 ml capacity.

Procedure :

Weigh an empty 50 ml bottle. Fill the bottle with oven dry soil upto the brim by tapping and weigh it. Empty the bottle and determine its exact volume using burette.

Observations & Calculations :

Mass of empty bottle	= M_1 gm.
Mass of bottle + soil	= M_2 gm.
Mass of the soil	= $(M_2 - M_1)$ gm.
Volume of water filling the bottle	= V cm ³ .
Bulk density	= $(M_2 - M_1) / V$ gm.cm ⁻³ or Mg. m ⁻³

2. Clod Method :**Principle :**

A few pieces of soil clods are oven dried, weighed and coated with a water repellent substance (rubber solution) just enough to check the water entry into them. A number of such clod pieces are placed inside a graduated cylinder filled with water and the volume of water displaced by them is noted. Knowing the dry weight of the soil and its volume, the bulk density is calculated. The clod must be sufficiently stable to cohere during coating, weighing and handling.

Apparatus and Reagents :

Crepe rubber or smoke sheets, benzene, toluene, electric stirrer, wide mouthed container, a thin wire mesh, 250 ml. Graduated cylinder, weighing balance and oven.

Procedure :

Method of preparation of rubber solution :

Weigh 50 gm rubber sheets. Cut them into smaller pieces and soak in toluene (rubber to toluene ratio is kept at 1:5 by weight). Keep the mixture overnight in a tight container. Next day add benzene (440-

500 ml) and stir the contents thoroughly with the help of electric stirrer so that swollen rubber pieces are shattered and homogeneous solution is obtained. Dilute this solution with benzene to achieve concentration of 1:30 by weight. Instead of benzene, toluene can also be used for dilution but the benzene has the advantage that it dries rapidly when applied over the soil clod. Instead of rubber solution we can also use paraffin wax.

Coating Procedure :

Take a soil clod, oven dry it and note its weight. Wrap the clod in a thin wire mesh, dip in the rubber solution placed in wide mouthed container. Remove it momentarily. Repeat the process 3-4 times.

Volume Measurement :

Take a 250 ml graduated cylinder containing 150 ml of water. Put the coated soil clod into the cylinder and note the volume of water displaced by the clod. The volume of the displaced water will be equal to the volume of the clod.

Observations & Calculations :

Mass of oven dry clod	= M gm.	Volume of water in the
cylinder	= $V_1 \text{ cm}^3$	Volume of water + Clod
in the cylinder	= $V_2 \text{ cm}^3$	Volume of the clod =
$(V_2 - V_1) \text{ cm}^3$		
Bulk density of the clod	= $M / (V_2 - V_1) \text{ gm.cm}^3$	or Mg.m^{-3}

3. Core Method :

This is a field method for bulk density determination.

Principle :

In this method a cylindrical metal sampler or core of known volume is driven into the ground to the desired depth and carefully removed to preserve a known volume of sample as it existed in situ. This core sample is dried at 105°C and weighed. Bulk density is the oven dried mass divided by volume of the sample. The core method is usually unsatisfactory if gravels are present in the soil.

Apparatus :

A core sampler, sharp knife, a tray, moisture boxes and oven.

Procedure :

Drive the sample into either a vertical or horizontal soil surface far enough to fill the sample but not to compress the soil in the confined space. Carefully remove the sampler and its contents. Trim the soil extending beyond the sampler with a sharp knife. The soil sample volume is the same as the volume of the sampler or the core. Transfer the wet soil to a tray and weight it. Take a portion of the sample in a moisture box, weigh and place it in an oven at 105°C for about 24 hours and weigh it again.

Observation & Calculations :

Mass of wet bulk soil sample	= M_1 gm.
Mass of the moisture box	= M_2 gm.
box + wet soil	= M_3 gm.
box + oven dry soil	= M_4 gm.
Mass of wet soil	= $(M_3 - M_2)$ gm.
Mass of oven dry soil	= $(M_4 - M_2)$ gm.

Oven dry mass of bulk soil sample

$$= (M_4 - M_2) M_1 / (M_3 - M_2) \text{ gm or}$$

$$\text{say } = M_5 \text{ gm.}$$

Volume of bulk sample / core sampler = $V \text{ cm}^3$ or $\pi r^2 h \text{ cm}^3$ Where r is the radius in cm. and h is the height of the core in cm.

$$\text{Bulk density} = M_5 / V \text{ gm.cm}^3$$

Precautions :

Core samples should not be taken in too wet or too dry condition of the soil. In wet soils, the friction along the sides of the sampler and vibrations due to hammering are likely to result in viscous flow of the soil and thus, compression of the sample. When this occurs, the sample obtained is unrepresentative, being more dense than the soil in situ. Compression may occur even in dry soils if they are very loose. Also, hammering the sampler into dry soil often shatters the sample and makes it loose.

Ratings :

Textural Class	Bulk Density	Pore Space (%)
Sandy Soil	1.6	40
Loam	1.4	47
Silt Loam	1.3	50
Clay	1.1	58

Reference Documents :

Kadam, J. R., Shinde P. B., 2005, Practical Manual on Soil Physics
– A method manual, Department of Agricultural Chemistry and Soil Science, P.G.I., Rahuri,
P-24.

Soil Testing Procedure Manual 2008, Marathwada Agricultural University,
Parbhani, P-30.

4. DETERMINATION OF HYDRAULIC CONDUCTIVITY OF SOIL

Purpose :

The saturated hydraulic conductivity is a measure of readiness with which a saturated soil transmits water through its body and is expressed as length per unit time. Hydraulic conductivity is of a considerable importance for irrigation, drainage and evaporation studies. It depends upon properties of water / fluid and on the porosity, pore size distribution and continuity of soil pores. It is generally assumed to be a constant physical property of a soil for any given positioning the field at any given time varying only with respect to water content or water potential. Since viscosity and density of water passing through the soil affect the hydraulic conductivity, this soil property varies for different quality of waters. The hydraulic conductivity of soil varies from 0.001 cm/hr in a fine clay to over 25.0 cm/hr. on coarse sand.

There are several methods for determining the saturated hydraulic conductivity in the laboratory and in the field. In principle the hydraulic conductivity of soil is calculated from Darcy's Law after measuring the soil water flux and hydraulic gradient.

1. Constant Water Head Method :

Principle :

When a constant head of water is maintained (Fig. 1) on one end of saturated column of soil of length L cm, the volume of water Q cm³, percolating through the other end per unit cross-sectional area A cm² of the soil column per unit time t minute, is directly proportional to the hydraulic gradient ($\Delta H/L$) over the length of the soil. Thus ,

$$Q / At = - K_s \Delta H / L \quad \dots \quad (1)$$

Fig. 1 : Darcy's Experimental Arrangement

According to Darcy's Law the proportionality constant K_s in equation (1) is the saturated hydraulic conductivity of the soil. The symbol H stands for the total head, which is the sum of hydraulic head (h) and the gravity head (z) at any point in the soil column. The difference of H at the top and the bottom of soil column divided by the length of the column gives the hydraulic or the total head gradient. Thus,

$$H_{\text{top}} = h_{\text{top}} + Z_{\text{top}} \quad \dots (2)$$

$$H_{\text{bottom}} = h_{\text{bottom}} + Z_{\text{bottom}} \quad \dots (3)$$

$$\begin{aligned} \Delta H &= h_{\text{top}} - H_{\text{bottom}} \\ &= (h_{\text{top}} + Z_{\text{top}}) - (h_{\text{bottom}} + Z_{\text{bottom}}) \quad \dots (4) \end{aligned}$$

Apparatus :

Brass permeameters of 7 cm inner diameter and 10 cm length with the perforated bottoms (the dimensions of permeameters can vary but smaller diameter causes more error due to wall effect), a stand for supporting the permeameter, a water reservoir with an arrangement for maintaining a constant head over the soil column, a stop watch, graduated cylinders and measuring rods.

Procedure :

1. Place a filter paper disc on the screen of the permeameter.
2. Take 200 gm of 2 mm sieved air dry soil.
3. Dump the entire sample in one lot into the permeameter and pack it by tapping the permeameter 15 to 20 times on a wooden block from a height of 2 to 3 cm.
4. Place the filter paper disc on the top of the soil.
5. Place the permeameter in a tray filled with water, keeping the water level slightly above the bottom of the sample and allow it to soak overnight (14 –16 hrs) or longer till fully wet at the surface.
6. Raise the water level in the tray to coincide with the top of the soil in the permeameter for complete saturation.
7. Place the permeameter on the stand and start the siphon to maintain a constant head (2 – 3 cm) on the top of the soil (do not allow the water to flow over the top of the permeameter).
8. Keep at least 4 to 6 replications (if different students are using the same soil, then their data can be used as replicates).
9. Note down the time when the water head on soil sample becomes constant and a steady flow is obtained.

10. Collect the percolate in a graduated cylinder and measure the volume at pre-decided interval of time.
11. Record a few consecutive readings till the flux becomes constant and measure the exact water head on the soil and then discontinue the experiment. Measure the length of soil column with the measuring scale.

Observations & Calculations :

Diameter of the permeameter = d cm Cross sectional
 area of the permeameter = A cm² Depth of water above the soil = h
 cm.
 Length of soil column = L cm Time for which
 percolate collected = T min.
 Volume of percolate collected = Q cm³ or ml Hydraulic gradient
 = $(L + h) / L$
Saturated hydraulic conductivity = $QL / At (L + h)$ cm./min

2. Falling head method :

Principle :

In this method, drop in water level in a narrow tube is measure instead of flow. This method is better adopted for slowly permeable soils.

Suppose in a small time ' dt ', there is drop of ' dh ' height of water in a narrow tube of cross sectional area ' a ' inverted over a saturated soil whose cross sectional area ' A ' and length ' L '.

The flux ' dq ' in time ' dt ' will be –

$$dq = a.dh \dots (1)$$

In terms of Darcy's Law,

$$dq = -K.A.dt (h/L) \dots (2) \text{from equations (1)}$$

and (2),

$$K.A.dt (h/L) = -a.dh \quad \text{or} \quad dt = -a.(dh/h) L/K.A \text{Integrating}$$

between t_1, h_1 and t_2, h_2 gives

$$K = (aL/At) \ln (h_1/h_2)$$

Apparatus :

Special apparatus (Fig....) consists of a galvanized iron cylinder (40 cm in length and 30 cm in diameter) with a conical top to which a vertical glass tube of small diameter is attached.

Fig. : Falling head permeameter

Procedure :

1. Push the cylinder into the soil to a depth for which determination is to be made and assemble the whole apparatus.
2. Wet the sample from below by water supply through a three way stop cock and lower porous plate.
3. Fill the space above the sample by introducing water with pipette or syringe at the top of the sample until it overflows.
4. Record the time for water level to fall from h_1 to h_2 . make additional 2 – 3 such measurements.

Observations and Calculations :

Diameter of stand pipe	= d cm
Cross sectional area of stand pipe	= $a \text{ cm}^2$
Length of the sample	= L cm
Diameter of the sample	= D cm
Cross sectional area of the sample (A)	= $\pi r^2 \text{ cm}^2$
Initial hydraulic head	= $h_1 \text{ cm}$
Final hydraulic head	= $h_2 \text{ cm}$
Time taken for change in head	= t sec
Saturated hydraulic conductivity	= $(a_l / A t) \ln (h_1/h_2) \text{ cm./s}$

Hydraulic conductivity ratings :

Rating	Ks (cm./hr)
Very slow	< 0.125
Slow	0.125 – 0.50
Moderately slow	0.50 – 2.0
Moderate	2.0 – 6.25
Moderately rapid	6.25 – 12.5
Rapid	12.5 – 25.0
Very rapid	> 25.0

Reference Documents :

Kadam, J. R., Shinde P. B., 2005, Practical Manual on Soil Physics
– A method manual, Department of Agricultural Chemistry and Soil Science, P.G.I., Rahuri,
P-59.

Soil Testing Procedure Manual 2008, Marathwada Agricultural University,
Parbhani, P-35.

5. DETERMINATION OF SOIL MOISTURE CONTENT

Purpose :

There are numerous methods available for the determination of soil water content. These methods can be divided into two categories i.e. the direct and indirect methods.

In direct method, the amount of water present in a given soil is directly determined whereas in indirect methods, a soil property or some reaction products relating to soil water content is determined. In other words, a calibration curve indicating the relation of the soil properties and soil water content is first prepared and then used for the estimation of water content.

1. Gravimetric Method (Direct Method) :

Gravimetric method is the simplest and most widely used direct method. It is frequently used for the calibration of other indirect methods.

Principle :

Disturbed or undisturbed wet soil samples are weighed, dried to constant weight in an oven at 105°C and reweighed. From these measurements, the water content on dry mass basis is calculated. It can be expressed on a volume basis by multiplying it with the bulk density.

Apparatus :

A sampling tool – auger, Soil cores or some other suitable device, Moisture box, oven and a desiccators with an active desiccant (calcium chloride).

Procedure :

1. Place the moist soil sample in a moisture box and weight it immediately.
2. Place the box with lid off in an oven (105°C) and dry the soil to a constant weight.
3. Remove the sample from the oven, replacing the lid, and place the box in the desiccators until it is cool.
4. Weigh it and also determine the mass of the empty moisture box. Determine the mass of the moisture.

Observations :

Mass of empty moisture box	= M_1 gm.	Mass of moisture
box + Moist soil	= M_2 gm.	Mass of moisture
box + oven dry soil	= M_3 gm.	

Calculations :

Mass of water in the soil	= $(M_2 - M_3)$ gm.
Mass of the oven dry soil	= $(M_3 - M_1)$ g.
Percentage moisture content on dry mass basis (θ_g)	= $(M_2 - M_3) 100 / (M_3 - M_1)$
Percentage moisture content on volume basis (θ_v)	= $(\theta_g) \times D_b / D_w$ Where D_b is the bulk density of soil & D_w is the density of water

2 . Infra Red Moisture Meter Method (Direct Method) :

This method is the simplest and quick method. It gives moisture content on wet weight basis. This method is useful for determining moisture content in soils which are hygroscopic.

Principle :

Wet soil samples are dried in Infra Red Moisture meter under exposure to infra red radiation. The balance scale of meter is divided directly in moisture percentages from 0 to 100% with least count of 0.2%

Apparatus : Infra red moisture meter with all accessories.

Procedure :

1. Turn the scale lamp ON by means of toggle switch.
2. Turn the scale adjusting knob and rotate scale until 100% mark coincides with the index
3. Move the pointer to index by turning pointer adjusting knob in opposite direction to the direction pointer must move to coincide with index.
4. Rotate scale until 0% mark coincides with index.
5. Raise the lamp housing and carefully distribute wet soil on sample pan until pointer returns to index. This weight of material corresponds to 100 divisions of the scale.
6. Lower the lamp housing and turn the infra red lamp ON.
7. Set proper drying temperature by adjusting auto transformer control.
8. Rotate the scale by scale adjusting knob until pointer returns to index. Read the percentage of moisture lost. (P)

Observations :

Percentage moisture on wet weight basis = P

Calculations :

Percentage moisture on dry weight basis = $100P/100-P$

Reference Documents :

Kadam, J. R., Shinde P. B., 2005, Practical Manual on Soil Physics
– A method manual, Department of Agricultural Chemistry and Soil Science, P.G.I., Rahuri,
P-29.

Soil Testing Procedure Manual 2008, Marathwada Agricultural University,
Parbhani, P-22.

II. CHEMICAL TESTS

1. SOIL REACTION (pH) Purpose :

The determination of pH in soil is important as it plays a great role in availability of nutrients to plants. This determination can be done more accurately in the laboratory by electrometric method.

pH determination is useful for soil classification on the basis of acidity or alkalinity.

Principle :

The electrometric determination of pH by a pH meter is based on measuring the e.m.f. (millivolts) of a pH cell both a reference buffer and then with a test solution. The change in the potential difference at 25°C for 1 pH unit is 59.1 mV. The pH of a soil is a measure of the hydrogen or hydroxyl ion activity of the soil – water system. It indicates whether the soil is acidic, neutral or alkaline in reaction. By shaking a certain amount of soil with a certain amount of liquid, soil suspension is brought in equilibrium with a supernatant solution. In the supernatant solution the pH is measured potentiometrically on a direct reading pH meter using a glass electrode with a saturated KCl – calomel reference electrode.

Reagents :

- i) Standard buffer solutions : Dissolve one commercially available buffer tablet each of pH 4.0, 7.0 and 9.2 in freshly prepared distilled water separately and make up the volume to 100 ml. Prepare the fresh solution every week as these solutions are unstable. Alternatively the buffer solutions can be prepared in the laboratory as given below.

- ii) 0.05 M Potassium hydrogen phthalate ($\text{KHC}_3\text{H}_4\text{O}_4$, Mol. Wt. 204.22) : Dissolve 10.21 gm AR grade potassium hydrogen phthalate in warm water and making volume to 1 L. This gives a pH of 4.00 at 25°C and can be used as standard buffer.
- iii) Buffer solution pH 6.86 : Potassium dihydrogen phosphate + Disodium hydrogen phosphate, each 0.025 M – Dissolve 3.40 gm of potassium dihydrogen orthophosphate and 4.45 gm disodium hydrogen orthophosphate dihydrate (Sorenson's salt – $\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$) to 1 L in distilled water.
- iv) Buffer solution pH 9.2 : Dissolve 3.81 gm sodium tetraborate (A.R.) in water and dilute to 1000 ml.

Apparatus :

- i) pH meter with glass electrodes
- ii) Thermometer
- iii) Glass beaker (100 ml)
- iv) Glass rod

Procedure :

- 1) Weigh 20 gm of 2.0 mm air dry soil into a beaker. Add 50 ml of distilled water and stir with a glass rod thoroughly for about 5 minutes and keep for half an hour.
- 2) In the mean time turn the pH meter ON, allow it to warm up for 15 minutes. Standardize the glass electrode using standard buffer of pH = 7 and calibrate with the buffer pH = 4 or pH = 9.2.
- 3) Dip the electrodes in the beakers containing the soil water suspension with constant stirring.
- 4) While recording pH, switch the pH meter to pH reading, wait 30 seconds and record the pH value to the nearest 0.1 unit. Put the pH meter in standby mode immediately after recording.

