INTRODUCTION TO SOIL AND WATER ANALYSIS SOILANALYSIS

Prior to planting, the soil should be tested for at least sodium, magnesium, calcium, potassium, and pH. If there are questions about the history of the soil, if existing vegetation shows odd symptoms and poor growth, or if other soils in the area have shown toxic levels of excess minor nutrients, more tests may be needed. Soil testing is now an intrinsic part of modern farming in the West, as well as in many developing countries. Tests primarily focus on the elements in most demand by crops which are supplied by fertilizers: nitrogen (N), phosphorus (P), and potassium (K). Depending upon the soil types, in some regions tests are also conducted for secondary nutrients: calcium (Ca), magnesium (Mg), and sulfur (S). In drier areas, micronutrients such as iron (Fe), zinc (Zn), manganese (Mn), copper (Cu), and boron (B) are often measured, since deficiencies of these elements are more frequently associated with calcareous soils. Indeed such areas may also have excessive or toxic levels of some elements, such as B, and high levels of elements such as Na and Mg, which can adversely affect soil physical properties. As nutrient behavior in soils is governed by soil properties and environmental conditions, measurement of such properties is often required. These include pH, salinity, organic matter (OM), calcium carbonate (CaCO3), and texture and aggregate stability. In drier areas, the presence of gypsum (CaSO4.2H2O) is also of concern.

Soil testing involves four distinct phases:

1. Sample Collection: This should be such that it reliably reflects the average status of a field for the parameter considered.

2. Extraction or Digestion and Nutrient Determination: The reagents used and the procedures adopted should extract and reflect all or a portion of the element in the soil which is related to the availability to the plant, i.e., it should be correlated with plant growth.

3. Interpreting the Analytical Results: The units of measurement should reliably indicate if a nutrient is deficient, adequate, or in excess (in some cases toxic to plants).

4. Fertilizer Recommendation: This is based upon the soil test calibrated for field conditions, and considers other factors such as yield target, crop nutrient requirement, management of the crop, soil type, and method of fertilizer application, etc.

WATERANALYSIS

Irrigation water should be tested for pH, electro-conductivity (EC), sodium, bicarbonate (HCO₃), sodium absorption ratio (SAR), chloride, boron, nitrate (NO₃), and any other suspected regional problem materials.

Water is essential for all aspects of life, domestic use, industrial processes, and agricultural production. Its suitability for a particular purpose can be determined by analysis. Obtaining good results depends to a great extent on the following factors: - Ensuring that the sample taken is truly representative of the water under consideration - Using proper sampling techniques - Protecting and preserving the samples until they are analyzed (in a timely manner) The water and wastewater should be checked for chemical, physical (turbidity and solid matter) and biological contamination before used it, to avoid environmental pollution (most of diseases can be spread to plant, animal, and human by water contaminated).

PURPOSE OF SOIL AND WATER ANALYSIS

PURPOSE OF SOIL ANALYSIS

A **soil test** can determine fertility, or the expected growth potential of the **soil** which indicates nutrient deficiencies, potential toxicities from excessive fertility and inhibitions from the presence of non-essential trace minerals. The **test** is used to mimic the function of roots to assimilate minerals.

PURPOSE OF WATER ANALYSIS

- 1. To know the quality of Irrigation water
- 2. To establish the Sustainability of certain types of crops
- 3. To know the Nature, Amount and Type of salts present in water
- 4. To recommend Suitable Measures to use the available water

METHODS FOR SOIL ANALYSIS

DETERMINATION OF MOISTURE PERCENTAGE:

Procedure:

Take soil sample in china dish. Place china dish in an oven at 105°C for 24 hours. Then take it from oven and cool it in desiccators.

Calculations:

Wt. of china dish = A gm

Wt. of china dish + paste = B gm

Wt. of paste = B-A = C gm

Wt. of china dish + paste (oven dried) = X gm

Wt. of paste (oven dried) = X - A = Y gm

Loss in wt. = C - Y = Z gm

Moisture % percentage = $\underline{\text{Loss in wt. on drying}} \times 100$ Oven dried wt.

DETERMINATION OF SATURATION PERCENTAGE

Procedure:

Take 100 g of soil sample in 250 ml plastic beaker. Take distilled water in the burette. Saturate the soil sample to reach the characteristics of saturated paste.

CHARACTERISTICS OF SOIL PASTE AND PRECAUTIONS FOR ITS PREPARATION:

- Soil paste should glisten and reflect light from the surface.
- Soil paste should slide freely from the spatula.
- Water should not stand on surface when placed for sometimes.
- When small is made on the surface with glass rod, water should not accumulate in it.
- Cover the beaker with lid for 30 minutes or one hour to reach equilibrium.

Note the reading on the burette that will be the saturation percentage.

Conclusion:

For example: For 100 gm of soil, 60 ml distilled water is used in preparation of saturated soil paste then saturation % age should be 60.

Criteria used for Saturation Percentage:

0-19%	Sandy (S)	Light
20-29%	Sandy Loam (S.L)	Medium
30-45%	Loam (L)	Medium
46-60%	Clay Loam (C.L)	Неаvy
>60%	Clay (C)	Heavy

DETERMINATION OF pHs:

(Electrometric Method)

<u>pH:</u>

It is negative log of hydrogen ion activity.

Buffer solutions of 4.0, 7.0 and 9.2 pH:

These can be prepared by dissolving the pH tablets in 100 ml boiled, cooled CO₂ free, distilled water separately.

Standardization of a pH meter:

Standardization of pH meter is different depending on the model. Usually standardization procedure is provided along with meter. However general procedure is as follow.

- Note soil paste temperature and adjust on pH meter.
- Dip electrode in solution of pH 4.0, adjust meter by knob.
- Then dip the electrode in the solution of pH 9.2, adjust the reading accordingly.
- Now dip the electrode in solution of pH 7.0, check whether reading is same or not.
- If pH 4.0 and 9.2 is correctly adjusted, pH meter is standardized and ready for pH determination of saturated soil paste.

Apparatus:

- pH meter
- Cylinder 25 ml
- Beaker plastic 250 ml
- Standard buffer solution

Procedure:

Take 25 g of air dried soil in plastic beaker, add 25 ml distilled water, stir with glass rod and leave it for overnight, So that maximum salts may be dissolved. After standardizing the pH meter, dip the electrode in the sample about 3cm deep and note the reading (with decimal) after 30 seconds. Wash the electrode with distilled water and dry it with tissue paper before dipping in new soil sample.

pH Range	Remarks
7.0-7.5	Normal, Soil contains no alkaline earth carbonates.
7.6-8.0	Slightly Alkaline
8.1-8.5	Alkaline, some quality of HCO ₃ is present. ESP major may not exceeds 15.
>8.5	Highly Alkaline, carbonate is dominant anion and ESP exceeds 15.

Criteria used for Soil pH:

DETERMINATION OF EC

Standardization:

To standardize the EC meter, 0.01N KCl solution is used. Its reading should be 1413 μ S/cm 0r 1.413 dS/m at 25°C.

Cell Constant:

Where K = C/D

K= Cell constant C= Known EC of saturated KCl solution D= Observed EC of saturated KCl solution <u>Units of EC:</u>

Its unit is mS/cm = dS/m Old unit: mmhos/cm. **Procedure:**

- Take 10 g soil in conical flask and add 100 ml distilled water.
- Shake it well and left it for overnight.
- Adjust the temperature and conductivity meter with the help of knob.
- Dip the cell into the sample and note the reading that is displayed on the EC meter screen.
- Reading will be in mmhos/cm.
- If the sample is very small either use micro-cell or dilute the sample
- Sometime salt concentration is too high to be measured, so make appropriate dilutions and multiply the final reading with the dilution factor (D.F).

Criteria used for Soil EC:

EC (dS/cm)	Remarks
<4.0	Normal
4.1-8.0	Medium salinity: yield of sensitive crops may be restricted.
8.1-16.0	High salinity: Only a few salt tolerant crops can grow.
>16	Very high salinity: Only a few tolerant crops can grow.

DETERMINATION OF AVAILABLE POTASSIUM FROM GIVEN SOIL <u>SAMPLE:</u>

Flame photometer:

Flame photometer is a device used in inorganic chemical analysis to determine the concentration of certain metal ions, among them sodium, potassium, lithium, and calcium.