- 5) Remove the electrodes from soil suspension and clean the electrodes with distilled water.
- 6) Rinse the electrodes after each determination and carefully blot them dry with filter paper before the next determination. Standardize the glass electrodes after every 10 determinations.
- 7) Dip the electrodes in distilled water, when not in use and ensure that the reference electrode always contains saturated potassium chloride solution in contact with solid potassium chloride crystals.
- 8) Three to four drops of toluene are added in standard buffer solutions to prevent growth of mould.

Ratings :

< 4.5	Extremely Acidic
4.6 to 5.2	Strongly Acidic
5.3 to 6.0	Moderately Acidic
6.1 to 6.5	Slightly Acidic
6.6 to 7.0	Neutral
7.1 to 7.5	Slightly Alkaline
7.6 to 8.3	Moderately Alkaline
8.4 to 9	Strongly Alkaline
> 9	Extremely Alkaline

Reference Documents :

Davis, J. and Freitas F., 1970, In Physical & Chemical Methods of Soil & Water Analysis, FAO of United Nations, Rome, Soil Bulletin, 10, III-1 : 65-67.

Singh, Dhyan, Chhonkar, P.K. and Pande R.N., 1999, Soil Reaction in Soil, Plant, Water analysis Method Manual, IARI, ICAR, New Delhi, 1 : 4.2 (b) 11-13.

2. Measurement of Electrical Conductivity (EC) in Soil Purpose :

Salted soils are classified on the basis of two criteria, one is on the basis of total soluble salt (TSS) content and another is exchangeable sodium percentage (ESP) or more recently sodium Absorption ration (SAR). Ions in water conduct electrical current, therefore electrical conductivity is fast, simple method of estimating amount of total soluble salt (TSS) in soil sample. Electrical conductivity is expressed in dS/m.

Principle :

The electrical conductivity of water extract of soil gives a measure of soluble salt content of the soil. Pure water is very poor conductor of electric current, whereas water containing the dissolved salts in soil conducts current approximately in proportion to the amount of soluble salts present. Based on this fact, the measurement of electrical conductivity of an extract gives a satisfactory indication of the total concentration of ionized constituents. The conductivity of the soil is the specific conductivity at 25°C of water extract obtained from a soil and water mixture of a definite ration. It is measured on a conductivity meter and normally reported in dS/m or milimhos/cm and the value gives information on the total amount of the solublesalts present in soil, i.e. on the degree of salinity.

Apparatus :

- 1) Digital conductivity meter
- 2) Conductivity cell.
- 3) Glass beaker (100 ml)
- 4) Glass rod

Reagent :

0.01N Potassium chloride solution : Dry a small quantity of A.R. grade Potassium chloride at 60°C for 2 hours. Weigh 0.7456 gm of it and dissolve in freshly prepared distilled water and make to one litre. This solution gives and electrical conductivity of 1411.8×10^{-3} i.e. 1.41 dS/m at 25°C.

Procedure :

- i) Calibrate the conductivity cell with the help of standard KCL solution and determine the cell constant.
- ii) The soil water suspension of 20 gm : 50 ml ratio prepared for the determination of pH can also be used for conductivity measurements. After recording the pH, allow the soil water suspension in the beaker to settle for additional half an hour (the total intermittently shaking period is 1 hr.)
- iii) After the calibration dip the conductivity cell in the supernatant liquid of the soil water suspension. Read the conductivity of test solution in proper conductance range,
- iv) Remove the cell from soil suspension, clean with distilled water and dip into a beaker of distilled water. EC is expressed as dS.m^{-1}
- v) Dip the conductivity cell in distilled water when not in use.
- vi) Record the temperature of soil water suspension during the test. (See Appendix-9).

Calculations :

The cell constant K is given by

$$\text{Cell constant (K)} = \frac{\text{Known conductivity of 0.01 N KCL}}{\text{Conductivity of 0.01 N KCL measured}}$$

$$\text{EC}_{e25} = \text{EC}_T \times K \times ft$$

Where EC_{e25} is the conductivity of the extract at 25°C .

Ratings : EC in dS.m^{-1}

0 to 1	Good soil
1 to 2	poor seed emergence
2 to 4	Harmful to some crops, e.g. Pulses.
Above 4	Harmful to most of crops.

Reference :

Davis, J. and Freitas F., 1970, In Physical & Chemical Methods of Soil & Water Analysis, FAO of United Nations, Rome, Soil Bulletin, 10, III-1 : 68-70.

Singh, Dhyani, Chhankar, P.K. and Pande R.N., 1999, Electrical Conductivity in Soil, Water analysis Method Manual, IARI, ICAR, New Delhi, 1 : 4.2 (b) 14-16.

3. DETERMINATION OF ORGANIC CARBON IN SOIL :

Purpose :

Besides its value as a source of plant nutrients, organic matter has a favourable effect upon soil physical properties. Organic matter also contains the informable effective of exchangeable sodium on soils. The determination of organic carbon in soil serves indirectly as measure of available nitrogen. The organic matter content of a typically well drained mineral soil is low varying from 1 to 6% by weight in the top soil and even less in the subsoil. The influence of organic matter (OM) on soil properties and consequently on plant growth is far greater even though the percentage of organic matter (OM) is less in the soil.

1. Walkley and Black Method :

Principle :

Organic carbon is oxidized with potassium dichromate in the presence of concentrated sulphuric acid. Potassium dichromate produces nascent oxygen, which combines with the carbon of organic matter to produce CO_2 . The excess volume of $\text{K}_2\text{Cr}_2\text{O}_7$ is titrated against the standard solution of ferrous ammonium sulphate in presence of H_3PO_4 , using ferroin to detect the first appearance of unoxidised ferrous iron and thus volume of $\text{K}_2\text{Cr}_2\text{O}_7$ can be found out which is actually required to oxidize organic carbon.



(Mol. wt. of $\text{K}_2\text{Cr}_2\text{O}_7 = 294.212$, $\longrightarrow$

Eq. wt. of $\text{K}_2\text{Cr}_2\text{O}_7 = 294.212/6 = 49.03$) $2 \text{K}_2\text{Cr}_2\text{O}_7 = 3\text{C}$,

$$\text{K}_2\text{Cr}_2\text{O}_7 = 3\text{C}/2 \quad \text{OR}$$

$49.03 \text{ g K}_2\text{Cr}_2\text{O}_7 = 12\text{C}/4 = 3.0 \text{ g CAs } 1000 \text{ cc (N)}$

$\text{K}_2\text{Cr}_2\text{O}_7 = 3.0 \text{ g C}$

$1 \text{ cc (N) K}_2\text{Cr}_2\text{O}_7 = 3 \text{ g C} / 1000 = 0.003 \text{ g C}$

Apparatus :

1. Conical flask – 500 ml
2. Pipettes – 2 ml, 10 ml, & 20 ml capacity
3. Burette – 50 ml capacity
4. Volumetric measuring flask – 2 Nos. (1 Lit. capacity)
5. Reagent bottles.
6. Asbestos sheet

Reagents :

1. 1 N potassium dichromate : Dissolve 49.04 AR grade $K_2Cr_2O_7$ (dry) in distilled water and make up the volume to one litre.
2. Concentrated sulphuric acid (Sp. Gravity 1.84, 96%) : If the soil contains chloride, then 1.25% silver sulphate may be added in H_2SO_4 .
3. Orthophosphoric acid (Sp. Gravity 1.75, 85%)
4. Sodium Fluoride (chemically pure)
5. 0.5 N Ferrous ammonium sulphate – Dissolve 196.0 gm of AR grade Ferrous ammonium sulphate in distilled water, add 20 ml of concentrated H_2SO_4 and make volume to one litre. The ferrous ammonium sulphate should be from a fresh lot and light green in colour.
6. Ferroin indicator

Procedure :

1. Weigh 1 gm. of 0.5 mm sieved soil into dry 500 ml conical flask. Add 10 ml of $K_2Cr_2O_7$ into the flask with pipette and swirl.
2. Add rapidly with a burette 20 ml conc. H_2SO_4 and swirl gently until soil and reagents are mixed then more vigorously for one minute.

3. Allow the reaction to proceed for 30 min on asbestos sheet to avoid burning of table due to release of intense heat due to reaction of sulphuric acid.
4. Add slowly 200 ml of distilled water, 10 ml of concentrated orthophosphoric acid and add about 0.2 gm NaF (one small teaspoon) and allow the sample to stand for 1.5 hrs. The titration end point is clear in a cooled solution.
5. Just before titration add 1 ml ferroin indicator into the conical flask. Titrate the excess $K_2Cr_2O_7$ with 0.5 N ferrous ammonium sulphate till the colour flashes from yellowish green to greenish and finally brownish red at the end point.
6. Simultaneously blank test is run without soil.

Observation Table :

Sr. No.	Lab. No.	Blank reading (B)	Burette reading (S)	Difference (B – S)	% Organic carbon	% Organic matter

Calculations :

$$\% \text{ Organic carbon} = (B - S) \times N \times 0.003 \times \frac{100}{\text{Wt. of soil (oven dry)}}$$

Where, B = ml of std. 0.5 N ferrous ammonium sulphate required for blank.

S = ml of std. 0.5 N ferrous ammonium sulphate required for soil sample. N = Normality of std. ferrous ammonium sulphate (0.5N)

The correction factor 1.3 is multiplied as according to Walkley and Black method only estimated 77% carbon (av. Value).

The result can be converted to corrected total organic carbon by multiplying the factor $100/77 = 1.3$

Soil organic matter contains (58%) of organic carbon, the percentage of organic carbon multiplied by $100/58 = 1.724$ which gives the percentage of organic matter i.e.

$$\text{Organic matter} = \text{Organic Carbon} \times 1.724$$

2. UV Spectrophotometer Method :**Apparatus :**

1. UV spectrophotometer
2. Volumetric flask

Reagents :

1. 1N potassium dichromate : Dissolve 49.04 AR grade $\text{K}_2\text{Cr}_2\text{O}_7$ (dry) in distilled water and make up the volume to one litre.
2. 97% conc. Sulphuric acid

Procedure :**Preparation of Standard Curve :**

Take 1 gm sucrose and add to it 1000 ml distilled water. From this solution take 0,1, 5, 10, 15, 20, 25, 30, 35, 40, 45, 50 ml in 100 ml flask and add 10 ml potassium dichromate and 20 ml sulphuric acid. Shake well and allow the mixture to cool on asbestos sheet, Make the volume of each solution to 100 ml with distilled water and observe optical densities at 660 nm. wavelength. Prepare standard curve and calculate factor F.

Sucrose Sol. Ml	Sucrose ppm	Carbon ppm	Carbon %	O. D. Reading	Carbon % for 1 O. D.
Blank	0	0	0		
1	10	4.2	0.04		
5	50	21	0.21		
10	100	42	0.42		
15	150	63	0.63		
20	200	84	0.84		
25	250	105	1.05		
30	300	126	1.26		
35	350	147	1.47		
40	400	168	1.68		
45	450	189	1.89		
50	500	210	2.10		
Total					X

$$\text{Factor F} = X/11$$

Determination of Organic Carbon

Sieve the soil sample with 1 mm sieve and take 1 gm of sieved soil sample in 100 ml flask. Add to it 10 ml potassium dichromate and 20 ml sulphuric acid, shake well and allow it to cool on asbestos sheet. Make the volume to 100 ml with distilled water and keep it overnight. Measure optical density at 660 nm wavelength on spectrophotometer.

Organic Carbon % = Optical density x Factor F

Ratings :

% Organic carbon		
1)	Less than 0.20	Very low
2)	0.21 to 0.40	Low
3)	0.41 to 0.60	Moderate
4)	0.61 to 0.80	Moderately high
5)	0.81 to 1.0	High
6)	More than 1.0	Very high

Reference Documents :

- 1) Singh, Dhyan, Chhonkar, P.K. and Pande R.N., 1999, SoilOrganic Carbon in “Soil, plant, water Analysis” A methods manual, Indian Agricultural Research Institute, Indian Council of Agricultural Research, New Delhi, 1.4.6 : 19-19.
- 2) Page, A.L., Miller, R.H. and Keeny, D.R. 1982, Organic Carbon in Methods of Soil Analysis, Chemical & Microbiological properties, American Society of Agronomy, Inc, Soil Science Society of America, Inc, Madison, Wilconsin, USA, 9(2) : 570-571.
- 3) Soil & Water Analysis Methods – Soil Survey & Analysis dept., Commissionorate of agriculture, Maharashtra State, Pune-5, P. 5-7.

4. DETERMINATION OF CALCIUM CARBONATE (FREE LIME) IN SOIL :

Purpose :

Alkaline earth carbonates that occur in significant amounts in soils consist of calcite, dolomite and possibly magnesite and occur commonly in the silt size fraction. These influence the texture of the soil when present in appreciable amounts. These are important constituents of alkali soils, they constitute a potential source of calcium and magnesium. Lime aids in preserving soil structure and may serve as a source of calcium in the reclamation of alkali soil.

Zonal soils of arid regions usually contains accumulation of lime at some point in the profile. Calcareous soil contains an accumulation of calcium and magnesium carbonated in varying proportions throughout the soil profile.

In calcareous soils if CaCO_3 is present in problematic amount improvement may be done by drainage of sub-soil for breaking the hard pan formed due to CaCO_3 accumulation at lower depth and leaching. Acid forming substances like S, FeSO_4 , $\text{Al}_2(\text{SO}_4)_3$ may also used followed by leaching.

1. Acid Neutralization Method :

Principle :

Soil is treated with an excess of standard hydrochloric acid, destroying carbonates. The amount of excess acid is determined by titration with standard sodium hydroxide, after separation from the soil by filtration or centrifugation. The acid dissolves a certain amount of iron and aluminium from oxides and soil materials and these metals are precipitated as hydroxides when the pH rises towards the end of the filtration with alkali. The amount of acid destroyed in dissolving the metals is equivalent to the amount of alkali used to precipitate., their hydroxides, so no error arises from

this source. However, acid is lost by reaction with primary minerals. Alkaline soils of high pH also destroy some acid through neutralization by sodium carbonate.

Apparatus :

- 1) Glass beaker 100 ml.
- 2) Conical glass flask 250 ml.
- 3) Glass funnel
- 4) Volumetric flask (100 ml)
- 5) Erlenmeyer flask (150 ml)

Reagents :

- 1) Hydrochloric acid (1N) : Dissolve 89 ml of conc. HCL in distilled water and make upto 1 lit.
- 2) Hydrochloric acid (0.2N) : Take 25 ml of 1N HCL and dilute it to 100 ml.
- 3) Sodium hydroxide (0.2N) : Dissolve 8.0 gm of sodium hydroxide in distilled water and make up the volume upto 1000 ml.
- 4) Potassium hydrogen phthalate (0.2N) : Dissolve 4.084 gm of KHP in distilled water and make up the volume upto 100 ml.

Procedure :

- 1) The preliminary tests are carried out on the soil to establish a rough idea of the amount. Of carbonate present from the degree of effervescence with dilute acid.
- 2) Based on the degree of effervescence the soil is taken for analysis as per the following table.

Degree of effervescence	Wt. of air dry soil (g) (W)
No effervescence	10.0 to 20.0
Moderate	5.0
Fairly vigorous	2.0
Vigorous to very vigorous	1.0

- 3) Transfer (W) g air dried soil of 0.5 mm sieve to a 250 ml plastic conical flask and carefully add 25 ml of 1N HCl down from the side of the flask. Cover with a watch glass and allow standing for 1 hour, swirling occasionally to mix the contents.
- 4) Then transfer the mixture quantitatively to a 100 ml volumetric flask and make up the volume with distilled water and mix. Filter through a dry filter paper into a dry flask.
- 5) Transfer 20 ml. of the clear liquid to a 150 ml Erlenmeyer flask. Add a little distilled water and bring just to the boil. Cool for a minute.
- 6) Add about 6 – 10 drops of bromothymol blue and titrate hot with 0.2N NaOH until the blue colour persists for 30 sec.
- 7) Rub the blank simultaneously (Blank is made by taking 25 ml 1N HCL in 100 ml volumetric flask and diluting with distilled water upto mark. 20 ml of this solution is taken as a blank for titration with 0.2N NaOH.)
- 8) Titrate 10 ml of 0.2N NaOH with 0.2N KHP using phenolphthalein and determine the factor of NaOH.

Observation Table :

Sr. No.	Lab. No.	Blank Reading	Burette (Sample) Reading	Difference	Std. HCl in ml.	% CaCO ₃

Calculations :

Let W be the moisture corrected weight of the soil in gm.

‘T’ is the volume in ml of the titration with 0.2 N sodium hydroxide.

Since 20 ml of liquid, after reaction with soil contains excess acid equivalent to “T” ml of 0.2N NaOH, so 100 ml contains T ml of 1N excess acid.

Thus, the acid neutralized in reaction with the soil is (Blank – T) ml of 1N.

Thus W gm soil contains 0.05 (Blank – T) gm calcium carbonate.

$$\text{CaCO}_3 \% \text{ in soil} = \frac{5 (\text{Blank} - T)}{W} \times F$$

Where,

F = factor of 0.2N NaOH and

W = wt. of air dry soil after correcting the moisture percentage.

Exact normality of NaOH = 0.2

This may be reported as an apparent CaCO_3 % or as a neutralizing value.