Reagents / Medias Required:

<u>1N Ammonium Acetate (CH₃COONH₄) :</u>

Dissolve 77.08 g of Ammonium Acetate (CH₃COONH₄) in 800 ml distilled water and make the volume to 1 litre. Adjust pH 7.0 using either NH₄OH or HCl.

Method:

Weigh 2.5 g air dried, grind soil sample and pass it through 2 mm sieve. Add 50 ml extracting reagent 1N Ammonium Acetate (CH_3COONH_4). Shake on a flat-bed reciprocating shaker for 30 minutes and filter the extract. Determine K by flame photometer in ppm using graph readings.

Calculation Required:

Extractable K (ppm) = Reading (ppm) \times 20

Criteria used for K:

Range	Remarks
<80 ppm	Low
81-180 Ppm	Medium
>180 ppm	High

DETERMINATION OF AVAILABLE PHOSPHORUS FROM SOIL SAMPLE:

(Olsen's Method, 1954)

This method estimates the relative bioavailability of ortho-phosphate (PO4-P) in soils by extraction using alkaline sodium bicarbonate (pH 8.5) solution and determining the P concentration in the extract. It is applicable to soils that are mildly acidic to alkaline pH and is based on the method developed by Olsen et al., (1954) to correlate crop response to fertilizer on calcareous soils (the soils mostly or partly composed of <u>calcium carbonate</u> e.g. containing <u>lime</u> or being <u>chalky</u>.)

Reagents/Medias Required:

A) Sodium bicarbonate Solution, (Extracting Reagent) 0.5M NaHCO3:

Dissolve 42.0 g NaHCO₃in appropriate 700 ml distilled water, shake well and make the volume 1000 ml. Adjust pH 8.5 using 5N NaOH.

5N NaOH = dissolve 200 g NaOH in distilled water and let it cool and make volume 1 litre.

B) Mixed Reagents:

a) <u>Ammonium HeptaMolybdate</u> 4.8% (NH₄)₆Mo₇O₂₄.4H₂O (f.wt.1235.9)

Dissolve 12.0 g in distilled water and volume 250 ml.

b) <u>Potassium Antimony tartrate</u> (KsbO.C₄H₂O₆):

Dissolve 0.291 g in distilled water and make the volume 100 ml.

- c) <u>5N H₂SO₄</u>. Dilute 140 ml concentrated Sulphuric Acid (H₂SO₄), (in fume hood) in distilled water, let it to cool and make the volume 1 litre.
- **d**) Add both the dissolved reagents (a+b) in 1000 ml 5N H₂SO₄ and make volume 2000 ml with distilled water. Store in a Pyrex bottle in a dark, cool place.

C) Colour Developing Reagent:

Weigh 0.528 g ascorbic acid to 100 ml of mixed reagent. This reagent should be prepared freshly as required because it does not give accurate results after 24 hours.

D) Stock solution (1000ppm):

Dissolve 4.3937 g Potassium Di-hydrogen Phosphate (KH₂PO₄) in distilled water and make volume to 1 litre.

E) Stock solution (100ppm):

Take 10 ml of Phosphorus CRM and make upto 100 ml, which is 100 ppm.

F) Standards (5-20ppm):

Make standards of different concentration i.e. 5 ppm, 10 ppm, 15 ppm and 20 ppm from stock solution by using the formula; $C_1V_1=C_2V_2$

Procedure:

Weigh 5 g air dried and ground soil, add 100 ml extracting solution. Shake for 30 minutes and filter with what man No.42. Pipette out 5 ml aliquot. Add 5ml of colour developing reagent, in 25 ml volumetric flask. Shake to remove gas bubbles. Let stand for 15 minutes. Make volume up to mark. Bluish colour will develop.

Concentration of phosphorus in soil is directly proportional to the intensity of blue colour developed. Take reading on concentration mode at 880 nm wavelength on Spectrophotometer.

Calculation:

Extractable Phosphorus in soil (ppm) = P ppm $\times \underline{A \times 5}$ Wt. \times Vol.