2. Schrotus Apparatus Method :

Apparatus

Glass Beaker, Conical Flask, Glass Funnel, Erlenmeyer Flask

Reagents

3 N Hydrochloric acid

Procedure

Pipette 10 ml of hydrochloric acid into 50 ml Erlenmeyer flask. Weigh the Erlenmeyer flask with cork stopper. Transfer 1 to 10 gm of soil to the flask. After effervescence has largely subsided make the stopper loose and swirl the flask. The reaction is usually complete in two hours. Displace the carbon dioxide in the flask with air and weigh the flask with stopper.

Calculations

Wt. of CO_2 lost = (Initial wt. of flask + acid + soil) – (Final wt. of flask + acid + soil)

Ratings :

	% CaCO_3	
1)	Less than 1	Low
2)	1 – 5	Medium
3)	5 – 10	High
4)	10 – 15	Very high

Reference Documents :

Dewis J. and Freitas F., 1970. calcium carbonate – Acid neutralization in Physical and Chemical Methods of Soil and Water Analysis. Food and Agricultural Organization of the United Nations, Rome, Italy, III.2-2, P. 71-72.

Diagnosis and improvement of saline & alkaline soils – United States Salinity Laboratory Staff, Agricultural Handbook No. 60, United State Dept. of Agriculture, P. 105.

5. DETERMINATION OF NITROGEN – (ALKALINE PERMANGNATE METHOD)

Purpose :

Soil nitrogen occurs largely in the organic form (97-99%). The availability of N is associated with the activity of micro-organisms which develops the organic matter ($\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$). The nitrification rate of a soil is measure of the rate of release of available nitrogen from the organic matter in the soil.

A discrete fraction of the soil organic nitrogen is attacked by KMnO_4 and that this fraction was most readily susceptible to biological mineralization. This forms the basis for determination of available nitrogen by alkaline permanganate method (Subbiah and Asija, 1856).

Principle :

The organic matter in the soil is oxidized by KMnO_4 in presence of NaOH . The ammonia released during oxidation is absorbed in boric acid to convert the ammonia to ammonium borate. The ammonium borate formed is titrated with standard H_2SO_4 . From the volume of standard H_2SO_4 required for the reaction with ammonium borate, the N is calculated.

Apparatus :

- 1) 100 ml conical flask
- 2) Funnels, filtration stands
- 3) 100 ml volumetric flask
- 4) Beaker
- 5) One lit. round bottom flask
- 6) Distillation unit (Kjeldhal Digestion Unit)

Reagents :

- 1) Potassium permanganate, KMnO_4 (0.32%) : Dissolve 3.2 g of potassium permanganate in 1 lit distilled water with the intermittent shaking till it is completely dissolved. Store in amber coloured bottle and in the dark
- 2) Sodium hydroxide solution, NaOH (92.5%) : Dissolve 25 g of pure sodium hydroxide pellets in one lit. distilled water.
- 3) Boric Acid H_3BO_3 (2%) : Dissolve 20 g boric acid of AR grade in 800 ml distilled water by heating the content. Cool it and dilute to 1000 ml volume.
- 4) Mixed indicator - Bromocresol green + Methyl red : Weigh out separately 99 mg of Bromocresol green and 66 mg of well powdered methyl red and dissolve them together in 100 ml ethyl alcohol.
- 5) Working Boric Acid solution : Add 20 ml of the mixed indicator to one lit. of 2% boric acid solution and adjust the pH to 5.0 after shaking, or add 0.1N NaOH continuously until the solution assumes reddish purple tinge red colour.
- 6) Standard sulphuric acid, H_2SO_4 (0.02N) : Standardise the H_2SO_4 solution using standard NaOH . NaOH be standardised against 0.02N $\text{H}_2\text{C}_2\text{O}_4$ or 0.02N potassium phthalate.
- 7) Liquid paraffin
- 8) Glass beads

Procedure :

- 1) Transfer 20 g of sieved soil into 1lit. round bottom flask.
- 2) Add little distilled water with the help of jet in such a way that the particles of soil do not remain stuck to the sides of the flask.

- 3) Add 2 to 3 glass beads to prevent bumping and 1 ml of liquid paraffin to prevent frothing.
- 4) Add 100 ml of potassium permanganate and 100 ml of sodium hydroxide solution to the flask (both the solutions should be prepared fresh).
- 5) Distill and collect the distillate in a beaker containing 20 ml of boric acid working solution.
- 6) Collect approximately 150 ml of distillate.
- 7) Titrate the distillate with standard H_2SO_4 0.02N till the colour changes from green to red and record the burette reading.
- 8) Carry out blank without soil.

Observations :

- | | | | |
|----|---|---|-----------------------|
| 1) | Weight of soil sample taken | = | 20 g |
| 2) | Volume of standard H_2SO_4 required for | = | ... A ml soil sample |
| 3) | Volume of standard H_2SO_4 required for | = | ... B ml blank sample |
| 4) | Normality of H_2SO_4 | = | 0.02N |

Calculations :

$$\text{N\%} = (A - B) \times \text{Normality of } \text{H}_2\text{SO}_4 \times \text{Equi. Wt. of N} \\ \times \frac{100 \text{ g}}{\text{of soil sample}} \times \frac{1}{\text{convert N mg into g}} \times 100 \text{ to}$$

$$\text{N\%} = (A - B) \times \text{Normality of } \text{H}_2\text{SO}_4 \text{ 0.02N} \times \text{Equi. Wt. of N (14)}$$

$$\times \frac{100 \text{ g}}{\text{sample g}} \times \text{Wt. of soil} \times \frac{1}{\text{convert N mg into g}} \times 100 \text{ to}$$

$$N\% = (A - B) \times \text{Normality of } H_2SO_4 \times 0.014$$

$$\times \frac{100 \text{ g}}{\text{Wt. of soil (g)}}$$

$$\text{Available N kg/ha} = N\% \times \frac{2240000}{100}$$

$$\text{Available N kg/ha} = (A - B) \times N \times 0.014 \times \frac{2240000}{\text{Wt. of soil sample g}}$$

N = Normality of H_2SO_4 . (See Appendix-2).

Ratings :

Nitrogen	Kg/ha
Very low	< 140
Low	140 – 280
Medium	281 – 420
Moderately High	421 – 560
High	562 – 700
Very High	> 701

Reference Documents :

- 1) Black, C.A. 1965, Methods of Soil Analysis, Part-2, American Society of Agronomy, INC. Soil Society of America, Madison, Wisconsin, USA.
- 2) Birch, H.F., 1960, Ibid, 12, 81.
- 3) Nelson, D.W. and J.M. Bremner, 1972. Preservation of soil samples for inorganic nitrogen analysis, Agron. J., 64 : 196 – 199.
- 4) Page, A.L. 1983 (Ed.), The Methods of Soil Analysis, Part-2, American Society of Agronomy, INC. Soil Society of America, Madison, Wisconsin, USA.
- 5) Perur, N.G., C.K. Subramaniam G.R., Muhr and H.E. ray, 1973, Soil Fertility Evaluation to serve Indian Farmers, Mysore Dept. of Agri., Mysore University of Agricultural Sciences, U.S. Agency for International Development, Bangalore, India.
- 6) Somwansi, R.B., B.D. Tamboli, V.M. Patil, D.B. Bhakare and P.P. Kadu, 1994, twenty Five years of research on soil test crop response correlation studies in Maharashtra. 1968 – 93, MPKV Res. Publ. No. 14.
- 7) Subbaiah, B.V. and G.L. Asija, 1956, A Rapid Procedure for the Estimation of Available Nitrogen in soils. Curr. Sci., 25 :259 – 260.

6. DETERMINATION OF PHOSPHOROUS IN SOIL :(OLSEN'S METHOD)

Purpose :

Next to nitrogen, phosphorous is most critical essential element in influencing plant growth & production throughout the world. Among the more significant functions & qualities of plants on which phosphorous has an important effect are –

- 1) Photosynthesis
- 2) Nitrogen fixation
- 3) Crop maturation – flowering and fruiting including seedformation
- 4) Root development
- 5) Protein synthesis

Thus, it is essential to calculate the available phosphorous present in the soil.

It is determined by Olsen's Method.

Principle :

Under neutral to alkaline soil conditions, Olsen's P (0.5 M NaHCO_3 solution at pH 8.5) is the most widely used extractant for estimation of available phosphorous in soil. The reagent is designated to control the ionic activity of calcium through the solubility product of CaCO_3 in case of neutral and calcareous soil. In this process the most effective form of "P" is extracted from the phosphates of Fe, Al & Ca present in different type of soils. The extracted phosphorous is measured calorimetrically.

Apparatus :

- 1) Conical flask – 150 ml.
- 2) Funnel
- 3) Pipettes
- 4) Volumetric flask – 25 ml.
- 5) Reagent bottles
- 6) Spectrophotometer

Reagents :

- 1) 0.5M NaHCO_3 – Dissolve 42.0 gm of P-free sodium bicarbonate in about 500 ml of hot distilled water and dilute to 1 litre. Adjust the pH to 8.5 using dilute NaOH or dilute HCL. Prepare fresh solution before use.
- 2) Activated Charcoal – wash pure activated charcoal or commercially available Darco G-60 with acid to make P-free, even if having traces of P.
- 3) Ammonium Paramolybdate $[(\text{NH}_4)_6 \text{MO}_7 \text{O}_{24} \cdot 4\text{H}_2\text{O}]$ – Dissolve 12.0 gm of ammonium paramolybdate in 250 ml of distilled water to get solution 'A'. Prepare solution 'B' by dissolving 0.2908 gm of potassium antimony tartarate ($\text{KsbO} \cdot \text{C}_4\text{H}_4\text{O}_6$) in 100 ml of distilled water. Prepare one litre of 5N H_2SO_4 (14 ml of concentrated H_2SO_4 diluted to 1 lit.) and add solutions "A" and "B" to it. Mix thoroughly and make the volume to 2 lit with distilled water. Store in amber coloured bottle in dark and cool compartment. (Reagent C).
- 4) Ascorbic Acid Solution – Dissolve 1.056 gm of ascorbic acid in 200 ml of molybdate tartarate solution (reagent C) and mix well. This ascorbic acid (reagent D) should be prepared as required because it does not keep more than 24 hrs.
- 5) P - nitrophenol indicator – Dissolve 0.5 gm of p-nitrophenol in 100 ml of distilled water to get approximately 5N H_2SO_4 .

- 6) Standard P Solution (Stock Solution) – Analytical grade (AR) KH_2PO_4 is dried in an oven at 60°C for one hour and after cooling in desiccator, weigh 0.4393 gm and dissolve in about 500 ml distilled water (shake the content until the salt dissolves.) Add 25 ml of approximately 7N H_2SO_4 & make the volume to 1 lit. Add 5 drops of toluene to diminish microbial activity. This gives 100 ppm stock solution of P (100 mg/ml).
- 7) P solution (5 ppm) – Pipette out 5 ml of stock solution of P and make up the volume to 1 lit with distilled water. This solution contains 5 mg P/ml (i.e. 5 ppm solution).
- 8) Hydrochloric Acid (0.02 N) – Dilute 1.8 ml of concentrated HCl to 1 lit.
- 9) Standardization of sodium hydroxide (NaOH) – Pipette out 10 ml of 0.02 potassium hydrogen phthalate in a 250 ml conical flask. Add 3 drops of phenolphthalein indicator. The end point is appearance of pale permanent pink colour.

Procedure :

- 1) Weight 2.5 gm of soil sample in 150 ml plastic conical flask, add pinch (0.3 gm) of phosphate free activated charcoal AR grade. Add 50 ml of Olsen reagent and shake for 20 minutes exactly on platform type shaker at 180 rpm.
- 2) Filter the contents immediately through filter paper. Transfer 5 ml of aliquot into 25 ml volumetric flask.
- 3) Pipette out 5 ml of filtrate into 25 ml volumetric flask. Add 4 ml of the freshly prepared ascorbic acid and ammonium molybdate solution. Shake well and keep it for 30 minutes then make the volume.

- 4) Prepare the standard curve using 0, 1, 2, 3, 4 & 5 ml of 5 ppm standard P solution into 25 ml volumetric flask and develop the colour using the same procedure as above. The corresponding P concentration will be 0, 0.2, 0.4, 0.6, 0.8 & 1 ppm.
- 5) Measure the absorbance and colour intensity at 882 nm after half an hour.
- 6) Run a blank method sample with the extracting solution.

Observation Table :

Sr. No.	Lab. No.	Reading on Spectrometer	P (ppm)	P (kg/ha.

Calculation :

$$P \text{ (ppm)} = \text{Where, } \frac{GR \times 50 \times 5}{\text{Corrected Ht. Of Soil}}$$

GR – Concentration of P in analysed sample. (read from std. curve)
 $P \text{ (kg/ha)} = P \text{ (ppm)} \times 2.24$ (See Appendix-7).

Rating –

Phosphorous (kg/ha)

1)	Very low	-	< 7
2)	Low	-	7 – 13
3)	Medium	-	13 – 22
4)	Moderately high	-	22 – 28
5)	High	-	28 – 35
6)	Very high	-	> 35

Reference Documents :

Dewis J and Freitas F, 1970, Ammonium fluoride – Hydrochloric acid and Extraction.

Olsen S. R. and Sommers L. E. 1982, Phosphorous in method of soil analysis, Part-2, Chemical and microbiological properties – Agronomy monograph number 9 (2nd Ed.)

American Society of Agronomy, Inc, Soil Science Society of America, Inc, Madison, Wisconsin, USA, 24 : 421 – 422.

7. DETERMINATION OF POTASSIUM ON FLAME PHOTOMETER :

Purpose :

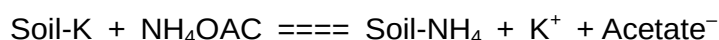
Next to nitrogen and phosphorous, potassium is the most critical essential element in influencing plant growth and production throughout the world. Potassium plays essential role in plants. It is an activator for dozens of enzymes responsible for plant process. Potassium is essential for photosynthesis, for protein synthesis, for starch formation and for translocation of sugars. Also it exerts a balancing effect on the effects of both nitrogen and phosphorous.

Thus, it is essential to calculate the available potassium present in soil. Purpose of potassium determination is to determine available potassium content in given soil.

Principle :

When a solution of the metallic salt is atomized into a non luminous flame, electrical K atoms get excited and emit light when come to ground state. The light emitted is filtered through a glass filter which allows light to definite wavelength of that element, 766.5 nm for K, to pass. The light falls on photocell emitting electrons generating an electric current. This current is measured on the galvanometer and is proportional to the concentration of metal element present in solution atomized.

Reaction :



Apparatus :

- 1) 100 ml Conical flask
- 2) Funnels, filtration stands
- 3) 100 ml volumetric flasks
- 4) Flame photometer
- 5) 50 ml volumetric flasks

Reagents :

- 1) Neutral normal ammonium acetate solution – Take 58 ml of glacial acetic acid in 500 ml volumetric flask. Also take 71 ml of concentrated ammonium hydroxide solution in another 500 ml volumetric flask. Dilute both the solutions with distilled water upto the 2/3 volume and mix both in 1 lit. flask then adjust pH to 7.0 and finally make up the volume to 1 lit. For bringing pH of solution to 7, add dilute acetic acid or ammonium hydroxide, or dissolve 77 gm/lit. NH_4OAC and adjust pH to 7 by acetic acid or ammonium hydroxide.
- 2) Standard potassium stock solution (100 $\mu\text{g K / ml}$) – Dissolve 1.908 gm chemically pure KCl in distilled water, make up the volume to 1 L. This solution contains 1000 $\mu\text{g / ml}$ of K. It serves as standard stock solution. Also prepare secondary stock solution of 100 $\mu\text{g K / ml}$ from this primary stock solution by taking 10 ml and making 100 ml volume.
- 3) Working solution – Pipette 0, 0.5, 1, 2, 4, 6, 8 and 10 ml of 100 $\mu\text{g K / ml}$ solution in 100 ml volume flask separately and make up the volume with NH_4OAC solution. This gives 0, 0.5, 1, 2, 4, 6, 8 and 10 $\mu\text{g / ml}$ respectively.

Observation Table :

Sr. No.	Lab. Sample No.	Reading on flame photometer for potassium	Potassium in ppm

Precautions while handling flame photometer :

- 1) All the necessary safety precautions meeting the appropriate specifications for use of LPG burner should be strictly followed.
- 2) Never view the flame from the top of the chimney. It should be viewed from the round viewing indicator provided on the front side.
- 3) Ensure air is flowing through the burner before LPG is allowed in to the burner and lighting it.
- 4) Always start the air compressor first and then the LPG gas to avoid inadequate air and gas accumulation.
- 5) While switching off, turn off the LPG gas supply first. After the flame goes off, switch off the compressor.
- 6) Inadequate air and more fuel would result in accumulation of fuel gas, which will cause flame to appear above the chimney and burn the chamber.
- 7) Ensure that all the end clamps are tight and that there is no leakage. The leakage can be checked using soap solution at the nozzle end.

Procedure :

- 1) Add 25 ml of NH_4OAc extracting solution to a conical flask containing 5 gm air dry soil sample.
- 2) Shake on a reciprocating shaker at 200 to 220 oscillations per minute for 5 min and filter.
- 3) Determine potassium as indicated in preparation of standard curve, dilute if necessary.

Calculation :

$$K \text{ (ppm)} = \text{Reading from graph } \mu\text{g K / ml in extract (R)} \times 5 \times \text{Dilution Factor (Df)}$$

$$K \text{ (kg/ha)} = \begin{array}{c} \text{Reading from graph } \mu\text{g} \\ \text{K / ml in extract (R)} \end{array} \times \frac{\text{Aliquot used 25 ml}}{\text{Soil used 5 g}} \times \frac{1 \text{ Kg soil 1000 g}}{1} \times \frac{1}{1000 \text{ to convert } \mu\text{g into mg}} \times \frac{1}{1000 \text{ to convert mg to g}} \times \frac{1}{\text{convert gm into kg}} \times 1000 \text{ to} \\ \times \frac{\text{Wt. of soil 2240000 kg/ha}}{1} \times \text{Dilution Factor (Df)}$$

$$K \text{ (kg/ha)} = R \times (25/5) \times (1000/1) \times (1/1000) \times (1/1000) \times (1/1000) \times (2240000/1)$$

$$= R \times 5 \times 2.24 \times \text{Dilution Factor (Df)} \quad K_2O^+ \text{ (kg/ha)} = R \times$$

$$11.20 \times 1.2$$

To convert K to K_2O multiplied by 1.2 and to convert K_2O to K multiply by 0.83.