A = Total volume of Extractant (ml)

B = weight of air dry soils (g)

V = Volume of extract used (ml)

DETERMINATION OF ORGANIC MATTER:

(Chromic Acid Method)

Chemistry:

During analysis, soil organic matter (Organic matter) is oxidized with excess chromic acid and that portion of chromic acid not used in the oxidation is determined by titration against Ferrous Sulphate.

Reagents for Organic matter determination:

1. <u>Reagent of K₂Cr₂O₇ Potassium Di-chromate (1N)</u>

Take 49.04 g of $K_2Cr_2O_7$ and dissolve in 1 litre of distilled water.

2. <u>Iron Sulphate FeSO_{4.6H2}O: (Titrant for O.M determination)(1N)</u>

Take 278.02 g of Ferrous Sulphate (FeSO₄.6H₂O) in distilled water and add 15ml concentrated H_2SO_4 and dissolve with distilled water to make the volume up to 1 litre.

3. <u>Sulphuric Acid (H₂SO₄):</u>

Commercial H₂SO₄ is used.

4. Phosphoric Acid (H₃PO₄) 85% or Sodium fluoride (NaF)

5. <u>Barium diphenylamine Sulphonate Indicator, (0.16%):</u>

Dissolve 0.5 g barium diphenylamine as indicator in 100 ml concentrated H_2SO_4 and 20 ml distilled water.

Procedure:

Weigh 1.0 g air dried, ground soil sample into 500 ml conical flask, Add 10 ml K₂Cr₂O₇and mix well. Add 20 ml sulphuric acid and mix it. Allow the flask to stand for 30 minutes to cool. Then add 180 ml distilled water. Add0.5 g NaF (sodium fluoride), 5-10 drops of di-phenyl amine indicator and finally titrate against standardized ferrous sulphate to sharp green end point from violet blue colour. Run a blank and subtract sample reading from blank to get the actual volume of ferrous sulphate used to reduce Potassium Di-chromate.

CALCULATIONS:

% O.M = $\underline{ml \text{ for blank} - ml \text{ for sample}} \times 0.698$ Weight of sample (g) % O.M = $\underline{mEq. of K_2Cr_2O_7 reduced} \times 0.698$ Weight of sample (g)

$$\begin{split} mEq. \ of \ K_2Cr_2O_7 \ reduced &= (mEq \ of \ K_2Cr_2O_7 \ added - mEq \ of \ FeSO4 \ used) \\ mEq \ of \ K_2Cr_2O_7 \ added &= (ml \ of \ K_2Cr_2O_7 \times N) \\ mlof \ FeSO4 \ used &= (ml \ of \ FeSO4 \ used \times N) \end{split}$$

% O.C = <u>ml for blank - ml for sample</u> ×N FeSO4 × 0.337 Weight of sample (g)
% O.C = % Organic matter/ 1.724
% O.M = % O.C × 1.724

Note:

 $0.698 = 0.003 \times \frac{100}{74} \times \frac{100}{58} \times 100$

How can be measured as 0.003:

If 1 me of O.M in soil = $1 \times \text{Eq.wt.}$ of C /1000

Equivalent wt. of C = $\underline{m.wt.} = \underline{12} = \underline{3} = 0.003$ g of C of O.M in soil Valency 4/1000 1000

1 ml of N $K_2Cr_2O_7$ when reduced = 0.003 g of C of O.M in soil

100 / 74 = 1.3514, efficiency factor for determining O.C of soil

100 / 58 = 1.724, for conversion of % O.M of soil to % O.C (If divided)

100 =for converting O.M in percentage 0.337

Walkley's rapid method (1935, 1947) for the determination of Organic Carbon in soil has been found to give approximate 89% recovery of carbon as compared to dry combustion method.

The conversion factor 0.337 was obtained by dividing 0.003, the milli-equivalent weight of carbon by 89 and multiply by 100 to convert to percent.

Criteria used for Organic Matter %:

< 0.86	Poor
0.87–1.29	Satisfactory
>1.29	Adequate

DETERMINATION OF SOIL MICRO-NUTIENTS

Apparatus:

- Atomic Absorption Spectrophotometer with all accessories.
- Volumetric flasks 2 litre
- pH Meter
- Weighing Balance
- Mechanical Shaker
- What man No.42 filter paper

Reagents:

- DTPA (Diethylenetriaminepentaaceticacid) extracting solution 0.005M.
- DTPA 0.005M: 3.934 gm dissolved in 2 litre flask.
- CaCl₂ 0.01M 2.94 gm dissolved in 2 litres.
- TEA (Tri-ethylene Amine) 0.01M 26.64 ml in 2 litre flask add approximately 800 ml distilled water and adjust pH 7.3 with the help of pH meter by using 1:1 HCl solution.