Ratings :

Potassium (K) – kg/ha

Very low	Less than 120
Low	121 – 180
Moderate	181 – 240
Moderately high	241 – 300
High	301 – 360
Very high	Above 360

Reference Documents :

Ghosh A. B. and Bajaj J.C., Finanul Hasan and Dhyan Singh, 1983, Soil and water testing methods, Laboratory Manual, P. 21-22 (ICAR Publication)

Knudsen D and G. A. Peterson, 1982, Lithium, Sodium and Potassium, P. 225 to 246, In A.L. Page (Ed.) Methods of Soil Analysis. Agronomy Monogram No. 9, American Society of Agronomy, Inc. Soil Sci. Soc. Of America Inc. Publisher Madison WB Consin, USA.

Somawanshi R. B., Tamboli B.D., Patil Y. M. and Kadu P. P., 1994, twenty Five years of Research on soil test Crop Response Studies in Maharashtra. 1968 – 1993, M.P.K.V. Res. Publ. No. 14.

8. DETERMINATION OF SODIUM ON FLAME PHOTOMETER :

Purpose :

Sodium affects the permeability of soil by causing swelling and dispersion of clay particles and clogging the soil pores. It may also cause injury to crops specifically sensitive to sodium such as fruit crops. An alkali soil also called sodic soil contains sufficient exchangeable sodium to cause soil dispersion and increase the pH thereby adversely affecting both the physical and nutritional properties of the soil with consequent reduction in crop growth significantly or entirely.

Principle :

Available sodium includes exchangeable sodium and water soluble sodium. Exchangeable sodium in soils varies from trace amounts to a large portion of the exchange capacity depending on the soil environment. Likewise water soluble sodium varies dramatically depending on soil salinity level. Even though soils may have high sodium saturations, the actual quantity of exchangeable sodium compared with the total soil sodium content is generally small. The exchangeable sodium in soils is determined by extracting with neutral 1.0N ammonium acetate and then subtracting the water soluble sodium. The correction for water soluble sodium is negligible in many soils of the humid regions but is often important in the soils of dry regions. The sodium is estimated by flame photometer.

Apparatus :

- 1) Flame Photometer
- 2) Beaker – 250 ml, 100 ml.
- 3) Volumetric measuring flask – 50 ml capacity
- 4) Measuring flask – 1 lit capacity.

Reagents :

- 1) 1 N Ammonium Acetate - Dissolve 77.08 gm of ammonium acetate in distilled water and make the volume to 1 lit. Adjust the pH to 7.0 with glacial acetic acid or ammonia solution.
- 2) Standard Sodium Solution –
 - i) Dissolve 2.542 gm of dried NaCl (AR at 110°C for 1 hr) in distilled water and make the volume to 1 lit. i.e. 1000 ppm Na solution. 10 ml of 1000 ppm solution was diluted to 100 ml. The concentration of the sodium is 100 ppm.
 - ii) Take 2, 4, 6, 8 and 10 ml of 100 ppm Na solution in separate 100 ml volumetric flask and make up volume with distilled water. Thus 2, 4, 6, 8 and 10 ppm Na solutions are maintained and readings are taken on the flame photometer.

Precautions while handling flame photometer :

- 1) All the necessary safety precautions meeting the appropriate specifications for use of LPG burner should be strictly followed.
- 2) Never view the flame from the top of the chimney. It should be viewed from the round viewing indicator provided on the front side.
- 3) Ensure air is flowing through the burner before LPG is allowed in to the burner and lighting it.
- 4) Always start the air compressor first and then the LPG gas to avoid inadequate air and gas accumulation.
- 5) While switching off, turn off the LPG gas supply first. After the flame goes off, switch off the compressor.

- 6) Inadequate air and more fuel would result in accumulation of fuel gas, which will cause flame to appear above the chimney and burn the chamber.
- 7) Ensure that all the end clamps are tight and that there is no leakage. The leakage can be checked using soap solution at the nozzle end.

Procedure :

- 1) Weigh 5 g of 2 mm sieved soil sample in 250 ml plastic conical flask.
- 2) Add 25 ml of the neutral 1N ammonium acetate solution and shake for 30 minutes on mechanical shaker at 110 rpm. Filter through whatman No. 1 filter paper.
- 3) Take the readings on flame photometer.
- 4) Feed the working standard solution and prepare a standard curve.
- 5) If the sample reading is not found within the standard reading range in that case the appropriate dilution of the filtrate may be made to bring the reading within standard range.

Observation Table :

Sr. No.	Lab. Sample No.	Reading on flame photometer for sodium	Sodium in ppm

Calculation :

$$\begin{aligned}
 \text{Na (ppm)} &= \frac{\text{GR} \times \text{Vol. of extractant}}{\text{Corrected wt. of the soil}} \times \text{Dilution factor (Df)} \\
 &= \frac{20}{20} \frac{\text{GR} \times 100}{100} \times \text{Dilution factor (Df)} \\
 &= \text{GR} \times 5 \times \text{Dilution factor (Df)}
 \end{aligned}$$

Where GR stands for concentration of Na obtained from the flamephotometer

ESP and SAR is calculated on the basis of sodium and Ca + Mg values. $\text{SAR} = \text{Na}^+ /$

$\sqrt{(\text{Ca}^{++} + \text{Mg}^{++})/2}$ Na^+ , Ca^{++} , Mg^{++} in meq/lit

$$\text{ESP} = 100(-0.0126 + 0.01475\text{SAR}) / (1 - (-0.0126 + 0.01475\text{SAR}))$$

Rating :

Name of Soil	Sodium Adsorption Ratio (SAR)	Exchangeable Sodium Percentage (ESP)
Saline Soils	< 13	< 15
Alkali or Sodic Soils	> 13	> 15
Saline Alkali Soils	> 13	> 15
Degraded Alkali Soils	> 13	> 15

Salinity & Alkalinity Appraisal

Type of Soil	pH	EC dS/m	ESP %
Saline	< 8.5	> 4	< 15
Saline Alkali	> 8.5	> 4	> 15
Alkali	> 8.5	< 4	> 15

Reference Documents :

Knudsen D., Peterson G. A. and Pratt P. F. 1982, Lithium, sodium and potassium in methods of soil analysis – Chemical and microbiological properties, 2nd edition of American Society of Agronomy, Inc and Soil Science Society of America Inc., Madison, Wisconsin, USA, Number 9 (Part-2), 13:238-241.

9. DETERMINATION OF CALCIUM AND MAGNESIUM : (E.D.T.A. TITRIMETRIC METHOD BY EL MAHI, et.al. (1987))

Purpose :

Calcium and Magnesium ions serve as plant nutrients in cation exchange capacity of soils. Calcium and magnesium form the predominant exchangeable base, constituting 60 to 80% of total exchangeable cations.

Calcium clay and magnesium clay possess excellent physical conditions. It develops good crumb structure by virtue of the flocculation and aggregation of primary particles. This allows free movement of water without stagnation and contains sufficient air for the proper aeration of plant roots. Such a soil is highly productive; it supplies necessary plant nutrients.

Principle :

The method is based on the fact that Ca, Mg and number of other ions form stable complexes with EDTA (Ethylenediamine tetra acetic acid disodium salt) at different pH and Sn, Cu, Mn, Zn may interfere in the determination of calcium and magnesium if present in appropriate amounts. Their interference is prevented by use of 2% NaCN solution.

Apparatus :

- 1) 1 litre flask
- 2) Porcelain dishes 3" to 4" diameter
- 3) Burette of 50 ml capacity
- 4) Pipette of 5 ml and 10 ml.
- 5) Beaker of 100 ml.
- 6) Centrifuge tubes / polythene shaking bottle.

Reagents :

- 1) Standard Ca solution, 0.01 N : Weigh 0.05 gm CaCO_3 and dissolve in 10 ml of 2N HCl. Heat till the solution boils and CO_2 is completely driven off. Cool and make the volume accurately to 1 litre.
- 2) EDTA Solution : Dissolve 2 gm disodium EDTA in distilled water and make the volume 1 litre. Standardize against standard Ca Solution.
- 3) $\text{NH}_4\text{Cl} - \text{NH}_4\text{OH}$ buffer solution : Dissolve 67.5 gm NH_4Cl in 570 ml of concentrated NH_4OH solution and make 1 litre volume.
- 4) NaOH 10% : Add 10 gm NaOH to 90 ml distilled water.
- 5) $\text{NH}_2\text{OH} \cdot \text{HCl}$: Dissolve 5 gm hydroxylamine hydrochloride in 100 ml of distilled water.
- 6) $\text{K}_4\text{Fe}(\text{CN})_6$: Dissolve 4 gm potassium ferrocyanide in 100 ml of distilled water.
- 7) Triethanolamine : TEA.
- 8) EBT : Dissolve 0.2 gm Erichrome black T in 50 ml of methanol.
- 9) Calcon indicator : Dissolve 20 mg calcon in 50 ml methanol.
- 10) Neutral normal ammonium acetate solution – Take 58 ml of glacial acetic acid in 500 ml volumetric flask. Also take 71 ml of concentrated ammonium hydroxide solution in another 500 ml volumetric flask. Dilute both the solutions with distilled water upto the 2/3 volume and mix both in 1 lit. flask then adjust pH to 7.0 and finally make up the volume to 1 lit. For bringing pH of solution to 7, add dilute acetic acid or ammonium hydroxide, or dissolve 77 gm/lit. NH_4OAc and adjust pH to 7 by acetic acid or ammonium hydroxide.

Procedure :**Extraction :**

- 1) Weigh 2 – 4 gm of soil sample (2mm sieved) in conical flask or polythene shaking bottle or 100 ml centrifuge tubes.
- 2) Add 30 ml of NH_4OAc and shake for 5 min and decant.
- 3) Then add 30 ml of 0.5N HCl to each sample and agitate the contents, shake for 5 min. in a upright loosened position.
- 4) Then filter the solution using whatman No. 1 filter paper. Collect the filtrate.

Estimation of Ca & Mg (determination of ca & Mg together) :

- 1) Pipette out 20 ml of the filtrate into a 150 ml conical flask.
- 2) Add 50 ml distilled water.
- 3) Add 10-15 ml $\text{NH}_4\text{Cl} - \text{NH}_4\text{OH}$ buffer solution and add 10 drops each of $\text{NH}_2\text{OH.HCL}$, $\text{K}_4\text{Fe(CN)}_6$, TEA and EBT indicator.
- 4) Titrate with standard EDTA to permanent blue colour.

Determination of Calcium alone :

- 1) Pipette out 5 or 10 ml extract, add 10 drops each of $\text{NH}_2\text{OH.HCL}$, $\text{K}_4\text{Fe(CN)}_6$ and TEA and enough of 10% NaOH to raise pH to 12.
- 2) Add 5 drops of calcon indicator.
- 3) Titrate against standard EDTA. The end point is the change of colour from red to blue.

The value obtained from Ca plus Mg and Ca alone are used to calculate the Ca and Mg in the soil samples respectively.

Observation Table :

A) For Ca determination :

Sr. No.	Lab. Sample No.	Burette reading	Ca meq/lit.

B) For Ca + Mg determination :

Sr. No.	Lab. Sample No.	Burette reading	Ca + Mg meq/lit.

Calculations :

Meq (Ca + Mg) or Ca/100 gm

$$= \frac{\text{ml. of EDTA required} \times \text{Normality of EDTA (0.01)} \times \text{vol. made 100 ml} \times 100}{\text{titration (ml)} \times \text{Weight of soil in gm.}}$$

$$\text{Meq (Mg)} = \text{Meq (Ca + Mg)} - \text{Meq (Ca)}$$

Reference Documents :

Devis J. and Freitas, 1970, Calcium plus Magnesium and Calcium in Physical and Chemical Methods of Soil & Water Analysis, Food and Agriculture Organization of the United Nations, Rome, Italy, Soil Bulletin – 10 : 212-217.

Nelson R.E., 1982, Carbonate and Gypsum, In A. L. Page (Ed.). Methods of Soil Analysis, Part-2. Agronomy 9 : 181-197. American Society of Agronomy Ins. Soil Science Society of America Inc., Madison Wisconsin.

Somwanshi et.al., (1999), Analysis of Plants, Irrigation Water and Soils, MPKV, Rahuri, P. 225-228.

10. DETERMINATION OF CATION EXCHANGE CAPACITY IN SOIL

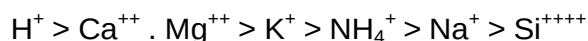
Purpose :

The Cation Exchange Capacity (CEC) is the capacity of soil to hold an exchangeable cations. The higher the CEC of soil, the more cations it can retain. Soil differ in their capacities to hold exchangeable K^+ and other cations. The cations exchange capacity depends on amount and kind of clay and organic matter present. High clay soil can hold more exchangeable cations than a low clay soils. CEC also increases as organic matter increases. Clay mineral usually range from 10 to 150 meq./100g. In CEC values organic matter ranges from 200 to 400 meq/100g. So the kind and amount of clay an organic matter content greatly influence the CEC of soil. Cation exchange is an important reaction in soil fertility in causing and correcting soil acidity and basisity.

1. Ammonium Saturation Method :Principle :

1. The cation exchange capacity (CEC) determination involves measuring of total quantity of negative charges per unit weight of the soil which are neutralize by the exchangeable cations. It is defined as the capacity of the soils to adsorb the sum total of exchangeable cations expressed as milliequivalents per 100.0 gm of soil or C mol (P^+)/kg of soil on oven dry basis.

Clay particles and organic matter carry negative charges over their surface due to which they adsorb positively charged particles (cations). There are different types of cations e.g. H^+ , Ca^{++} , K^+ , NH_4^+ , Mg^{++} etc. These cations can replace each other depending upon their concentration (mass action) and replacing power. The replacement of cations by one another is known as cation exchange. The order of replacing power of some cations are given below –



2. The CEC of different colloids are as follows –

Inorganic Colloid (average 8 to 100 me/100 gm soil)

Kaolinite	3 to 15 me/100 gm soil
Halloysite 2H ₂ O	5 to 10 me/100 gm soil
Illite	10 to 40 me/100 gm soil
Chlorite	10 to 40 me/100 gm soil
Sepionite, Attapulgite, Polygorstite	20 to 40 me/100 gm soil
Halloysite 4H ₂ O	40 to 50 me/100 gm soil
Montmorillonite	80 to 150 me/100 gm soil
Vermiculite	100 to 150 me/100 gm soil
Organic colloid :	
Humus	150-300 me/100 of material

3. Soil is treated with 1(N) ammonium acetate at pH 7.0 to saturate colloidal complex and the excess salt is removed with methanol / ethanol (60%). Then the ammonium ion is displaced with potassium by titrating with 10% potassium chloride at pH 2.5 and finally the ammonia is measured by distillation from alkaline solution, absorption in hydrochloric acid and titration with standard acid.

Reagents :

- 1) Ammonium Acetate, 1N, pH 7.0 + 0.1 : Dissolve 77.08 gm of ammonium acetate in distilled water and makeup the volume to one litre. Adjust the pH at 7 with ammonia or acetic acid.
- 2) Potassium Chloride (10%, pH 2.5 + 0.1) : Dissolve 10 gm of potassium chloride in distilled water and make up the volume to 100 ml. Adjust the pH at 2.5 with 1N HCl.

- 3) Methanol or ethanol, 60% : Dilute 60 ml methanol with distilled water and make up the volume to 100 ml.
- 4) Sodium hydroxide, 40% : Dissolve 400 gm NaOH in water in a 1 litre volumetric flask. Let it cooled and make it upto the mark.
- 5) Methyl red : Dissolve 0.1 gm methyl red in 100 ml ethanol (98%).
- 6) Hydrochloric acid (0.02N) : Take 1.8 ml of concentrated HCl in one litre volumetric flask and make the volume with distilled water.

Procedure :

- 1) Weigh 10.0 gm of 5 mm sieved air dried soil sample into 500 ml plastic conical flask. Add 250 ml of neutral ammonium acetate solution.
- 2) Shake the contents on mechanical shaker at 110 rpm for an hour and keep it over night. Next day again shake it for 1 hour.
- 3) Filter the contents through Whatman No. 1 filter paper receiving the filtrate in a 250 ml volumetric flask.
- 4) Transfer the soil completely on the filter paper and continue to leach the soil with the neutral solution (using 20-25 ml at a time).
- 5) Allowing the leachate to drain completely before fresh aliquots added. If it is not enough add some ammonium acetate to make up the mark. The solution is now ready for determination of individual cations.

- 6) The residue left on the filter paper is intended for the determination of cation exchange capacity of soil. Wash the leached soil with 60% methanol or ethanol to remove the excess ammonia. Each time 10 ml of alcohol in interval is added, draining between each addition is necessary. Nessler's reagent does the test of ammonia. If Nessler's reagent is not available the test of ammonia is done by HCl.
- 7) Collect the leachable washing of ethanol / methanol in watch glass and add few drops of concentrated HCl if it fumes that indicates presence of ammonia otherwise not. After washing, the soil is leached with acidified potassium chloride (10%, pH = 2.5 ± 0.1) and the extract is collected finally in a 250 ml volumetric flask.
- 8) 25–30 ml of KCl solution is added. Each time draining between each addition is necessary. The volume is adjusted to the mark by the replacing solution of KCl. It is shaken to make the concentration solution homogeneous.
- 9) Transfer 20 ml of KCl leachate and 10 ml of distilled water in a Gerhardt tube, add 10 ml of 40% NaOH and distilled into 20 ml of HCl (0.02N) containing 6 drops of methyl red indicator. The approximate distillate is collected about 150 – 200 ml. Titrate the solution until the yellow colour appears with 0.02N NaOH.
- 10) The changes from red to yellow, the end point being taken at the first appearance of the yellow colour. 20 ml of 10% KCl solution is distilled in the same manner for blank.