Procedure:

Weigh 20 gm of Soil and add 40 ml DTPA extracting solution then a continuous shaking for 2 hours on mechanical shaker. Keep it overnight and filter it with what man No.42 filter paper. Standard solution used for Cu 0.5, 1.0, 2.0 and 4.0 and for Zn 0.5, 1.0 and 2.0 ppm.

While for Mn and Fe standard solutions are of 1.0, 2.0, 4.0 and 8.0 ppm. Then, take reading of sample after completing the graph of standard.

Elements	Range	Remarks
Zn	<1.0 ppm	Poor
	>1ppm	Adequate
Fe	<4.5ppm	Poor
	4.5-6.5 ppm	Satisfactory
Cu	<0.4ppm	Poor
	>0.4ppm	Adequate
Mn	<2.0pm	Poor
	>2.0ppm	Adequate
В	(By Spectrophotometer)	

<u>Critical Limits for Soil:</u>

<0.5ppm	Poor
>0.5ppm	Adequate

DETERMINATION OF BORON IN SOIL:

REAGENTS/MEDIAS REQUIRED:

A. Buffer Solution:

Dissolve 250 g ammonium acetate and 15 g of Ethylene Di-amine Tetra Acetic Acid (EDTA) in 400 ml of distilled water. Slowly add 125 ml glacial acetic acid and mix.

B. Azomethine-H reagent:

Dissolve 0.45 g of reagent in 100 ml of 1 % L-ascorbic acid solution. Fresh reagent should be prepared and store in a refrigerator.

C. Boron Standard Solution:

Dissolve 5.709 g of Boric acid in distilled water (for hot water extraction) or in 0.05 M HCl for HCl extraction and adjust the volume of 100ppm B. Then make working standard as desired.

 $C_1V_1 = C_2V_2$

 $100\times V1=0.5\times 100$

 $V1 = \frac{E \times 1}{E} = 0.5$ ml of sub-stock solution to make 0.5ppm B working standard.

METHOD

1. HOT WATER EXTRACTION

Boil 10g soil in 20ml distilled water for 5 minutes in boron free apparatus (Polypropylene) and then filter. If boron free apparatus is not available then Pyrex glass apparatus should be dipped in concentrated HCl for a week and then used.

2. HCL EXRACTION

10 grams soil was shaken with 20ml of 0.05 M HCl for 5 minutes and then filter. Transfer 1 ml aliquot in 50 ml polypropylene volumetric flask, add 2 ml buffer solution, and add 2 ml Azomethine- H reagent and mix. After 30 minutes read absorbance at 430 nm on spectrophotometer at concentration mode or by graph using standards are prepared in 0.05 M HCl extraction and in distilled water for hot water extraction using 420 nm wavelengths determine boron (ppm) by spectrophotometer using colour developing method.

CALCULATION

B (ppm) = $R \times 100*$ Where R = Spectrophotometer reading

 $100^* =$ Total dilution factor.

Criteria for Boron:

<0.2	Weak
0.2-0.5	Satisfactory
0.5 – 1.0	Adequate

METHODS FOR WATER ANALYSIS

SAMPLING OF IRRIGATION WATER

Irrigation water:

The water used for agriculture purposes is sometimes known as Irrigation water.

Irrigation water quality:

The concentration and composition of dissolved salts in any water determine its quality for irrigation. Mostly the concerns with irrigation water quality relate possibility of high salt concentration (sodium, carbonates, bicarbonates and toxic ions Br-, Cl-).

Purposes:

- 5. To know the quality of Irrigation water
- 6. To establish the Sustainability of certain types of crops
- 7. To know the Nature, Amount and Type of salts present in water
- 8. To recommend Suitable Measures to use the available water

Storage of sample

About 1 litre of water is sufficient for irrigation purpose analysis. Sample is put in clean bottle. Do not wash the bottle with any detergent. Sample should be analysed within seven days.

Labelling of Sample:

Label should indicate:

- Date of collection
- Name of person (Collector/Farmer)
- Depth of sample

DETERMINATION OF EC OF WATER

Electrical Conductivity (EC):

Electrical Conductivity is a measure of water's capability to pass electrical flow. This ability is directly related to the concentration of ions in the water ¹. These conductive ions come from dissolved salts and inorganic materials such as alkalis, chlorides, sulphides and carbonate compounds.

Standardization:

To standardize the EC meter we use 0.01 N KCl Solution and its reading should be 1413 μ S/cm or 1.413 dS/m at 25°C.

Cell Constant:

Where K = C/D

K= Cell constant

C= Known EC of saturated KCl solution

D= Observed EC of saturated KCl solution

Criteria for EC:

0-1000 µS/cm	Fit
1001-1250 µS/cm	Marginally fit
>1250 µS/cm	Unfit

TDS or TSS:

Dissolved solids" refer to any minerals, salts, metals, cations or anions dissolved in water. Total dissolved solids (TDS) comprise inorganic salts (principally calcium, magnesium, potassium, sodium, bicarbonates, chlorides, and sulphates) and some small amounts of organic matter that are dissolved in water.

Calculation of TDS:

TDS (mEq/L) = Reading of EC (ms/cm) $\times 10$

TDS (ppm) = Reading of EC (ms/cm) \times 640

Or

TDS (ppm) = Reading of EC (μ S/cm) × 0.64

REAGENTS FOR WATER ANALYSIS

1. <u>NH₄Cl+NH₄OH (Buffer Solution) (10drops for Ca⁺⁺ + Mg⁺⁺)</u>

Dissolve 67.5g of NH₄Cl in 570 ml of concentrated NH₄OH and makes the volume to 1 litre.

2. <u>EDTA (Di-Sodium salt of Ethylene Di-amine Tetra Acetic acid) 0.01 N</u> (For Ca⁺⁺ + Mg⁺⁺ as used titrant)

Dissolve 1.86g of Na2-EDTA in distilled water and make the volume up to 1 litre.

Molarity of Na2-EDTA= 372.24 g/mol

Equivalent weight = molarity/ionisable proton = 372.24/2 = 186.12

For 1N EDTA soln. = 186.12 g/L

For 0.01 N EDTA soln. = $186.12 \times 0.01 = 1.86$ g/L

Standardize this solution against standard CaCl₂.2H₂O solution (0.01N).

CaCl₂ (0.01N): (To standardize the EDTA)

Dissolve 0.55 g of pure $CaCO_3$ in 10 ml of approximately 3N (1+3) HCl and make the volume up to 1 litre.

Molecular weight 40+71=111/2 = 55.5

For 1N solution CaCl₂ dissolve 55.5 g in 1 litre.

For 0.01N = 55.5×0.01 =0.555g

3. <u>EBT (Eriochrome Black-T) indicator (for Ca⁺⁺ + Mg⁺⁺)</u>

Take 0.4 g of EBT and 4.5 g hydroxyl amine in 100 ml of 95% Ethanol.

4. <u>AgNO₃ (Silver Nitrate) (Titrant for CI)</u>

Take 8.5g (0.05N) of AgNO₃ and with distilled water make volume up to 1 litre. Standardize against 0.05 N NaCl solution

108+14+48 = 170 (For 1N)

For $0.05N = 170 \times 0.05 = 8.5g$

5. <u>Phenolphthalein Indicator: (for CO₃²⁻)</u>

Dissolve 1g of Phenolphthalein indicator in 100ml of 50% Ethanol.

6. <u>H₂SO₄ (0.1N) Titrant for CO₃²⁻and HCO₃⁻</u>

- a) % Purity (98%)
- b) Specific gravity (1.84)
- c) Equivalent weight (49g)

Normality (1N) = $\frac{E}{S} \frac{w}{g} \times \% a p$ Normality (1N) = $\frac{4}{1.8 \times 9/1} = \frac{4}{1.8 \times 0.9} = 27.17$ ml

So, Take 27.17ml of H₂SO₄ and make volume upto 1 litre to prepare 1N H₂SO₄ solution.