Calculations :

Let D = The weight of oven dry soil in the weight of air dry soil taken for analysis.

V = The total volume of the final solution containing the ammonium ion.

T = The volume of standard acid (0.02N) used for titrating the ammonia nitrogen after correction for the blank.

N = Normality of standard acid 0.02N
If 20 ml of ammonium solution is distilled,

$$V \text{ ml contains } \frac{0.01 T V}{20 D} \text{ milliequivalents} = \frac{1 T V}{2000}$$

$$\text{Thus, Cation exchange capacity} = \frac{NTV \times 100}{20 D} \text{ meq/100 gm of soil.}$$

If 10 ml ammonium solution is distilled,

$$\text{Cation exchange capacity} = \frac{NTV \times 100}{10 D} \text{ meq/100 gm of soil.}$$

Notes :

- The ammonium acetate method is suitable for neutral and non-calcareous soils, since displacement of hydrogen ions from acid soils may be incomplete. The extraction with ammonium acetate is also a reliable method for determination of CEC of acid soils if it is combined with another method for measurement of exchange acidity. The exchangeable calcium, magnesium, sodium, potassium and manganese are determined in the ammonium acetate extract and exchange acidity (exchangeable hydrogen plus exchangeable aluminium), is determined with 1N KCl or barium chloride triethanolamine. The sum of all the exchangeable cations then gives a good estimate of CEC.

- To ensure that the saturating cation displaces all the exchangeable cations, it is advisable to use on an average 25 parts of solution to 1 part of soil. Sandy soils low in exchange capacity may be extracted satisfactorily at a ration less than this, but on the other hand clay soils particularly organic ones, may need to be treated at a ratio of 40:1 or 50:1 for accurate results. Similarly consideration apply to the replacement process.
- For heavy clay soils it is better to take 5.0 gm of soil for analysis.
- After washing do not allow the soil to dry out as this may cause less of absorbed ammonia.
- Take the soil sample for analysis after sieving with 0.5 mm sieve.
- The Nessler's reagent is prepared by dissolving 45.5 gm of mercuric iodide and 35.0 gm of potassium iodide (KI) in a few ml. of water.



- The solution is transferred into a 1 litre volumetric flask. Then 112.0 gm of KOH is added and the volume is brought to about 800 ml. The solution is mixed well, cooled and diluted to 1 litre with water. The solution is allowed to stand for few days and the clear supernant liquid (Nesslers' reagent) is decanted off into amber coloured bottle for use.

2 . Sodium Saturation Method (Bower et. al.) :

Reagents :

- 1) 1 N Sodium Acetate - Dissolve 136 gm of sodium acetate in distilled water and transfer solution to 1 litre volumetric flask. Make the volume to 1 litre with distilled water. Add few drops of acetic acid or few pellets of NaOH to adjust the pH to 8.2
- 2) 1 N Ammonium Acetate – Dissolve 77 gm of ammonium acetate in distilled water and make the volume to 1 litre in volumetric flask. Adjust the pH to 7.0
- 3) 99% Isopropyl Alcohol
- 4) 1000 ppm Na Solution – Transfer 2.541 gm of pure dry NaCl equivalent to 1 gm Na in to 1 litre volumetric flask. Add distilled water and mix the contents by shaking
- 5) 250 ppm Na Solution – Transfer 25 ml of 1000 ppm Na solution in to 100 ml flask and dilute the solution with distilled water up to 100 ml mark.
- 6) 0 to 20 ppm Na Solution – Transfer 0,2,4,6,8 ml of 250 ppm Na solution in separate 100 ml volumetric flasks. Add 1 N ammonium acetate (pH 7.0) to each flask to make the volume to 100 ml mark for obtaining 0,5,10,15 and 20 ppm Na solution. Add few drops of butyl alcohol in each flask to improve spraying property of the solution.

Procedure :

- 1) **Preparation of Standard Curve for Sodium** – Prepare standard curve as per procedure given in test for sodium
- 2) **Preparation of Soil Extract for CEC Determination** – Weigh 6 gm of coarse textured or 4.5 gm of medium or fine textured soil passing through 2 mm sieve in to 50 ml centrifuge tubes. Add 33 ml of 1 N

sodium acetate (pH 8.2) and triturate with a glass rod for 5 minutes and centrifuge the tubes at 2000 to 2440 rpm for 10 minutes or till supernatant liquid becomes clear, placing the tubes opposite to each other. Decant supernatant liquid. Repeat the procedure twice to complete the process of saturation of the exchange complex with sodium. Wash the soil saturated with sodium in identical manner with three 33 ml 99% isopropyl alcohol washings. This will remove soluble sodium acetate in soil without causing any hydrolysis of exchangeable sodium.

- 3) Determine the concentration of sodium in CEC extract as per procedure given for determination of sodium.

Calculations :

Meq of Na / litre in CEC extract = ppm Na in CEC undiluted extract / Eq. Wt. of Na
Meq of Na / 100 gm of soil

= Meq of Na / litre in CEC extract x 100

$$\frac{10 \times \text{Soil in gm taken for preparation of CEC extract}}{\text{CEC of Soil}} = \text{meq of Na / 100 gm soil}$$

Ratings :

Result	Low	Medium	High	Very High
CEC meq/100 g or C mole (P ⁺)/kg.	Less than 10	10 - 25	25 - 45	More than 45

Reference Documents :

Devis J. and Freitas, 1970, Cation Exchange Capacity, Physical and Chemical Methods of Soil & Water Analysis, Food and Agriculture Organization of the United Nations, Rome, Italy, Soil Bulletin – 10 : 94-130.

P. C. Jaiswal, 2003, Soil, Plant and Water Analysis, Kalyani Publishers, p 123-126

Soil Testing Manual of B.Sc.(agri.) 2007, Marathwada Agricultural University, Parbhani, P. 33.

Notes on Soil Science, DIRD, Pune, 2008, P. 196.

C. RECLAMATION OF PROBLEMATIC SOIL

1. DETERMINATION OF GYPSUM REQUIREMENT OF SOIL Principle :

Gypsum requirement of alkali soils can be determined by treating the soil with known amount of excess saturated gypsum solution, and then estimating the unreacted or unutilized amount by versenate titration method as suggested by Schoonover (1952). Though, Ca can be estimated by other methods also but the versene titration is more suitable.

Instrument : Mechanical shaker.

Reagents :

Saturated gypsum solution : Add 5 g of chemically pure $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ to one litre of distilled water. Shake vigorously for 10 min. using a mechanical shaker and filter through Whatman No. 1 paper.

0.01N CaCl_2 solution : Dissolve exactly 0.5 g of AR grade CaCO_3 powder in about 10 ml of 1:3 diluted HCl. When completely dissolved, transfer to 1 litre volumetric flask and dilute to the mark with distilled water. CaCl_2 salt being highly hygroscopic should not be used.

0.01N Versenate solution : Dissolve 2.0 g of pure EDTA-disodium salt and 0.05 g of magnesium chloride (AR) in about 50 ml of water and dilute to 1 litre. Titrate a portion of this against 0.01N CaCl_2 solution to standardize.

Erichrome black T indicator : Dissolve 0.5 g of EBT dye and 4.5 g of hydroxylamine hydrochloride in 100 ml of 95% ethanol. Store in a stoppered bottle or flask.

Ammonium hydroxide - ammonium chloride buffer : Dissolve

67.5 g of pure ammonium chloride in 570 ml of conc. ammonia solution and dilute to 1 litre. Adjust the pH at 10 using dil. HCl or dil. NH_4OH .

Procedure :

1. Weigh 5 g of air dry soil in 250 ml conical flask.
2. Add 100 ml of the saturated gypsum solution. Firmly put a rubber stopper and shake for 5 minutes.
3. Filter the contents through Whatman No. 1 filter paper. Entire quantity needs to be filtered.
4. Transfer 5 ml aliquot of the clear filtrate into a 100 or 150 ml porcelain dish.
5. Add 1 ml of the ammonium hydroxide – ammonium chloride buffer solution and 2 to 3 drops of Erichrome black T indicator.
6. Take 0.01N versenate solution in a 50 ml burette and titrate the contents in the dish until the wine red colour starts changing to sky blue.
7. Run a blank using 5 ml of the saturated gypsum solution in place of sample aliquot.

Calculation :

$\text{Ca or Ca+Mg (me/L) in the aliquot} = 2 V$

Where, V stands for volume of versenate solution used. Since, 1 litre extract

= 50 g soil (5 g soil to 100 ml)

Ca retained (or Ca requirement) in me/100 g soil

$$= [2V \text{ for added gypsum solution} - 2V \text{ for filtrate}] \times 2 \dots\dots (A)$$

Gypsum requirement of soil in tons per hectare upto 30 cm soil depth

$$= A \times 3.852$$

Apply correlation depending on purity of gypsum.

Reference Documents :

Singh, Dhyan, Chhonkar, P.K. and Pande R.N., 1999, Assessment of Irrigation Water Quality in "Soil, Plant, Water Analysis" - A methods manual, Indian Agricultural Research Institute, Indian Council of Agricultural Research, New Delhi, 3 : 48-50.

D. WATER SAMPLE ANALYSIS

1. DETERMINATION OF pH

Principle :

The determination of pH of water sample is based on the same principle as mentioned in section of pH determination of soil.

- Apparatus :**
- i) pH meter with glass electrodes
 - iii) Thermometer

Reagents :

Standard buffer solution – Dissolve one commercially available buffer tablet each of pH 4.0, 7.0 and 9.2 in freshly prepared distilled water separately and make up the volume to 100 ml. Prepare the fresh solution every week as these solutions do not keep for long. Three to four drops of toluene are added in standard buffer solutions to prevent growth of mould.

Procedure :

- 1) Turn the pH meter ON and allow it to warm for 15 minutes.
- 2) Standardize the glass electrode using standard buffer of pH 7.0 and calibrate with the buffer pH = 4 or pH = 9.2.
- 3) Take 50 ml of filtered water sample in 100 ml beaker and immerse the glass and calomel electrodes or combined electrode of the pH meter. Never allow the lower portion of glass electrodes to touch the bottom of the beaker.
- 4) While recording pH, switch the pH meter to pH reading, wait for 30 seconds and record the pH value to the nearest 0.1 unit. Put the pH meter in stand by mode immediately after recording.
- 5) Remove the electrodes after each determination and carefully blot them dry with filter paper before the next determination. Standardize the glass electrodes after every ten determinations.

- 6) Keep the electrodes in distilled water, when not in use and ensure that the reference electrode always contains saturated potassium chloride solution in contact with solid potassium chloride crystals.

Based on pH values, neutral water can be divided into 3 classes –

- 1) Those, which contain carbonates, with or without bicarbonates, do not have free carbonic acids. The pH value of these waters are always above 8.0.
- 2) Those, which contain no carbonates but contain bicarbonates and carbonic acid, the pH values of these waters range from 4.5 to 8.0. Most of neutral waters fall under this category. The pH value of neutral water usually lie between 6.5 and 7.5.
- 3) Those which contain free acid in addition to carbonic acid, do not contain carbonates or bicarbonates. The pH value of these water is 4.5 or below 4.5.

Reference Documents :

Devis J. and Freitas, 1970, Analysis of Water and Water Extracts, Physical and Chemical Methods of Soil & Water Analysis, Food and Agriculture Organization of the United Nations, Rome, Italy, Soil Bulletin – 10 : 208.

Singh, Dhyan, Chhonkar, P.K. and Pande R.N., 1999, Assessment of Irrigation Water Quality in “Soil, Plant, Water Analysis” - A methods manual, Indian Agricultural Research Institute, Indian Council of Agricultural Research, New Delhi, 3 : 73-74.

2. DETERMINATION OF ELECTRICAL CONDUCTIVITY (EC) : Principle :

The determination of electrical conductivity of water sample is based on the same principle as mentioned in section under measurement of electrical conductivity in soil.

Apparatus :

- i) EC meter
- ii) Thermometer

Reagent :

0.01N Potassium chloride solution : Dry a small quantity of A.R. grade Potassium chloride at 60°C for 2 hours. Weigh 0.7456 gm of it and dissolve in freshly prepared distilled water and make to one litre. This solution gives an electrical conductivity of 1411.8×10^{-3} i.e. 1.41 dS/m at 25°C.

Procedure :

- 1) Calibrate the conductivity cell with the help of standard KCL solution and determine the cell constant.
- 2) Dip the conductivity cell assembly in water sample taken in a 50 or 100 ml beaker and record the conductivity. If the value is too low, change the adjustment accordingly. Record the temperature of water during the test.
- 3) Observed values of EC are multiplied by the cell constant (usually given on conductivity cell) and a temperature factor to express results at 25°C.
- 4) Remove the cell from soil suspension, clean with distilled water and dip into a beaker of distilled water. EC is expressed as dS/m.
- 5) Keep the conductivity cell in distilled water when not in use.

Calculations :

The cell constant K is given by

$$\text{Cell constant (K)} = \frac{\text{Known conductivity of 0.01 N KCL}}{\text{Conductivity of 0.01 N KCL measured}}$$

$$EC_{w25} = EC_T \times K \times ft$$

Where,

EC_{w25} is the conductivity of the water at 25°C.

EC_T is apparent conductivity of water as measured. K is the cell constant

Ft is temperature correction factor. (See Annexure No. 9).

- The EC values can either be used as such categorizing the water on salinity basis or may be used to get the concentration as given below –
- Salt concentration (mg/L) = EC in $dS.m^{-1}$ at 25°C $\times$ 640
- Salt concentration in me/L (approx) = EC in $dS.m^{-1}$ at 25°C $\times$ 10

Note :

- Even if the scale is marked to read directly, it is necessary to check /calibrate the instrument with the standard KCl solution.
- Now a day, the temperature correction factor is provided in the instrument itself. Measure the temperature of the solution.

- Depending on EC_w values, irrigation waters are classified in following classes –

Class	Low salinity C1	Medium salinity C2	High salinity C3	Very high salinity C4
EC_w in $dS.m^{-1}$	< 0.25	0.25 to 0.75	0.75 to 2.25	> 2.25

Reference Documents :

Singh, Dhyan, Chhonkar, P.K. and Pande R.N., 1999, Assessment of Irrigation Water Quality in “Soil, Plant, Water Analysis” - A methods manual, Indian Agricultural Research Institute, Indian Council of Agricultural Research, New Delhi, 3 : 74-75.

Diagnosis and improvement of saline & alkali soils – United States Salinity Laboratory Staff, Agricultural Handbook No. 60, United States of Agriculture, P. 79-81.

3. DETERMINATION OF CARBONATES AND BICARBONATES Principle :

Carbonate and bicarbonate ions in the sample can be determined by titrating it with against standard sulphuric acid (H_2SO_4) using phenolphthalein and methyl orange as indicators. Addition of phenolphthalein gives pink red colour in the presence of carbonates and titration with H_2SO_4 converts these CO_3^{2-} into HCO_3^- and decolourises the red colour as shown below –



Thus the carbonates neutralization is only half way. These carbonates along with the already present ones are then determined by continuing the titration using methyl orange indicator which gives yellow colour in presence of bicarbonates. On complete neutralization of bicarbonates the yellow colour will change to red.



Obviously the bicarbonate titre value will be less if carbonates were not present. (absence of pink colour). In such a situation, either the same aliquot is used for bicarbonate titration or a fresh sample is analyzed for this. If carbonates are present and neutralized, the volume of H_2SO_4 used in the first phase (carbonate titration) is to be doubled to get the actual volume needed for complete neutralization of the carbonates.

Reagents :

- 1) Saturated H_2SO_4 (0.01N) : Carefully add 2.8 ml of conc. H_2SO_4 to one litre volumetric flask and dilute to one litre with distilled water, the strength will be approximately 0.1N H_2SO_4 . Dilute 100 ml of this solution to 1 litre to obtain 0.01N H_2SO_4 . Standardize it against primary standard, Na_2CO_3 .

- 2) Standard Na_2CO_3 (0.01N) : Dissolve 5.3 gm of A.R. grade Na_2CO_3 in one litre volumetric flask with distilled water, the strength will be 0.1N Na_2CO_3 . Dilute 100 ml of this solution to get 0.01N. This may be used for standardization of 0.01N H_2SO_4 .
- 3) Phenolphthalein (0.25%) : Dissolve 25 gm of pure Phenolphthalein powder in 100 ml of 60% ethyl alcohol.
- 4) Methyl Orange (0.50%) : Dissolve 0.5 gm of dry methyl orange powder in 100 ml of 95.0% ethyl alcohol.

Procedure :

- 1) Transfer 25 ml of water sample to a 150 ml conical flask. Add 2-3 drops of Phenolphthalein.
- 2) If pink red colour appears, titrate it against standard H_2SO_4 till colour disappears. The burette reading (volume used) is designated as Y ml.
- 3) To this colourless solution or in original sample (25 ml) add 2-3 drops of methyl orange. This will develop the yellow colour.
- 4) Again titrate with standard H_2SO_4 till colour changes from yellow to rosy red. Record the volume of H_2SO_4 as Z ml. This volume corresponds to initial carbonate changed to bicarbonates plus initial bicarbonates present in irrigation water.
- 5) Run a blank (25 ml distilled water) and subtract from the titre value to avoid error due to any impurity of chemicals.

Calculations :

a) Carbonates (milliequivalents / litre) CO_3^{2-} (me/L)

$$= \frac{2 (Y - B) \times \text{Normality of H}_2\text{SO}_4 \times 1000}{\text{Sample in ml.}}$$

b) Bicarbonates (milliequivalents / litre) HCO_3^- (me/L)

$$= \frac{(Z - B) - 2 (Y - B) \times \text{Normality of H}_2\text{SO}_4 \times 1000}{\text{Sample in ml.}}$$

Here Y is the burette reading (ml of H_2SO_4) after phenolphthalein is neutralized and Z is the final burette reading (total volume of H_2SO_4) after methyl orange.

Reference Documents :

Singh, Dhyan, Chhonkar, P.K. and Pande R.N., 1999, Assessment of Irrigation Water Quality in "Soil, Plant, Water Analysis" - A methods manual, Indian Agricultural Research Institute, Indian Council of Agricultural Research, New Delhi, 3 : 76-78.

4. DETERMINATION OF CALCIUM AND MAGNESIUM(EDTA TITRIMETRIC METHOD) :

Principle :

The extent of sodium hazard in irrigation water is determined in terms of the sodium concentration in relation to the two useful divalent cations namely Ca^{++} and Mg^{++} . The most common method of calcium and magnesium determination in irrigation water is by complexometric titration using sodium salt of ethylene-diamine tetra acetic acid. (EDTA).

Ethylene diamine tetra acetic acid (EDTA) form soluble complexes with calcium and magnesium ions at an optimum pH of 10.0 and thus removing them from solution without precipitation. The reaction is stoichiometric and essentially instantaneous at temperature near 60°C and the complex formed are very stable. At the same pH the dye erichrome blue-black B has a turquoise blue colour in the absence of calcium and magnesium ions but forms red compounds with them which are less stable than the EDTA-Ca and EDTA-Mg complexes. The formation of Ca and Mg complexes at pH 10.0 is achieved by using ammonium hydroxide-ammonium chloride buffer.

A number of polyvalent ions are preferably complexed by EDTA as these are less dissociated than those of Ca and Mg and thus included in the titration. Fortunately, the concentration of such interfering metals e.g. Fe, Cu, Pb, Cd, Zn, Co and Mn is quite low and negligible in most waters and can be ignored. However, the interference, if high, can be prevented by using 2% solution of NaCl. If the sample is made strongly alkaline (pH about 12.0), magnesium is selectively precipitated as magnesium hydroxide. At the same pH Patton and Reeder's indicator / ammonium purpurate (murexide) forms a red compound with calcium ions but is not affected by magnesium present as magnesium hydroxide. If EDTA is then closely added, the calcium ions are gradually transferred from the dye complex to the more stable EDTA complex until when all have been transferred, the liquid acquired a pure turquoise blue colour. The reaction is virtually instantaneous at normal room temperature.

Reagents :

- 1) **Standard Calcium Chloride Solution (0.01N)** : Dissolve exactly 0.5005 gm of A.R. grade CaCO_3 (dried at 150°C) in minimum (about 10 ml) of 0.2N HCl (AR). Boil gently to expel the CO_2 . Cool and neutralize the excess acid with ammonia (pH near 5.0). Then make the volume accurately to 1 litre. This solution is for standardizing EDTA.
- 2) **EDTA 0.01N** : Dry the disodium salt of EDTA ($\text{Na}_2\text{H}_2\text{C}_{10}\text{H}_{12}\text{O}_8\text{N}_2 \cdot 2\text{H}_2\text{O}$) at 80°C for about 2 hrs and cool in a desiccator. Dissolve 1.8613 in one litre of distilled water.
- 3) **Ammonium Hydroxide – Ammonium Chloride Buffer (pH 10.0)** : Dissolve 67.5 gm of ammonium chloride (NH_4Cl) in 200 ml of distilled water. Add 570 ml of concentrated ammonium hydroxide (NH_4OH) and dilute the solution to a volume of 1 litre with distilled water and adjust the pH at 10.0.
- 4) **Erichrome Black T Indicator** : Homogenize 0.2 gm of EBT in 50 gm of KCl or NaCl. Erichrome blue-black – B, 0.5% in ethanol. Dissolve 0.5 gm Erichrome blue-black – B in 100 ml of 95% of ethanol.
- 5) **Standard Magnesium Chloride (0.01N)** : Dissolve 1.0165 gm magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in distilled water and make up the volume to 1 litre with distilled water. Standardize by titration with 0.01N EDTA and dilute to 0.01N exactly.
- 6) **Sodium Hydroxide (10%)** : Dissolve 10 gm of NaOH in 100 ml of distilled water.
- 7) **Patton's and Reeder's Indicator (HHSNN Indicator)** : Mix 1.0 gm of HHSNN intimately with 100 gm anhydrous sodium sulphate. Store in a dark bottle away from light. If Patton's and Reeder's indicator is not available, Murexide indicator may be used.

- 8) **Murexide Indicator** : Take 0.2 gm of murexide (also known as ammonium purpurate) and mix it with 40 gm of powdered potassium sulphate. This indicator is kept in powdered form as it goes oxidized in the solution form.

Procedure for Ca + Mg :

- 1) Take 25 ml of sample in 100 ml of conical flask and dilute the content by adding about 25 ml of distilled water.
- 2) Add 4 ml of $\text{NH}_4\text{Cl} + \text{NH}_4\text{OH}$ buffer (see note). Warm to about 60°C (see note).
- 3) Add a pinch of EBT indicator and titrate with 0.01 N EDTA to a pure turquoise blue without any traces of red. This titre value may be considered as "A".
- 4) Before carrying out a batch of determinations, titrate 20 ml 0.01N magnesium chloride with 0.01N EDTA in order to check the EDTA concentration and provide a pure blue standard for use in the subsequent titration.

Procedure for Calcium (Ca^{++}) :

- 1) Take 10 ml water sample in 100 ml of conical flask and dilute the content by adding about 25 ml of distilled water.
- 2) Add approximately 5 ml of 10% NaOH solution to raise the pH to 12.0. warm to about 60°C .
- 3) Add a pinch of HHSNN indicator mixture or Murexide indicator and titrate with 0.01N EDTA to a pure turquoise blue without any traces of red. This titre value may be considered as "B".
- 4) Before carrying out a batch of determinations, titrate 20 ml 0.01N calcium chloride. (A little 0.01N Magnesium chloride may be added) with 0.01N EDTA in order to check the EDTA concentration.

Calculations :

$\text{Ca}^{2+} + \text{Mg}^{2+}$ milli equivalents per litre (me/L) of water

$$= \frac{\text{ml of EDTA} \times \text{Normality of EDTA} \times 1000}{\text{water sample (ml)}}$$

$$= \frac{A (\text{titre value}) \times 0.01 \text{ N} \times 1000}{\text{water sample (ml)}}$$

(1 ml of 0.01N EDTA = 0.01 me of Ca or Ca+Mg or Mg in given aliquote).

Ca^{2+} (me/L)

$$= \frac{\text{ml of EDTA} \times \text{Normality of EDTA} \times 1000}{\text{water sample (ml)}}$$

$$= \frac{B (\text{titre value}) \times 0.01 \text{ N} \times 1000}{\text{water sample (ml)}}$$

Mg^{2+} (me/L)

$$= \text{me / L } (\text{Ca}^{2+} + \text{Mg}^{2+}) - \text{me / L } (\text{Ca}^{2+})$$

Notes :

- Erichrome blue – black B is stable in solution for some weeks, whereas erichrome black T must be used in solid form to give good colours.
- Erichrome blue – black B is sodium 1 – (1-hydroxy – 2- naphthylazo) – 2 – naphthol – 4 – Sulphonate; erichrome black T contains a nitro group in addition and is sodium 1 – (1 – hydroxy – 2 – naphthylazo) – 6 – nitro – 2 – naphthol – 4 – sulphonate.

- Add 5 ml of buffer to 50 ml water and read the pH, which should be 10.0 ± 0.1. Adjust the volume of buffer in sample to bring the pH 10.0
- Preliminary tests with about 50 ml water the time of heating to bring the volume of water to near 60°C (say about 2 minutes). In routine work, time is more convenient than temperature measurement for determination.
- The HHSNN colour sometimes tends to fade rather quickly and a permanent blue standard cannot normally be used for matching. If HHSNN is not available, murexide may be used although the colour change is much less satisfactory.
- Atomic absorption spectrophotometry may be used to determine calcium and magnesium separately, if the equipment is available.

Reference Documents :

Page A. L., Miller R.H., and Keeney D.R., 1982, calcium and Magnesium by EDTA titrimetry in Method of Soil Analysis, Chemical & microbiological properties, 2nd ed., American Society of Agronomy, Inc and Soil Science Society of America, Inc, Madison, Wisconsin, USA, 9(2) : 252-256.

Devis J. and Freitas, 1970, Calcium plus Magnesium, Physical and Chemical Methods of Soil & Water Analysis, Food and Agriculture Organization of the United Nations, Rome, Italy, Soil Bulletin – 10 : 212-215.

5. DETERMINATION OF SODIUM ON FLAME PHOTOMETER :Principle :

Irrigation water may have two types of hazards viz. salinity and sodium hazard. The latter is expressed as the residual sodium carbonate (RSC) and sodium adsorption ratio (SAR). Sodium constitutes 50% or more of cations of saline and sodic water. The content of sodium may be quite high in saline water with EC greater than 1 ms/cm, and containing relatively less amount of Ca^{+2} and Mg^{+2} .

At higher levels, it also exerts a toxic effect on plant growth. Therefore determination of sodium in irrigation water is very important for predicting its harmful effect on soil and crops and judging the suitability of water for irrigation. The concentration of sodium in water sample is determined by flame photometer.

Reagents :

Standard Stock Solution (100 me Na / L) : Dissolve 5.845 gm of AR grade dried NaCl in water and make the volume to 1 litre with distilled water.

Working Standard Solution of Na : Dilute 5, 10, 15, 20, 30, 40 & 50 ml portions of the stock solution (containing 100 me Na / L) to 100 ml in volumetric flasks to get working standards of 5, 10, 15, 20, 30, 40 & 50 me Na / L concentrations.

Procedure :

- 1) Filter a portion of the water sample if suspended material is visible. Filtration is desirable as it prevents chocking of the capillary tube of the flame photometer.

- 2) Take the working standard solutions and record the flame photometer reading against each, after sterling zero with distilled water and 100 with highest concentration i.e. 50 meNa / L.
- 3) Feed the test sample and record the reading.
- 4) Draw a standard curve by plotting the readings against Na concentrations.

Calculations :

- 1) Na concentration in water (me/L) is directly obtained from X- axis against reading.
- 2) Residual Sodium carbonate (RSC) : The residual sodium carbonate may be calculated by subtracting the quantity of $\text{Ca}^{2+} + \text{Mg}^{2+}$ from the sum total of carbonates and bicarbonates determined separately in a given sample and expressed in me/L. Thus,

$$\text{RSC} = [\text{CO}_3^{--} + \text{HCO}_3^-] - [\text{Ca}^{++} + \text{Mg}^{++}]$$

- 3) Sodium Adsorption ratio (SAR) : Sodium adsorption ratio is calculated using the formula –

$$\text{SAR} = \frac{\text{Na}^+}{\sqrt{\text{Ca}^{++} + \text{Mg}^{++} \times 2}}$$

- 4) Quality of irrigation water is judged by RSC as under -

Class	1	2	3
RSC meq/litre	< 1.25	1.25 to 2.5	> 2.5
Interpretation	Safe	Marginal	Unsuitable

Note :

When water sample contains lower Na (meq/L), prepare the standard range as 1, 2, 4, 6, 8 and 10 meq/L from stock solution (100 me Na / L)

Reference Documents :

Singh, Dhyan, Chhonkar, P.K. and Pande R.N., 1999, Assessment of Irrigation Water Quality in "Soil, Plant, Water Analysis" - A methods manual, Indian Agricultural Research Institute, Indian Council of Agricultural Research, New Delhi, 3 : 78-79.

Diagnosis and improvement of saline & alkali soils – United States Salinity Laboratory Staff, Agricultural Handbook No. 60, United States of Agriculture, P. 79-81.

6 DETERMINATION OF CHLORIDE :

Principle :

Chlorides being highly soluble are present in water but the amount is often very low in natural water. However, their contents may be appreciable when the electrical conductivity is high. The determination of chloride is easily made by AgNO_3 titration (Mohr's titration) method in which silver reacts with chloride forming white AgCl precipitate in the presence of sulphuric acid. When all the chlorides are precipitated, potassium chromate (the indicator used) shows the brick red colour at the end point due to the formation of silver chromate. The reaction involved are as follows –



Initially the Cl^- ions are precipitated as AgCl and dark brick red precipitate of Ag_2CrO_4 starts just after the precipitation of AgCl is over.

The optimum acidity for the reaction is about pH 3.0 since the amount of carbonates and bicarbonates present is known from determinations. This optimum pH or one near it may easily be obtained by adding a calculated volume of standard sulphuric acid.

Reagents :

- 1) Standard sodium chloride solution (0.02N) : Dry AR grade NaCl in oven at 80°C for one hour and dissolved 1.170 g in distilled water and make up the volume to one litre.
- 2) Standard Silver Nitrate Solution (0.02N) : Dissolve 3.40 g AgNO_3 in distilled water and make up the volume to one litre.

- 3) Potassium Chromate Indicator (5%) : Dissolve 5.0 g of K_2CrO_4 in 100 ml of distilled water.

Procedure :

- 1) Transfer 25 ml of water sample to a 150 ml of conical flask.
- 2) Add 0.01N H_2SO_4 with methyl orange to neutralize the amount of carbonate and bicarbonate and provide 1 ml in excess.
- 3) Add 5-6 drops of potassium chromate indicator making it dark yellow.
- 4) Titrate the contents against 0.02N $AgNO_3$ solution with continuous stirring till the first brick red tinge appears. Note the volume of the $AgNO_3$ required (ml.)
- 5) Run a blank of 25 ml of distilled water and subtract from the titre value to avoid error due to any impurity of chemicals.
- 6) Determine the normality of $AgNO_3$ by standardizing it against NaCl solution.

Calculation :

$$\text{Chloride (meq/lit.)} = \frac{(V - B) \times N \times 1000}{\text{sample (ml)}}$$

Where N = Calculated normality of $AgNO_3$, B = Blank Titre value

Notes :

- 1) After appearance of white $AgCl$ precipitate, add $AgNO_3$ dropwise to get exact start of red precipitation.
- 2) Always store silver nitrate in dark amber coloured bottle.

Reference Documents :

Devis J. and Freitas, 1970, Calcium plus Magnesium, Physical and Chemical Methods of Soil & Water Analysis, Food and Agriculture Organization of the United Nations, Rome, Italy, Soil Bulletin – 10 : 231-232.

Jackson M.L., 1967, Chloride Determination, in Soil Chemical Analysis, Prentice hall of India Pvt. Ltd., New Delhi, 10:261-263.

7. DETERMINATION OF SULPHATE ON SPECTROPHOTOMETER(CHESNIN AND YIEN)

Principle :

The traces of sulphate occur universally in all types of waters. Its content may be appreciable in most saline water showing EC greater than a dS/m (25°C). Sulphate can be determined turbidimetrically.

The sulphate content is determined by the extent of turbidity created by precipitated barium Sulphate suspension. Barium chloride is added to ensure fine and stable suspension of BaSO₄ at a pH of about 4.8. It also eliminates the interference of phosphate and silicate. Fine suspension of BaSO₄ is stabilized by Gum Acacia and the degree of turbidity measured by Turbidimeter or Spectrophotometer.

Reagents :

- 1) Barium Chloride Crystals (AR grade) : Pure BaCl₂ crystals ground to pass through 0.5 mm sieve but retained on a 0.25 mm sieve.
- 2) Gum Acacia (stabilizing reagent) : Dissolve 0.25 g of gum acacia in 100 ml of distilled water. Keep overnight and filter.
- 3) Sodium Acetic Acid Buffer : Dissolve 100 g of pure sodium acetate in 200 ml of distilled water. Add 31 ml of glacial acetic acid and make volume to one litre. The solution pH should be 4.8.
- 4) Standard Sulphate Stock Solution : 10 me/L of SO₄²⁻S : Dissolve 0.870 g of AR grade potassium sulphate (K₂SO₄) in about 800 ml of S free distilled water and adjust the volume to one litre. The strength of solution will be 10me/L of SO₄²⁻S.

Preparation of Standard Curve :

- 1) Take 25 ml volumetric flask, add 0.0, 1.0, 2.0, 4.0, 6.0, 8.0 and 10.0 ml of 10 me/L of SO_4^{2-} (for obtaining a concentration of SO_4^{2-} 0.0, 0.4, 0.8, 1.6, 2.4, 3.2 and 4.0 me/L)
- 2) Add 7.5 ml of sodium acetate acetic acid buffer to maintain the pH around 4.8, Add 2.0 ml of gum acacia and 1 g BaCl_2 crystals and shake well. Make the volume of 25 ml mark by adding distilled water. Invert the flask several times.
- 3) Measure the turbidity after 30 minutes with Spectrophotometer at 490 nm wavelength.
- 4) From this standard curve SO_4^{2-} content in the water is determined.

Procedure :

- 1) Pipette out 10 ml of water sample in the 25 ml of volumetric flask.
- 2) Add 7.5 ml sodium acetate acetic acid buffer to maintain the pH around 4.8. Add 2 ml of gum acacia and 1.0 g BaCl_2 crystals and shake well.
- 3) Make the volume to 25 ml mark with distilled water, invert the flask several times and measure the turbidity after 30 minutes with spectrophotometer at 499 nm wavelength.
- 4) If the water has $\text{EC} > 1.0 \text{ dS/m}$, dilute it with distilled water to bring the electrical conductivity around 1.0 dS/m.

Calculation :

$$\text{SO}_4^- = \frac{\text{GR} \times 25}{\text{sample (ml)}}$$

Where GR = SO_4 (me/L) concentration from the standard curve. If the dilution of the sample is done, the dilution factor may be taken into account.

Notes :

Buffer solutions should be prepared very carefully so that it gives pH of 4.8.

BaCl_2 should be added as a solid crystal to the sulphate solution.

Reference Documents :

Singh, Dhyan, Chhonkar, P.K. and Pande R.N., 1999, Assessment of Irrigation Water Quality in "Soil, Plant, Water Analysis" - A methods manual, Indian Agricultural Research Institute, Indian Council of Agricultural Research, New Delhi.

Diagnosis and improvement of saline & alkali soils – United States Salinity Laboratory Staff, Agricultural Handbook No. 60, United States of Agriculture.

LIST OF APPENDICES

SECTION - A :

APPENDIX –1	International Atomic Weights
APPENDIX –2	Normality, Specific Gravity and Percent by weight of Acids and Ammonia Reagent in the Laboratory
APPENDIX –3	Approximate pH values of some acid and alkali
APPENDIX –4	Preparation of 1000 ppm solutions of different elements
APPENDIX –5	List of the chemicals that serve as primary standard for the respective reactions with their equivalent weights
APPENDIX –6	Preparation of Standard Solutions
APPENDIX – 7	Some Important Conversion Factors
APPENDIX – 8	Conversion factors for different elements
APPENDIX – 9	EC reading according to temperature
APPENDIX – 10	Some Indicator plants of nutrient deficiency
APPENDIX – 11	Instrument settings for flame atomic absorption analysis
APPENDIX – 12	Terminology

SECTION - B :

APPENDIX –1	General safe laboratory operation procedures
1.	General safe procedures
2.	Bases
3.	Acids
4.	Flammables & Combustibles
5.	Compressed gas cylinders

SECTION - A

APPENDIX - 1 : International Atomic Weights

Element	Symbol	At. Wt.	Element	Symbol	At. Wt.
Aluminium	Al	26.97	Neon	Ne	20.18
Antimony	Sb	121.76	Nickel	Ni	58.69
Argon	A	39.94			
Arsenic	As	74.91	Columbium	Cb	92.91
Barium	Ba	137.36	Nitrogen	N	14
Beryllium	Be	9.02	Osmium	Os	190
Bismuth	Bi	209	Oxygen	O	16
Boron	B	10.92	Baltadium	Pd	106.7
Bromine	Br	79.92	Phosphorus	P	30.98
Cadmium	Cd	112.41	Platinum	Pt	195.23
Caesium	Cs	132.91	Potassium	K	39.09
Calcium	Ca	40.08	Praseodymium	Pr	140.92
Carbon	C	12.01	Protoactinium	Pa	231
Cerium	Ce	140.13	Radium	Ra	226.05
Chlorine	Cl	35.46	Radon	Rn	222
Chromium	Cr	52.01	Rhenjum	Re	186.31
Cobalt	Co	58.94	Rhodium	Rh	102.91
Copper	Cu	63.57	Rubidium	Rb	85.46
Disprosium	Dy	162.46	Ruthenium	Ru	101.7
Erbium	Er	167.2	Samarium	Sm	150.43
Europium	Eu	152	Scandium	Sc	45.1
Fluorine	F	19	Selenium	Se	78.96
Gadolinium	Gd	156.9	Silicon	Si	28.06
Gallium	Ga	69.72	Silver	Ag	107.88
Germanium	Ge	72.6	Sodium	Na	23
Gold	Au	197.2	Strontium	Sr	87.63
Hafnium	Hf	178.6	Sulphur	S	32.07
Helium	He	4	Tantalum	Ta	180.88
Holmium	Ho	163.5	Tellurium	Te	127.61
Hydrogen	H	1.01	Terbium	Tb	159.2
Indium	In	114.76	Thallium	Tl	204.39
Iodine	I	126.92	Thorium	Th	232.12
Iridium	Ir	193.1	Thulium	Tm	169.4
Iron	Fe	55.84	Tin	Sn	118.7
Krypton	Kr	83.7	Titanium	Ti	47.9
Lanthanum	La	138.92	Tungsten	W	183.92
Lead	Pb	207.21	Uranium	U	238.07
Lithium	Li	6.94	Vandadium	V	50.95
Lutesium	Lu	175	Xenon	Xe	131.3
Magnesium	Mg	24.32	Ytterbium	Yd	173.04
Manganese	Mn	54.93	Yttrium	Y	88.92
Mercury	Hg	200.61	Zinc	Zn	65.38
Molybdenum	Mo	95.95	Zirconium	Zr	91.22

APPENDIX – 2 Normality, Specific Gravity and Percent by Weight of Acids and Ammonia Reagent in the Laboratory

Sr. No.	Reagent	Normality N*	Specific gravity	ml required to make 1 litre of 1N solution
1	Acetic acid glacial	17.4	1.05	58 ml
2	Hydrochloric acid Conc.	11.8	1.18	89 ml
3	Nitric acid Conc.	16	1.42	63 ml
4	Sulphuric acid Conc.	41.1	1.69	23 ml
5	Phosphoric acid Conc.	36	1.84	28 ml
6	Perchloric acid	11.6	1.66	86 ml
7	Ammonium Hydroxide Conc.	14.3	0.9	71 ml
8	Phosphoric acid	41.1	1.69	23 ml

N* = Normality approximate

Note : To prepare a diluted reagent from a concentrated one use the expression

$$V_1 = V_2 \times N_2 / N_1$$

Where -

V_1 – Vol. of the conc. reagent required
 V_2 – Vol. of the diluted reagent

N_1 – Normality of the conc. reagent
 N_2 – Normality of the diluted reagent

APPENDIX – 3 Approximate pH values of some acid and alkali

Substance	Normality	pH	Substance	Normality	pH
HCl	1	0.1	NaOH	1	14.05
	0.1	1.07		0.1	14.07
	0.01	2.02		0.01	12.12
	0.001	3.01		0.001	11.13
Acetic acid	1	2.37	NH ₄ OH	1	11.6
	0.1	2.87		0.1	11.1
	0.01	3.37		0.01	10.6
	0.001	3.87		0.001	10.1
H ₂ SO ₄	0.1	1.2	Oxalic acid	0.1	1.6

APPENDIX – Preparation of 1000 ppm solutions of different elements

Sr. No.	Elements	Quantity of salt to be dissolved in 1000 ml distilled water
1	Nitrogen	4.719 g of $(\text{NH}_4)_2\text{SO}_4$ Ammonium sulphate 2.140 g urea $\text{CO}(\text{NH}_2)_2$
2	Phosphorus	4.390 g of KH_2PO_4 Pot. Dihydrogen phosphate 5.555 g of $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$, Dicalcium phosphate dihydrate 14.31 g of single superphosphate containing 6.986% P
3	Potassium	1.906 g Potassium chloride (Muriate of potash) KCl
4	Zinc (Zn)	4.398 g Zinc sulphate $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$
5	Manganese (Mn)	3.609 g Manganese chloride, $\text{MnCl}_2 \cdot 2\text{H}_2\text{O}$ 3.077 g Manganese sulphate $\text{MnSO}_4 \cdot \text{H}_2\text{O}$
6	Copper (Cu)	3.929 g Copper sulphate $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$
7	Iron (Fe)	4.978 g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, Ferrous sulphate 7.021 g $(\text{NH}_4)_2 \cdot \text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ of Ferrous ammonium sulphate
8	Molybdenum	12.139 g Ammo. Molybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ 2.521 g of Sodium molybdate $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$
9	Boron	5.720 g Boric acid H_3BO_3

* Add 15 ml of conc. sulphuric acid in solution and then make volume to 1 litre.

APPENDIX-5
List of the chemicals that serve as primary standard for the respective reactions with their equivalent weights

Sr. No.	Name	Formula	Eq. wt.
I.	Acidimetry and alkalimetry		
1	Sodium carbonate	Na_2CO_3	52.99
2	Borax	$\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	190.72
3	Potassium hydrogen phthalate	$\text{KHC}_8\text{H}_4\text{O}_4$	204.22
4	Potassium bi-iodate	$\text{KH}(\text{IO}_3)_2$	389.95
5	Sulphamic acid	$\text{NH}_2\text{SO}_2\text{OH}$	97.09
6	Succinic acid	$\text{H}_2\text{C}_4\text{O}_4$	59.04
7	Benzic acid	$\text{HC}_7\text{H}_5\text{O}_2$	122.12
8	Furoic acid	$\text{HC}_3\text{H}_3\text{O}_2$	112.08
II.	Precipitation reactions		
9	Sodium chloride	NaCl	58.44
10	Potassium chloride	KCl	74.54
III.	Oxidation-Reduction		
11	Potassium dichromate	$\text{K}_2\text{Cr}_2\text{O}_7$	49.04
12	Potassium bromate	KBrO_3	27.84
13	Potassium bi-iodate	$\text{KH}(\text{IO}_3)_2$	389.95
14	Iodine	I_2	126.92
15	Sodium oxalate	$\text{Na}_2\text{C}_2\text{O}_4$	134.02
16	Oxalic acid	$\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$	63.034

Note : Such of the hydrated salts that do not effloresce on drying such as borax, oxalic acid, have been experimentally found to be satisfactory, are known as “secondary standards”.

APPENDIX – Preparation of Standard Solutions

To prepare 1 litre of 0.1 N or N/10 Solution (approx.)		Standardize with	
1	Sulphuric acid Dilute 3.0 ml of conc. 36N acid to 1 lit.	1	Sodium carbonate (N/10) For this weigh exactly 5.29 g of A.R. grade dry sodium carbonate and dissolve in water and make up to 1 litre. OR
2	Hydrochloric acid Dilute 10 ml of conc. 16N acid to 1 lit.		
3	Nitric acid Dilute 6.3 ml of conc. 12N acid to 1 lit.	2	Borax solution (N/10) Weigh exactly 1.907 g of pure A.R. grade borax $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ and dissolve in water & make upto 100 ml.
4	Sodium hydroxide 4.0 g to be dissolved in water and make upto 1 lit.	1	Potassium Hydrogen pthalate (N/10) Dissolve accurately 5.1 g Potassium hydrogen pthalate (A.R. grade) in water and make up to 250 ml.
5	Potassium hydroxide Dissolve 5.7 to 6.0 g of Potassium hydroxide in water and make up to 1 lit.		Succinic acid (N/10) Weigh 5.9 g of succinic acid (A.R.) to be dissolved in water and make up to 1 lit.
6	Silver nitrate Dissolve accurately 16.989 g crystallized (A.R.) silver nitrate in water and make up to 1 lit.		0.25 g of pure iron, 2 g of NaHCO_3 and 20 ml of 5N H_2SO_4
7	Potassium permanganate about 3.25 g Potassium permanganate to be dissolved in water and make up to 1 lit.		Oxalic acid 1.574 pure oxalic acid to be dissolved in water and make upto 250 ml, makes 0.1N (N/10) Sodium oxalate 1.67 pure sodium oxalate dissolve in water and make upto 1 lit, makes 0.01 N.

APPENDIX –6 (Cont.)

Sr. No.	Reagent	Preparation of Standard Solutions Quantity of reagent requires to prepare solution of known concentration
1	1N HCl	87.5 ml conc. HCl / litre
2	0.5N HCl	44 ml per litre
3	5N H ₂ SO ₄	140 ml conc. H ₂ SO ₄ per litre
4	0.1N H ₂ SO ₄	2.8 ml H ₂ SO ₄ per litre
5	0.2N H ₂ SO ₄	5.6 ml H ₂ SO ₄ per litre
6	0.01N	100 ml of 0.1N H ₂ SO ₄ per litre
7	0.1N Pot. Acid pthalate	5.1 g pure oven dried Pot. Acid pthalate in 250 ml distilled water.
8	0.25N NaOH	10 g NaOH per litre
9	0.1N NaOH	4 g NaOH per litre
10	4N NaOH	160 g NaOH per litre
11	0.02N AgNO ₃	3.4 g AgNO ₃ per litre
12	0.1N KCl	0.7456 g KCl per litre
13	0.1N NaCl	5.845 g NaCl per litre
14	100 ppm P solution	0.439 g Potassium hydrogen orthophosphate (A.R. grade) per litre
15	1000 ppm K solution	1.907 g KCl (A.R. grade) per litre
16	100 meq Na	5.845 g NaCl per litre
17	0.01N EDTA	2.0 g disodium dihydrogen EDTA + 0.05 g MgCl ₂ 6H ₂ O per litre

APPENDIX –6 (Cont.)

Normal solution : A normal solution contains 1 g equivalent weight of solute in a litre of the solution.
Preparation and Standardization of Solutions

To prepare 1000 ml (1 litre) of 0.1N solution (approximately)	Standardize with
Sodium Hydroxide 4.0 g to be dissolved in water and make up to 1 litre Potassium Hydroxide Dissolve 5.7 to 6.0 g of potassium hydroxide in water and make up to 1 litre.	Potassium Hydrogen Pthalate (0.1N) Dissolve accurately 5.0 g of potassium hydrogen pthalate (A.R. grade) in water and make up to 250 ml. Succinic acid (0.1N) Weigh 5.9 g of succinic acid (A.R. grade) to be dissolved in water to make up to 1 litre.
Silver Nitrate Dissolve accurately 16.989 g crystallized (A.R.) silver nitrate in water & make up to 1 litre.	NaCl sodium chloride (0.1N) Weigh 5.486 g of sodium chloride (A.R. grade) and dissolve in water to make up to 1 litre.
Sulphuric Acid Dilute 2.8 ml of conc. 36N sulphuric acid to 1 lit. $(36N \times ? \text{ ml} = 0.1 N \times 1000) = 2.8 \text{ ml}$ OR considering the gram equivalent wt., purity and sp. Gravity of the H_2SO_4 , dilute calculated amount of H_2SO_4 in distilled water and make up to 1 litre. Hydrochloric Acid Dilute 8.33 ml of conc. 12.0 N hydrochloric acid to 1 litre of distilled water. $(12N \times ? \text{ ml} = 0.1 N \times 1000) = 8.33 \text{ ml}$ Nitric Acid Dilute 6.3 ml of conc. 16N nitric acid to 1 litre of distilled water.	Sodium Carbonate (0.1N) For this weigh exactly 5.29 g of A.R. grade dry sodium carbonate and dissolve in water and make up to 1 litre.

APPENDIX – 7

Some Important Conversion Factors

To covert column-1 to column-2, multiply by the factor	Column-1	Column-2	To covert column-2 to column-1, multiply by the factor
0.892	Kg/ha.	lb/acre	1.121
220.2	Quintal	lbs	0.00454
2.205	Kg	lb	0.454
1.094	Meter	yard	0.914
0.394	cm.	inch	2.540
2.29	P	P ₂ O ₅	0.437
1.20	K	K ₂ O	0.830
1.39	Ca	CaO	0.715
1.66	Mg	MgO	0.602
2.24	ppm	Kg/ha	0.446
0.10	g/kg	Percent	10.0
0.0001	ppm	Percent	10000.0
22.4	mg/100 g	Kg/ha	0.0446
10.00	mg/100 g	ppm	0.10
(9.0/5.0 °C) + 32	°C	°F	(5.0/9.0 (°F – 32)

ppm = miliequivalent per litre x eq.wt.
 meq. Per litre = microgram per ml or mg per litre
 meq. = ppm / equivalent weight
 1 N solution = gram equivalent weight / litre
 mg / 100 g = ppm x 0.1

APPENDIX – 8

Conversion factors for different elements

1.	P_2O_5	x	0.44	=	P
2.	K_2O	x	0.83	=	K
3.	P	x	2.29	=	P_2O_5
4.	K	x	1.20	=	K_2O
5.	N	x	1.22	=	NH_3
6.	Ca	x	1.40	=	CaO
7.	CaO	x	0.71	=	Ca
8.	Mg	x	1.67	=	MgO
9.	MgO	x	0.60	=	Mg
10.	S	x	3.00	=	SO_4
11.	SO_4	x	0.33	=	S
12.	HNO_3	x	0.22	=	N
13.	H_3PO_4	x	0.32	=	P
14.	$Ca_3(PO_4)$	x	0.20	=	P
15.	KCl	x	0.52	=	K
16.	K_2SO_4	x	0.45	=	K
17.	$CaCO_3$	x	0.40	=	Ca
18.	$CaCO_4$	x	0.29	=	Ca
19.	$MgCO_3$	x	0.28	=	Mg
20.	$MgSO_4$	x	0.20	=	Mg
21.	H_2SO_4	x	0.33	=	S
22.	$CaSO_4$	x	0.24	=	S
23.	Na	x	2.305	=	Na_2CO_3

APPENDIX – 9

EC reading according to temperature

Temp.	EC	Temp.	EC	Temp.	EC
20.0	1.276	25.2	1.4185	30.2	1.556
20.2	1.281	25.4	1.4240	30.4	1.561
20.4	1.287	25.6	1.4294	30.6	1.567
20.6	1.292	26.8	1.4349	30.8	1572
20.8	1.298	26.0	1.4404	31.0	1.577
21.0	1.303	26.2	1.4459	31.2	1.583
21.2	1.309	26.4	1.4514	31.4	1.588
21.4	1.314	26.6	1.4569	31.6	1.594
21.6	1.320	26.8	1.4623	31.8	1.599
21.8	1.325	27.0	1.4678	32.0	1.605
22.0	1.331	27.2	1.4733	32.2	1.610
22.2	1.336	27.4	1.4788	32.4	1.616
22.4	1.342	27.6	1.4843	32.6	1.621
22.6	1.347	27.8	1.4898	32.8	1.627
22.8	1.353	28.0	1.4952	33.0	1.632
23.0	1.358	28.2	1.5007	33.2	1.639
23.2	1.364	28.4	1.5062	33.4	1.643
23.4	1.369	28.6	1.5117	33.6	1.649
23.6	1.375	28.8	1.5172	33.8	1.654
23.8	1.380	29.0	1.5226	34.0	1.660
24.0	1.386	29.2	1.5281	34.2	1.665
24.2	1.391	29.4	1.5336	34.4	1.671
24.4	1.397	29.6	1.5391	34.6	1.676
24.6	1.402	29.8	1.5446	34.8	1.682
24.8	1.408	30.0	1.5501	35.0	1.687
25.0	1.413				

APPENDIX – 10

Nutrient	Some Indicator plants of nutrient deficiency Indicator Plants
N	Maize, cereal (small grain), mustard, apple, citrus.
P	Maize, barley, lettuce, tomato.
K	Potato, lucern, beans, tobacco, cucurbits, cotton, tomato, maize.
Ca	Lucern, other legume crops.
Mg	Potato, cauliflower.
S	Lucern, raya.
Fe	Sorghum, barley, citrus, peach.
Zn	Maize, onion, citrus, peach.
Mn	Apple, cherry, citrus, maize, oats, pea, radish, wheat.
Cu	Apple, citrus, barley, maize, lettuce, oats, onion, tobacco, tomato,
B	Lucern, turnip, cauliflower, apple, peach.
Cl	Lettuce.
Mo	Cauliflower, other <i>Brassica</i> sp., citrus, legumes, oats, spinach.

APPENDIX – 11

Instrument settings for flame atomic absorption analysis

Element	Line (nm)	Slit width (nm)	Working range (µg m/L)	Sensitivity (µg m/L)	Lamp current (mA)	Flame type
Al	309.3	0.5	25-135	1.0	10.0	N ₂ O-C ₂ H ₂ (Red.)
Al	396.2	0.5	25-110	1.0	10.0	N ₂ O-C ₂ H ₂ (Red.)
Ca	422.7	0.5	1-4	0.02	10.0	N ₂ O-C ₂ H ₂ (Oxi.)
Ca	422.7	0.5	1-10	0.09	10.0	Air- C ₂ H ₂ (Sto.)
Cd	228.8	0.5	0.5-5	0.03	3.0	Air- C ₂ H ₂ (Oxi.)
Co	240.7	0.2	1-20	0.2	6.0	Air- C ₂ H ₂ (Oxi.)
Cr	357.9	0.2	2-20	0.2	6.0	Air- C ₂ H ₂ (Red.)
Cr	425.4	0.2	7-40	0.5	6.0	Air- C ₂ H ₂ (Red.)
Cu	324.7	0.5	1-20	0.1	3.0	Air- C ₂ H ₂ (Oxi.)
Cu	217.9	0.2	7.5-30	0.2	3.0	Air- C ₂ H ₂ (Oxi.)
Fe	248.3	0.2	2-20	0.1	7.0	Air- C ₂ H ₂ (Oxi.)
Fe	372.0	0.2	20-40	0.5	7.0	Air- C ₂ H ₂ (Oxi.)
K	766.5	0.5	1-10	0.01	6.0	Air- C ₂ H ₂ (Oxi.)
Mg	285.2	0.5	0.1-2	0.01	3.0	Air- C ₂ H ₂ (Oxi.)
Mg	202.6	1.0	5-20	0.1	3.0	Air- C ₂ H ₂ (Oxi.)
Mn	279.5	0.2	1-10	0.06	5.0	Air- C ₂ H ₂ (Sto.)
Mn	403.1	0.2	7-27	0.2	5.0	Air- C ₂ H ₂ (Sto.)
Mo	313.3	0.2	10-50	0.8	7.0	N ₂ O- C ₂ H ₂ (Red.)
Ni	232.0	0.2	2-20	0.2	4.0	Air- C ₂ H ₂ (Oxi.)
Ni	352.4	0.5	6-30	0.2	4.0	Air- C ₂ H ₂ (Oxi.)
Pb	283.3	0.5	4-40	0.2	5.0	Air- C ₂ H ₂ (Oxi.)
Se	196.1	1.0	45-185	1.0	10.0	N ₂ O- C ₂ H ₂ (Sto.)
Si	251.6	0.2	20-200	2.0	15.0	N ₂ O- C ₂ H ₂ (Red.)
Zn	213.9	0.5	0.5-5	0.03	5.0	Air- C ₂ H ₂ (Oxi.)

APPENDIX – 12

Standard Solution :

Terminology

The solution of accurately known strength (or concentration) is called standard solution. It contains a definite number of gram equivalent or gram moles per litre of solution. Strength of a solution refers to the weight of a solute dissolved in a unit weight of the solution. It can be expressed in many ways as follows –

Normal Solution and Normality : A normal solution is one which contains 1 gram equivalent (eq.wt. in grams) of the active reagent, dissolved in 1 litre of the solution. Normality is the number of gram equivalent of the substance, dissolved in 1 litre of the solution. If the number of gram equivalent is 1, it is expressed as 1 N. If the number is 1/10, 1/100 or 1/1000 then it is designated as 0.1 N (decinormal), 0.01 N (centinormal) or 0.001 N (millinormal) solution respectively.

$$\text{Normality} = (\text{No. of gram eq. of solute}) / (\text{No. of litre of solution})$$

Molar Solution and Molarity : A molar solution is one which contains a gram of molecular weight of the solute dissolved in 1 litre of solution. It is denoted by M. Whereas, molarity is the number of gram molecules of the substance dissolved in 1 litre of the solution.

Molal Solution : A molal solution is the one which contains a number of gram molecules of the solute dissolved in 100 g of water.

Percentage composition by weight : The concentration is expressed in terms of the gram of solute per 100 g of solution. e.g. 10% KCl solution is prepared by dissolving 10 g of the salt in 90 g of water.

Percentage composition by volume : The concentration is expressed in terms of volume of the solute and solvent. e.g. 25 g of solution of methanol is prepared by mixing 25 ml of methanol with 80 ml of water.

Parts per million (ppm) : The concentration is expressed in terms of grams of solute per million millilitres of solution or milligrams of solute per litre of the solution. Thus a solution containing 10 mg / litre of solute or 10 microgram of solute per millilitre of solution is 10 ppm solution.

Milli equivalent per litre : A solution containing milli (1/1000) g equivalent of substance in a litre of the solution is expressed as meq/litre.

Titration : titration is defined as the process of determining the volume of a substance required to just complete the reaction with a known amount of other substance. The quantitative analysis carried out by titration is known as titrimetric analysis.

a) **Titrant** : The solution of accurately known strength used in titration is called titrant.

b) **Titrate** : The substance (in solution) to be determined by titration is called titrate.

SECTION - B

GENERAL SAFE LABORATORY OPERATION PROCEDURES

1. GENERAL SAFETY PROCEDURES

Inform yourself :

- ❖ Consult the material safety data sheets (MSDS) to learn the hazards of each chemical. (MSDS can be obtained from chemical suppliers).
- ❖ It is highly recommended that all supervisors, employees, students and volunteers get Workplace Hazardous Materials Information System (WHMIS) certification. This system informs workers of commonly used warning labels and symbols for chemicals and other agents used in the workplace.
- ❖ Follow all policies, regulations and safety procedures (municipal, provincial/state and federal) detailed for your workplace.
- ❖ Verify that the appropriate personal protection equipment (PPE) is available and used as prescribed.
- ❖ Special attention is required if there are any level 4 hazards listed on the chemical's National Fire Protection Association (NFPA) label regarding health (blue), fire (red) or reactivity (yellow). Level 4 hazards indicate extreme hazard potential. Special training or safety requirements must be attained before handling these chemicals.
- ❑ Label chemical bottles and containers when received and opened, as per WHMIS guidelines. Most chemicals have a shelf life. Some of these chemicals may become unsafe and/or unstable after expired date.
- ❑ Ensure that there is an adequate supply of the reagents before starting any procedure.
- ❑ Do not carry glass bottles only by the finger-ring on the neck of the bottle. This ring is meant to help grip bottle when pouring its contents. Transport the bottle using both hands or use an appropriate rubber / plastic bottle holder.
- ❑ Store chemicals in an appropriate location as directed in MSDS. Pay special attention to non-compatible chemicals, shelf life and ventilation. Make sure chemicals are properly labeled and an accurate chemical inventory is kept.

2.

BASES

General characteristics and precautions :

- ❖ Bases are caustic and some have low surface tensions, making them difficult to wash off.
- ❖ Eye contact : causes severe eye burns. May cause irreversible eye injury.
- ❖ Skin contact : causes skin burns. May cause deep, penetrating ulcers of the skin.
- ❖ Ingestion : causes gastrointestinal tract burns. May cause perforation of the digestive tract.
- ❖ Bases and acids should be stored separately due to incompatibilities (i.e. potentially violent reaction).
- ❖ Strong bases include the following : LiOH (Lithium Hydroxide), NaOH (Sodium Hydroxide), KOH (Potassium Hydroxide), RbOH (Rubidium Hydroxide) and CsOH (Cesium Hydroxide).

Unique hazards :

Ammonium Hydroxide :

- ❖ Volatile : Produces ammonia fumes which are pungent and toxic. This chemical must be used in a fume hood.

Sodium Hydroxide, Lithium Hydroxide and Potassium Hydroxide :

- ❖ Substances are hygroscopic (i.e. absorb water from the atmosphere).
- ❖ Must be stored in plastic bottles since these bases can fuse glass.
- ❖ These bases are exothermic when dissolved / diluted with water. LiOH may boil if 10 M stock solution is made, NaOH & KOH will heat up significantly. There is a small risk of skin burns.

Sodium Hypochlorite (Bleach) :

- ❖ Toxic if ingested in sufficient quantities.
- ❖ Avoid skin contact as this can cause irritation.
- ❖ Avoid inhaling excessive quantities of vapour.
- ❖ Strong oxidizer : This chemical has several incompatibilities (i.e. acids, ammonia based compounds, hydrogen peroxide and flammables).

3.

ACIDS

General characteristics and precautions :

Most acids are volatile and produce acidic fumes.

Corrosive to most metals, this reaction can form explosive hydrogen gas.

- ❖ Eye contact : causes severe eye burns. May cause irreversible eye injury.
- ❖ Skin contact : causes skin burns. May cause deep, penetrating ulcers of the skin.
- ❖ Ingestion : causes gastrointestinal tract burns. May cause perforation of the digestive tract. Does not include vomiting.
- ❖ Inhalation : may be fatal if inhaled. Effects may be delayed. May cause irritation of the respiratory tract with burning pain in the nose and throat, coughing, wheezing, shortness of breath and pulmonary edema.
- ❖ Chronic effects : repeated inhalation may cause chronic bronchitis.
- ❖ Reacts exothermically with water, sometimes violently. Always add acid to water when making up solutions.
- ❖ Store acids and bases separately.
- ❖ Strong acids include : HCl (Hydrochloric acid), HNO₃ (Nitric acid), H₂SO₄ (Sulphuric acid), HBr (Hydrobromic acid), HI (Hydroiodic acid) and HClO₄ (Perchloric acid).

Unique Hazards :

Acetic Acid :

- ❖ Highly volatile, strong pungent, vinegar-like odour.
- ❖ Flammable in its concentrated form (i.e. glacial).

Hydrochloric acid :

- ❖ Volatile : Releases toxic chlorine gas, vapours are visible in high humidity.

Nitric acid :

- ❖ Strong oxidizer : Reacts violently with some chemicals.
- ❖ Volatile : vapours are visible, especially in high humidity.

Sulphuric acid :

- ❖ Hygroscopic : Absorbs moisture from the air. Keep tightly sealed.
- ❖ Strong inorganic acid. Mists containing sulphuric acid may cause cancer.
- ❖ Sulphuric acid reacts vigorously, violently or explosively with many organic and inorganic chemicals and with water.

Formic acid :

- ❖ Flash point is 69°C. Both liquid and vapour are combustible.
- ❖ Strong reducing agent. : fire and explosion risk if in contact with oxidizing agents. Keep refrigerated (store below 4°C)
- ❖ Lachrymator (i.e. a substance that produces the flow of tears).

Hydrofluoric acid :

- ❖ Poison, extremely hazardous liquid and vapour. Special safety training recommended.
- ❖ Neutralizing HF gel (2.5% calcium gluconate gel) must be kept on your person both at and away from the workplace. A person's reaction to exposure may be delayed by 8 h or longer, depending on the concentration of the acid. Fluoride ions readily penetrate skin, causing deep tissue and bone damage and can be fatal. Any exposure requires hospital care, even after neutralizing gel application.
- ❖ Hydrofluoric acid must be stored in plastic bottles, since HF can dissolve glass.

4. FLAMMABLES AND COMBUSTIBLES

General characteristics and precautions :

- These substances can result in a fire or explosion if in contact with a heat or ignition source.
- Most flammables are volatile and considered to be toxic. Many flammable solvents affect the central nervous system.
- To avoid potential contact with ignition sources, it is important to determine whether fumes are lighter or heavier than air (e.g. chloroform is heavier than air, while natural gases are lighter than air).
- Some flammables can become unstable through time due to peroxide formation, resulting in auto ignition (e.g. diethyl ether, tetrahydrofuran).

Safety Precautions :

- Store in a vented cabinet or room.
- Store away from ignition, heat or oxidizer sources (including sunlight and room heaters).
- If flammables need to be stored cold, they must be stored in a fridge which has been specifically designed by the manufacturer to be suitable for the storage of flammables. The fridge must be labeled as such.
- Reduce routine handling of large volumes of flammable or combustible materials by dispensing into smaller WHMIS-labeled containers. Ensure that metal containers are grounded to prevent static discharge.
- Dispense and use flammable or combustible materials in properly working fume hoods or well ventilated areas. Certification of fume hoods is often mandatory to ensure that adequate airflow is available for safe working conditions.

- Do not use the laboratory as a storage place. Return all containers to the volatile materials storage facility.
- Store flammables separately from other chemicals. It is especially important to store flammables separately from oxidizers.

5. COMPRESSED GAS CYLINDERS General

Characteristics and Precautions :

- Some gases support combustion (e.g. oxygen).
- Some gases are flammable (e.g. acetylene, hydrogen, propane).
- Some gases are asphyxiants (e.g. carbon dioxide, carbon monoxide).
- All gases (except air and oxygen) can displace breathable air if they are exhausted in to non vented, closed areas.
- Incorrect use of pressure regulators can cause fires or explosions.
- High temperatures can cause a buildup of pressure in cylinders.

Safety Precautions :

- Label all cylinders clearly. Do not use a cylinder if its contents cannot be unequivocally identified.
- Keep all unused cylinders well sealed.
- Use appropriate PPE while handling cylinders.
- Ventilate storage areas.
- Secure cylinders individually by using chains or straps.
- Do not store cylinders near open flame or heat source.
- Ground all flammable gas cylinders.
- Oxygen tanks : Ensure all surfaces on the tank and regulator are absolutely free of grease or any other lubricant.

Transportation of Gas Cylinders :

- The appropriate cap must be in place.
- Person(s) transporting the cylinder should wear gloves and safety shoes or boots (steel-toed or equivalent).
- Prior to transport : Ensure that suitable tie-down chains or straps are available immediately upon arrival at the destination place.
- Use freight elevators (where available) to transport cylinders.
- Person(s) transporting compressed gases in vehicles often requires specific (and mandatory) training and licensing.

Reference Documents :

Singh, Dhyan, Chhonkar, P.K. and Pande R.N., 1999, Assessment of Irrigation Water Quality in “Soil, Plant, Water Analysis” - A methods manual, Indian Agricultural Research Institute, Indian Council of Agricultural Research, New Delhi.

Analysis of Plants, Irrigation Water & Soils (MPKV, Rahuri Manual) by R.B. Somwanshi, P.P. Kadu, B.D. Tamboli, B.D. Bhakre.

Methods of Soil & Water Analysis, laboratory Manual, PKV, Akola, M.N. Patil, S.K. Thakare.

Laboratory Manual by National Bureau of Soil Survey & Land Use Planning, Nagpur.

Soil & Water Analysis Methods, Soil Survey & Analysis Department, Commissionerate of agriculture, Maharashtra State, Pune-5.

Soil Sampling & Method of Analysis – 2nd Edition, Canadian Society of Soil Science – 2008.

PREFACE

This un-priced laboratory manual is expected to serve as a source of information on laboratory testing procedure for soil and water sample analysis.

The technical information has been collected from several books, authorized manuals from agricultural universities, agricultural departments of Maharashtra State, National Bureau of Soil Science (NBSS), Nagpur, IARI, ICAR, New Delhi.

The information in this manual is exclusively written for the use of technical staff engaged in doing soil classification, soil survey work, laboratory testing of soil and water samples, Diagnosis of problematic soils in command area and allied research work, in the Directorate of Irrigation Research & Development, Pune.

I sincerely acknowledge the help received from several authors and researchers in the form of their published literature.



Er. K. M. Shah Superintending Engineer &
Director Directorate of Irrigation Research &
Development, Pune

Date : 14/05/2009 Place : Pune

PREPARED BY :

- 1) D. R. Pawar
Soil Survey Officer, Soil Survey Division,
Pune-37.
- 2) D. B. Randhe
Dy. Soil Survey Officer, Soil Survey
Division, Pune-37.
- 3) Dr. Mehraj Shaikh (S.S.A.)
- 4) Smt. A. K. Korde (S.S.A.)
- 5) K. A. Shinde (R.A.)
- 6) Miss. M. P. Yadav (R.A.)
- 7) Miss. A. E. Chavan (R.A.)
- 8) Miss. V. M. Sapkale (R.A.)

SPECIAL THANKS :

- 1) D. R. Joshi
Asst. Engineer (II)
- 2) S. M. Amrutkar Retd. Soil Scientist
- 3) H. Mulani Soil Scientist

APPROVED BY :

Er. K. M. Shah
Superintending Engineer & Director Directorate of
Irrigation Research
& Development, Pune.

[illegible]