For 0.1N H₂SO₄ then multiply 27.17 with 0.1N that will be,

27.17×0.1= 2.717 ml (for 1 litre)

DETERMINATION OF SOLUBLE CATIONS IN WATER SAMPLE:

Total Hardness:

The simple *definition of water hardness* is the amount of dissolved calcium and magnesium in the *water*.

1. <u>Calcium and Magnesium (Ca⁺⁺ + Mg⁺⁺)</u>

Principle:

The principle of this method is Complexometric Titration. Calcium and magnesium can be determined by titrating it against Disodium salt of EDTA (Disodium salt Ethylene Di-amine Tetra Acetic Acid) in the presence of Buffer Solution using EBT (Erio-Chrome Black-T) as an indicator and indigo/blue is the end point.

Reagents:

- Buffer Solution NH₄Cl-NH₄OH
- EBT (Eriochrome Black-T indicator)
- EDTA 0.01 N

Procedure:

Take 10 ml water sample with the help of pipette in a conical flask. Add 10-12 drops of buffer solution and few drops of EBT as an indicator. Titrate it against standardized 0.01 N EDTA solutions till indigo/blue end point appear.

Calculation:

 $Ca^{2+} + Mg^{2+} (as mEq/L)_{=} \underline{ml of EDTA used \times Normality of EDTA}_{Vol.of sample} \times 1000$

2. Sodium:

It is determined by Flame Photometer; Na^+ can also be determined by subtracting $Ca^{++} + Mg^{++}$ from EC value.

 $Na^{+}(mEq/L) = EC (mEq/L) - (Ca^{++} + Mg^{++} mEq/L)$

3. Potassium (K⁺):

It is determined by flame photometer.

DETERMINATION OF SOLUBLE ANIONS IN WATER SAMPLE:

1. <u>Carbonates (CO₃²⁻)</u>

It can be determined by titrating it against standard H_2SO_4 using phenolphthalein as an indicator to a colourless end point.

Reagents:

- Phenolphthalein indicator
- H₂SO₄ 0.1 N (0.05M)

Procedure:

Take 50 ml of water in a conical flask; add 2-3 drops of Phenolphthalein. If no colour then CO_3^{2-} are absent. If the solution turns pink then carbonates are present, then titrate the contents of flask against 0.1 N H₂SO₄ taken in a burette to a colourless end point.

Units:

milliequivalent/Litre (mEq/L)

milligram/Litre (mg/L) or ppm

Chemical Reaction:

This reaction is completed in two steps as shown by the chemical reaction

$$\begin{split} Na_{2}CO_{3} + H_{2}SO_{4} -----i \\ 2 \ NaHCO_{3} + H_{2}SO_{4} -----i \\ Na_{2}SO_{4} + 2 \ H_{2} \ O+ 2 \ CO_{2} -----i \\ \end{split}$$

R1 is the amount of acid used to convert the carbonates to bicarbonates (equation i, phenolphthalein part of reaction, while for the completion of the reaction, same amount of acid will be still needed to further convert bicarbonates so produced during step i to form CO_2 and H_2O as shown in equation ii. Thus R_1 is the amount of acid for half reaction and $2R_1$ will represent the amount of complete reaction. Hence R_1 is multiplied by a factor of 2.

Calculation:

 $CO_3^{2^-}=2R_1 \times N \text{ of } H_2SO_4 \times 1000$ Volume of sample taken R1= Amount of H2SO4 used $CO_3^{2^-}=R1 \times 4$

2. <u>Bicarbonates (HCO₃):</u>

It can be determined by titrating it against standard H_2SO_4 using methyl orange as an indicator to a pinkish end point.

Reagents for (HCO₃):

- Methyl Orange
- $H_2SO_4(0.1N)$

Procedure:

For the determination of bicarbonates to the same sample (50 mL) after titrating CO_3^{2-} , the indicator "methyl orange" is used and titrates against standardized 0.1N H₂SO₄ solution until the colour changes from golden yellow to light pinkish end point.

Chemical Reaction:

Bicarbonates react with H2SO4 to form CO2 gas and water. The chemical equation is given below:

 $2HCO_3^- + H_2SO_4 \rightarrow 2CO_2 + 2H_2O + SO_4^{2-}$

R2 is the total amount of acid used to neutralise the bicarbonates present in the sample after titration. Which means that sample contains bicarbonates coming from conversion of CO3 as well as originally present in the sample. Therefore the actual amount of acid used for neutralising the originally present HCO3 would be R2-R1.

Units:

milliequivalent/Litre (mEq/L)

milligram/Litre (mg/L) or ppm

Calculations:

 $\begin{array}{l} HCO_{3}^{-} \ (mEq/L) = (\underline{R2-R1}) \times N \ of \ \underline{H_2SO_4} \times 1000 \\ Vol. \ of \ sample \\ HCO_{3}^{-} = (R_2-R1) \times 2 = ... \ mEq/L \end{array}$

 $R1 = ml \text{ of } H_2SO_4 \text{for } CO_3^{2-}$

 $R2 = ml of H_2SO_4 for HCO_3$

3. Chloride (Cl⁻):

Chloride determination is performed by titration with silver nitrate (AgNO3) as titrantand using Potassium Chromate as an indicator. This method is known as Mohr method.

Reagents:

- AgNO₃ (0.05N or 0.05M)
- Potassium Chromate (5% Solution)

Procedure:

For the determination of Chlorides to the same sample after titrating HCO_3^- , the indicator Potassium Chromate is used and titrate against AgNO₃ (0.05N) solution until the colour changes from golden yellow to brackish red.

Chemical Reaction:

AgNO₃ reacts with chloride ions in the presence of H_2SO_4 (optimum pH for this reaction is 3.0) to yield AgCl (white ppt).

AgNO₃+ Cl⁻ \rightarrow AgCl (white ppt) + NO₃

When all the Cl ions consumed then Ag of AgNO3 will start to react with Potassium chromate and yield Silver chromate which have brick red colour. So brick red will be the indication of the completion of reaction.

 $2AgNO_3 + K2CrO4 \rightarrow Ag_2CrO_4$ (brick red ppt) + $2KNO_3$

So, brick red colour is the endpoint.

Units:

Milliequivalent/Litre (mEq/L)

Milligram/Litre (mg/L) or ppm

Calculation:

 Cl^{-} (mEq/L) = <u>ml ofAgNO₃ ×N of AgNO₃</u> ×1000 Volume of sample taken

Or

 Cl^{-} (mg/L or ppm) = <u>ml of AgNO₃ × Molarity of AgNO₃ × Mol. Wt of Cl</u> × 1000 Volume of sample taken

(Mol. Wt of Clis 35.45)

4. <u>Sulphates(SO₄²⁻):</u>

SO₄²⁻is usually determined by subtracting sum of anions from TSS (EC).

 $SO_4^{2-}(mEq/L) = TSS (mEq/L) - (CO_3^{2-} + HCO_3^{-} + Cl^{-})$

Determination of Residual Sodium Carbonate (RSC):

The RSC index is used to find the suitability of the water for <u>irrigation</u> in <u>clay</u> soils which have a high <u>cation exchange capacity</u>.

RSC can be calculated by the formula as follows;

 $RSC = (CO_3^{2^-} + HCO_3^-) - (Ca^{++} + Mg^{++}) mEq/L.$

RSC Criteria:

Range	Remarks
<1.25	Fit
1.25-2.5	Marginally Fit
>2.5	Unfit

Determination Of Sodium Adsorption Ratio (SAR):

The Sodium adsorption ratio (SAR) is an irrigation water quality parameter used in the management of sodium-affected soils. It is an indicator of the suitability of water for use in agricultural <u>irrigation</u>, as determined from the concentrations of the main alkaline and earth alkaline cations present in the water.

SAR can be calculated by following formula;

$$SAR = \frac{Na + \sqrt{\frac{C + M}{2}}}{\sqrt{\frac{C}{2}}}$$

The concentration of Na^+ and $Ca^{++} + Mg^{++}$ is taken in mEq/L. SAR Criteria:

Range	Remarks
<6	Fit
6-10	Marginally Fit
>10	Unfit

FEE CHART OF SOIL AND WATER ANALYSIS:

