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SOIL AND WATER ANALYSIS MANUAL

This Soil and Water Analysis Manual achieves the objective of implementing the standard approved procedures for all divisional and district soil and water testing laboratories working under the command of the Rapid Soil Fertility Survey and Soil Testing Institute Punjab, Lahore. Both Divisional Agricultural Chemists and District Assistant Agricultural Chemists have made the brain storm these protocols. All these chemists have also been trained for two days to become acquainted with all the methods, principles and calculations involved. Even a very thorough compilation has been made in writing of these methods, if any recent developments or flaws or faults are revealed, the opinion will be highly appreciated.

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SOIL AND WATER ANALYSIS MANUAL

BASIC GUIDELINES FOR LABORATORY ACTIVITIES

Real knowledge and interpretation of every context of study relies on a diligent correlation of facts and logical conclusions from them. Efficient laboratory work will give you unique and stimulating opportunities to learn many details about soil science. Out of such scientific insights, scientists have established standards that unify the disciplines of soil science and the concepts that offer existing interpretations. This is the scientific approach to be used. Here are a few tips that will help you get an outstanding start to your laboratory work.

- 1.** Read carefully safety precautions and laboratory rules and obey them. These will help you to safeguard not only your interest but also those of other research workers working in the laboratory.
- 2.** Each equipment used in the chemical laboratory is designed for a specific purpose. First become familiar with the same and then its purpose. For example, a graduated cylinder is intended to be used as a device for measuring the volume of a liquid and not as a reaction vessel. The objects which you shall use are shown on the latter pages. Try to learn their names.
- 3.** Before you come to the laboratory, thoroughly read the analysis procedure. Laboratory work is of much greater value and you can make more efficient use of your time if you are properly oriented with procedure.
- 4.** Before starting the analysis make sure that all the equipment's are properly washed and conveniently arranged.
- 5.** Gain self-reliance by working alone in the laboratory except when directed otherwise.
- 6.** Talk over the analysis to get more precision research workers.
Scientists learn much by discussion with each other.
- 7.** Keep your work bench and locker in a good order so that you can easily find what you need. If a substance is spilled, clean it up immediately. If you don't, your clothes, skin, books or papers may be damaged or ruined.
- 8.** Use care in handling laboratory materials.
- 9.** In all analytical work you should use distilled water.
- 10.** Be economical and specific in the use of all reagents, being costly items also become familiar with its appearance and properties.

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* = X = core sampling positions

B. SAMPLING IN FIELDS.

DIVISION OF FIELD INTO AREA FOR SAMPLING

If soils are fairly uniform , an area upto 10 acre can be sampled as a unit. In case of variation, field can be subdivided into uniform sampling units and each sampled separately in the form of composite sample.

SOIL SAMPLING FOR VEGETABLE FIELDS

As vegetables are grown in fertile soil and root remain in furrow slice. So the depth of 0-15, 15-30 and 30-45 cm is sufficient for soil fertility evaluation.

SOIL SAMPLING FOR FIELD CROPS

As roots of crops like wheat, rice, cotton and maize etc. go deeper in sub-soil so it is necessary to get knowledge about sub soil nutrient status and concretions. Sampling of soil for this purpose depends on the type of crop grown and study required. Soil sampling should be done upto 0-15 , 15-30 , 30-45, 45-60 and 60-90 cm depth.

SOIL SAMPLING FOR ORCHARDS/GARDENS

In case of fruit trees samples should be taken at the distance not less than three feet from the trunk of plant or under the crown of tree. As root system is more extensive than crops sampling should be done upto the depth of 150 cm to evaluate nutrient status and underlying properties and water logging. Sampling should be done as follow 0-15,15-30, 30-45,45-60,60-90,90-120 and 120-150 cm depth.

Carefully note the concretions especially. If more that 20%, it will hinder nutrient availability and free penetration of roots through the soil. In case of sandy soil it will not have positive effect on fruit trees from nutrient supplying point of view.

PRECAUTIONS FOR SOIL SAMPLE COLLECTION

Samples should not be taken:

1. Along the water channel, roadside, pathway and boundaries

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2. Under the tree shade.
3. Close to the heaps (manure).
4. Areas where plant population is not uniform.
5. Areas where domestic refuse are thrown.
6. Places in the field where fertilizers are dumped.
7. Places where organic matter or plant residues have been accumulated by natural agencies like wind or water, sampling should be avoided especially for fertility evaluation.
8. For salinity evaluation, if salt crust on the surface is present it should be removed before soil sample collection or it should be sampled separately.

PRREPARATION OF COLLECTED SOIL SAMPLES

1. BAGGING. Samples are placed in polyethylene bags and then in cloth bags.

2. LABELLING. Labelling should be made with lead pencil or water proof ink.

One small label should be placed inside the bag and same number should be written on the bag.

3. DRYING. Soil samples after collection should be brought to the laboratory. Dry under shade and where fumes are not present. Higher temperature should be avoided as reversible change may take place.

4. SIEVING. Air dried soil should be ground and then pass through a sieve of 2mm mesh opening for general purpose and 100 mesh (0.45mm) for trace elements and for other critical analysis.

5. STORAGE. Soil samples, after passing through 2mm sieve, should be transferred into wide mouthed screw topped, numbered plastic bottles/jars.

6. Before analysis samples must be uniformly mixed.

REFERENCE / RELATED DOCUMENTS:

Soil and Plant Analysis Laboratory Manual ICARDA, Syria, 2001

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WATER SAMPLING

1. PURPOSE.

To assist the quality of irrigation

2. EQUIPMENTS/APPARATUS REQUIRED:

Plastic bottles

3. REAGENTS/MEDIAS REQUIRED:

4. METHOD:

The minimum quantity of water needed for the chemical analysis is about half liter. Obtain a representative sample. Collect sample from pipe after the pump has been running for some time. In general, shorter the elapsed time between collection and analysis of a sample of a sampler, the more reliable will be the analytical data. Changes resulting from chemical and biological activity may alter the composition of the sampler

5. CALCULATIONS REQUIRED:

6. CAUTIONS / SAFETY REQUIREMENTS:

Bottle should be clean, never wash bottle with soap. Give detail of tube-well and along with location and depth.

7. REFERENCE / RELATED DOCUMENTS:

Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington.

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CHAPTER-2

SOIL ANALYSIS

DETERMINATION OF ELECTRICAL CONDUCTIVITY (EC_E) OF SOIL

1. PURPOSE:

Determination of Electrical Conductivity (EC_e) of Soil

2. EQUIPMENTS/APPARATUS REQUIRED:

Conductivity Meter, beaker, suction pump or filter press

3. REAGENTS/MEDIAS REQUIRED:

0.01 N KCl: Dissolve 0.7456 g KCl in 1 litre of distilled water.

4. METHOD:

SATURATION EXTRACT:

Saturation extract from the soil paste can easily be obtained with the help of suction pump using negative pressure (pressure is applied from below the soil paste) of 1 atmosphere. However, if the soil is highly sodic and contains high amount of sodium and clay, saturation extract can not easily be obtained by the negative suction. For such soils filter press apparatus (pressure from above the paste) with positive pressure of any desirable magnitude can be used.

PROCEDURE:

Adjust moist filter paper in side the funnel, add paste into it, place bottle below the funnel, apply vacuum to collect the extract.

In case of filter press, place a moist filter paper in stainless steel cup, add paste to it, closed and air tight the cup, apply pressure from above the paste gradually till all the extract is collected in the bottle placed under the cup.

DETERMINATION OF ELECTRICAL CONDUCTIVITY OF SATURATION EXTRACT (EC_e)

PROCEDURE:

Note and adjust the extract temperature on the conductivity meter with the help of knob provided. Wash the conductance cell with distilled water and then with extract (if extract is in reasonable amount). Dip the cell into the extract making sure that there is no air bubble in it. Conductivity corrected at 25 °C will be displayed in mS cm⁻¹ or dS m⁻¹.

If the sample is very small either use the 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.

SOIL WATER RATIO:

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In case of saline sodic or sodic soils, to get a sufficient amount of extract is difficult. In such cases soil: water ratio of 1:2, 1:5 or 1:10 should be used.

5. CALCULATIONS REQUIRED:

$$\text{Dilution Factor (d.f)} = \frac{V_f \text{ (total volume of diluted solution)}}{V_i \text{ (volume of aliquot taken for dilution)}}$$

CONVERSION OF 1:10 TO ECe

Divide reading of 1:10 EC by 1.33 to convert into ECe

So $EC_1 (1:10) / 1.33 = EC_2$ i.e ECe

6. CAUTIONS / SAFETY REQUIREMENTS:

- 6.1 Standardise the conductivity meter before analysis
- 6.2 Calibrate the instrument with 0.01 N KCl solution. The EC of this standard solution is 1.413 dS m⁻¹ at 25 °C. The cell constant (K) can be calculated by the formula.

$$K = \frac{1.413 \text{ dS m}^{-1}}{\text{Observed reading in dS m}^{-1}}$$

- 6.3 The cell constant is necessary when 0.01 N KCl solution gives an EC values different from 1.413 dS m⁻¹. In this case, samples readings are multiplied by the cell constant.

7. REFERENCE / RELATED DOCUMENTS:

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C. pp. 89-91.

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DETERMINATION OF SOIL REACTION (pH)

1. PURPOSE:

Determination of soil reaction (pH)

2. EQUIPMENTS/APPARATUS REQUIRED:

Plastic beaker

Spatula

pH meter

3. REAGENTS/MEDIAS REQUIRED:

Standard buffer solutions of pH 7.01 and 10.01

-Deionized water

4. CALIBRATION OF pH METER

- i. Calibrate the pH meter using at least two buffer solutions of different pH values, usually 7.01 and 10.01. First, measure the temperature of the solution and adjust the temperature knob, if not auto adjust.
- ii. Dip the combined electrode in pH 7.01, buffer solution, check the actual pH at measured temperature, and adjust with the buffer knob. Then dip the combined electrode in the pH 10.01 buffer solution and adjust with sensitivity knob. Repeat until pH meter gives correct reading of both buffers.

PROCEDURE:

Give over night stay to the soil paste sample so that it may be stabilized. Rejuvenate the paste next morning if desired. Dip the electrode in the paste, note the reading when it is stabilized. Wash the electrode with distilled water and dry with tissue paper before dipping in new soil paste sample.

5. CAUTIONS / SAFETY REQUIREMENTS:

5.1 After completion of work, wash and immerse the electrode in distilled water or in saturated KCl solution.

6. REFERENCE / RELATED DOCUMENTS:

Schofield, R. K. and A. W. Taylor. 1955. The measurement of soil pH. Soil Sci. Soc. Amer. Proc. 19:164-167.

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DETERMINATION OF SOIL SATURATION PERCENTAGE

1. PURPOSE:

Determination of Soil Saturation Percentage

2. EQUIPMENTS/APPARATUS REQUIRED:

Balance
 China Dish
 Spatula
 Plastic beaker
 Oven

3. METHOD:

PREPARATION OF SATURATED SOIL PASTE

Take desirable amount of sieved soil in a plastic beaker (500 ml capacity), add distilled water gradually while continuously stirring with spatula till the desirable paste is prepared. Note the following points.

1. Soil paste should glisten and reflect light from the surface.
2. Soil paste should slide freely from the spatula.
3. Water should not stand on the surface when placed for some time.
4. When small hole is made on the surface with glass rod, water should not accumulate in it and hole so made will be filled with paste under slow gravity movement.
5. Cover the beaker with lid for 30 minutes or one hour to reach equilibrium.

If the saturated soil paste fulfils the above points, it will be ready for the determination of different parameters.

4. CALCULATIONS REQUIRED:

Weight of china dish = M g

Weight of china dish + paste = U g

Weight of paste = U - M = S g

Place the china dish in an oven at 105 °C for 24 hours. Remove and cool in a desiccator until constant dry weight.

Weight of china dish + paste (oven dried) = T g

Weight of paste (oven dried) = T - M = A g

Loss in weight = S - A = F g

Saturation percentage = $F/A \times 100$

or

$$= \frac{\text{Loss in weight of soil}}{\text{Oven dry weight of soil}} \times 100$$

5. CAUTIONS / SAFETY REQUIREMENTS:

Soil paste and tarred china dish is placed in an oven at 105 °C for 24 hours, remove, cool and record the weight. Same china dish with paste is again placed in an oven at 105 °C for some time,

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remove, cool and weight is recorded. If there is no difference in previous and new weight, then it will be constant oven dry weight.

6. REFERENCE / RELATED DOCUMENTS:

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DETERMINATION OF CARBONATES AND BICARBONATES IN SOIL

1. PURPOSE:

Determination of Carbonates and Bicarbonates in soil

2. EQUIPMENTS/APPARATUS REQUIRED:

- Pipette, 50 ml
- Conical flask, 100ml
- Cylinder, 50 ml
- Wash bottle
- Burette, 50 ml

3. REAGENTS/MEDIAS REQUIRED:

- A. Phenolphthalein 1%: Dissolve 1.0 g phenolphthalein in 100 ml ethanol with constant stirring. Filter if needed.
- B. Methyl orange 0.1%: Dissolve 0.1 g methyl orange in 100 ml distilled water.
- C. Sulphuric Acid 0.1 N (standardised).

4. METHOD:

Pipette out 50 ml aliquot in conical flask, add 1-3 drops of reagent A. If no colour appears, carbonates are absent. If pink colour appears, carbonates are present. Titrate it against 0.1 N H₂SO₄ to a colourless end point. Take three readings. Reserve the flask along with its contents for bicarbonates determination.

To the same conical flask (after colourless endpoint), add 1-2 drops of methyl orange, titrate against 0.1N H₂SO₄ from golden yellow to a light pink or light orange colour endpoint. Take three readings. Reserve the flask for chloride determination.

5. CALCULATIONS REQUIRED:

$$\text{CO}_3^{-2} \text{ (me L}^{-1}\text{)} = \frac{2R_1 \times \text{Normality of H}_2\text{SO}_4}{\text{aliquot(ml)}} \times 1000$$

or

Volume of aliquot taken = × ml (50 ml)

Volume of 0.1 N H₂SO₄ used = Final reading - blank reading
 = R₁

Actual volume of 0.1 N H₂ SO₄ used = 2× R₁

= 2R₁*

$$\text{Carbonates (me L}^{-1}\text{)} = \frac{2R_1 \times 0.1 \text{ N H}_2\text{SO}_4}{50} \times 1000 = 4 R_1$$

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$$\text{Bicarbonates (me L-1)} = \frac{(\text{R}_2 - \text{R}_1) \times \text{Normality of H}_2\text{SO}_4}{\text{aliquot (ml)}} \times 1000$$

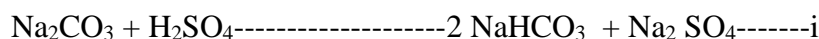
or

$$\begin{aligned} \text{Volume of aliquot taken} &= 50 \text{ ml} \\ \text{Volume of 0.1 N H}_2\text{SO}_4 \text{ used} &= \text{Final reading} - \text{Initial reading} \\ \text{**} &= \text{R}_2 \end{aligned}$$

$$\text{Actual volume of 0.1 N H}_2\text{SO}_4 \text{ used} = \text{R}_2 - \text{R}_1$$

$$\begin{aligned} \text{Bicarbonates (me L}^{-1}\text{)} &= \frac{(\text{R}_2 - \text{R}_1) \times 0.1 \text{ N H}_2\text{SO}_4}{50} \times 1000 \\ &= 2 (\text{R}_2 - \text{R}_1) \end{aligned}$$

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



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 CO₂ and H₂O as shown in equation ii. Thus R₁ is the amount of acid for half reaction and 2R₁ will represent the amount of complete reaction. Hence R₁ is multiplied by a factor of 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.

6. CAUTIONS / SAFETY REQUIREMENTS:

Always run blank for accuracy.

7. REFERENCE / RELATED DOCUMENTS:

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DETERMINATION OF CALCIUM AND MAGNESIUM

PURPOSE:

Determination of Calcium and Magnesium

2. EQUIPMENTS/APPARATUS REQUIRED:

China dish

Pipette, 10 ml

Cylinder, 50 ml

Glass rod

Wash bottle

3. REAGENTS/MEDIAS REQUIRED:

A. NH_4Cl - NH_4OH buffer solution

Dissolve 67.5 g NH_4Cl in 570 ml concentrated NH_4OH , make the volume upto 1 litre..

B Sodium hydroxide (NaOH), 4 N

Dissolve 160 g NaOH in distilled water, make the volume of the solution to 1 litre .

C. Standard calcium chloride solution (CaCl_2) , 0.01 N

Dissolve 0.5 g CaCl_2 pure (calcite crystals) in 10 ml of approx. 3 N (1+3) HCl and dilute to a volume of exactly 1 litre.

D. Eriochrome black-T indicator (EBT)

Dissolve 0.4 g of EBT and 4.5 g hydroxyl amine hydrochloride ($\text{NH}_2\text{O.H. HCl}$, $\text{H}_3\text{NO.HCl}$, m. wt. 69.49) in 100 ml of 95 % ethanol.

E. Ammonium purpurate indicator ($\text{C}_8\text{H}_4\text{N}_5\text{O}_6. \text{NH}_4$)

Thoroughly mix 0.5 g ammonium purpurate with 100 g of powdered potassium sulphate (K_2SO_4)

F. EDTA (Versenate) solution, 0.01 N

Dissolve 2.0 g of disodium salt and 0.5 g magnesium chloride hexahydrate ($\text{MgCl}_2. 6\text{H}_2\text{O}$) in distilled water and dilute to a volume of 1 litre . Standardised the solution against reagent C using given titration procedure.

4. METHOD:

Pre-treatment of soil extract .

Ammonium acetate and dispersed organic matter present in appreciable amount must be removed from soil extracts prior to titration with versenate. For this purpose soil extract is dried by evaporation after treating with aqua regia (3 part conc. HCl + 1 part conc. HNO_3) Second dryness is considered sufficient for the removal of ammonium acetate and organic matter. (Soil extract with dark colours require additional treatment with aqua regia).

CALCIUM:

Pipette out 10 ml of pre-treated aliquot in a conical flask. Add 5 drops of reagent B and add 50 mg of E. Titrate against F from orange red to lavender/purple and point. When closed to the endpoint,

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reagent F should be added drop wise (each drop after 5-10 second) as the colour change is not instantaneous.

CALCIUM PLUS MAGNESIUM:

Pipette out 10 ml of aliquot in a conical flask. Add 10 drops of reagent A, 3-4 drops of reagent D. Titrate against reagent F to a change in colour from wine red to bluish green.

5. CALCULATIONS REQUIRED:

ml of ETDA sol. for sample -ml of EDTA for blank
x N x 1000

$$\text{Ca}^{++} / \text{Ca}^{++} + \text{Mg}^{++} \text{ (me L}^{-1}\text{)} = \frac{\text{ml of ETDA sol. for sample} - \text{ml of EDTA for blank}}{\text{aliquot in ml}} \times \text{N} \times 1000$$

$$= \frac{R_1 - R_2 \times 0.01 \times 1000}{10}$$

$$= (R_1 - R_2) = R \text{ me L}^{-1}$$

$$\text{Mg}^{++} \text{ (me L}^{-1}\text{)} = \text{Ca}^{++} + \text{Mg}^{++} - \text{Ca}^{++} \text{ (All expressed as meL}^{-1}\text{)}$$

6. REFERENCE / RELATED DOCUMENTS:

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DETERMINATION OF CHLORIDE IN SOIL

1. PURPOSE:

Determination of soil chloride

2. EQUIPMENTS/APPARATUS REQUIRED:

- Pipette, 50 ml
- Conical flask, 100ml
- Cylinder, 50 ml
- Wash bottle
- Burette, 50 ml

3. REAGENTS/MEDIAS REQUIRED:

A. Potassium Chromate (K₂CrO₄), 5 % solution

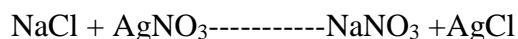
Dissolve 5.0 g of potassium chromate in 50 ml distilled water and add 1 N (saturated solution) AgNO₃ drop wise until red precipitate is produced. The solution is filtered and filtrate is diluted to 100 ml.

B. Silver Nitrate (AgNO₃), 0.05 N (F. wt. 169.888)

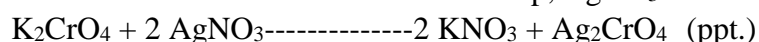
Dissolve 8.494 g silver nitrate in distilled water and dilute to 1 litre. Keep in a brown bottle away from light to avoid photolysis.

4. METHOD

To the same conical flask after light orange yellow end point, add 3-4 drops of reagents A. While stirring, titrate under bright light with reagent B., to a brick red precipitate./permanent reddish brown colour..



As soon as the amount of NaCl is used up, AgNO₃ reacts with potassium chromate.



5. CALCULATIONS REQUIRED:

$$\text{Cl (me L}^{-1}\text{)} = \frac{(\text{ml of AgNO}_3 \text{ for sample -ml of AgNO}_3 \text{ for blank)} \times \text{N}}{\text{aliquot (ml)}} \times 1000$$

or

Volume of aliquot taken = 50 ml

Volume of 0.05 N AgNO₃ used = Final reading - initial reading = R₃

$$\text{Cl (me L}^{-1}\text{)} = \text{R}_3 \times 0.05 \text{ N AgNO}_3 \times 1000/50$$

6. CAUTIONS / SAFETY REQUIREMENTS.

Always run a blank with actual titration.

7. REFERENCE / RELATED DOCUMENTS:

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DETERMINATION OF ORGANIC MATTER

1. PURPOSE:

Organic matter determination in soil

2. EQUIPMENTS/APPARATUS REQUIRED:

A. 1 N Potassium dichromate (K₂Cr₂O₇, f. wt. 294. 18): Dissolve oven dried 49.04 g potassium dichromate in distilled water, mix well and dilute to 1litre.

B. 1.0 N ferrous sulphate standardised: Dissolve 278.02 g ferrous sulphate (FeSO₄.7H₂O), in distilled water, add 15 ml concentrated sulphuric acid and dilute to one litre.

C. Sulphuric Acid (96 %, specific gravity 1.84)

D. Phosphoric acid (H₃PO₄) 85 % or sodium fluoride (NaF)

E. Barium diphenylamine sulphonate, 0.16 %: Dissolve 0.16 g barium diphenylamine sulphonate indicator in 100 ml conc. H₂SO₄ of 4:1 ratio.

3. REAGENTS/MEDIAS REQUIRED:

500 ml conical flask

Pipette, 10 ml

Burette, 50 ml

Beaker, 500 ml

Cylinder, 50 ml

Wash bottle

Weighing balance

4. METHOD:

Weigh 1.0 g air dried, ground soil sample into 500 ml conical flask, add 5 ml K₂Cr₂O₇ and mix well. Add 10 ml sulphuric acid and mix. Allow the flask to stand for 30 minutes to cool. Then add 100-150 ml distilled water. Add 3 ml phosphoric acid or 0.5 g sodium fluoride, 5-10 drops of indicator and finally titrate against standardised ferrous sulphate to sharp green end point through blue colour. Run a blank and subtract sample reading from blank to get the actual volume of ferrous sulphate used to reduce potassium dichromate.

5. CALCULATIONS REQUIRED:

$$\% \text{ O M} = \frac{\text{ml for blank} - \text{ml for sample}}{\text{weight of sample (g)}} \times 0.698^*$$

$$\% \text{ O M} = \frac{\text{me of K}_2\text{Cr}_2\text{O}_7 \text{ reduced}}{\text{weight of sample (g)}} \times 0.698$$

me of K₂Cr₂O₇ reduced = (me of K₂Cr₂O₇ added - me of FeSO₄ used)

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me of $K_2Cr_2O_7$ added = (ml of $K_2Cr_2O_7$ added x N)

me of $FeSO_4$ used = (ml of $FeSO_4$ used x N)

ml for blank - ml for sample

$$\% \text{ O C} = \frac{\text{ml for blank} - \text{ml for sample}}{\text{weight of sample (g)}} \times N \text{ FeSO}_4 \times 0.337$$

% O C = % organic matter / 1.724

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

How = 0.003

1 me of C of OM in soil = 1 x Eq. wt. of C / 1000

$$\text{Eq. Wt. of C} = \frac{\text{m. wt.}}{\text{valency}} \times \frac{12}{4} \times \frac{3}{1000} = \frac{3}{1000} = 0.003 \text{ g of C of OM in soil}$$

So

1 ml of 1 N $K_2Cr_2O_7$ when reduced = 0.003 g of C of OM in soil

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

100 / 58 = 1.724, for conversion of % OM 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 soils has been found to give app. 89 % recovery of carbon as compared to dry combustion method. The conversion factor, 0.337 was obtained by dividing 0.003, the milliequivalent weight of carbon by 89 and multiplying by 100 to convert to percent.

6. CAUTIONS / SAFETY REQUIREMENTS:

Always run blank along with samples.

7. REFERENCE / RELATED DOCUMENTS:

Walkley, A. 1947. A critical examination of a rapid method for determining organic carbon in soils – effect of variations in digestion conditions and of inorganic soil constituents. Soil Sci. 63:251-264.

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DETERMINATION OF SOIL TEXTURAL CLASS

1. PURPOSE:

Soil Textural Class Determination

2. EQUIPMENTS/APPARATUS REQUIRED:

Hydrometer
Graduated cylinder 1000 ml
Thermometer
Stop watch
Stirrer with cup
Beaker 500 ml
Perforated brass plunger
Wash bottle

3. REAGENTS/MEDIAS REQUIRED:

- i. Sodium hexametaphosphate (4%), dispersing reagent (NaPO_3)₆: Dissolve 40 g of sodium hexametaphosphate and 10 g of sodium carbonate in distilled water, mix well and make the volume one litre (new solutions should be prepared after 1 – 2 weeks).
- ii. Amyl Alcohol

4. METHOD:

1. Weight 40g air-dry soil (2-mm) into a 600mL beaker.
2. Add 60-mL dispersing solution
3. Cover the beaker with a watch-glass, and leave overnight
4. Quantitatively transfer contents of the beaker to a soil-stirring cup, and fill the cup to about three-quarters with water.
5. Stir suspension at high speed for 3 minutes using the special stirrer. Shake the suspension overnight if no stirrer is available.
6. Rinse stirring paddle into a cup, and allow to stand for 1 minute.
7. Transfer suspension quantitatively into a 1-L calibrated cylinder (hydrometer jar), and bring to volume with water.

A. Determination of Blank

- Dilute 60 mL dispersing solution to 1-L hydrometer jar with water
- Mix well, and insert hydrometer, and take hydrometer reading **Rb**.
- The blank reading must be re-determined for temperature changes of more than 2°C from 20°C

B. Determination of Silt plus Clay.

- Mix suspension in the hydrometer jar, using a special paddle carefully, withdraw the paddle, and immediately insert the hydrometer.
- Disperse any froth, if needed, with one drop of amyl alcohol, and take hydrometer reading 40 seconds after withdrawing the paddle. This gives reading **Rsc**.

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CALCULATIONS

Percentage **Silt** plus **Clay** in soil

$$\% \text{ [Silt +Clay] (w/w)} = (R_{sc}-R_b) \times \frac{100}{\text{Oven -dry soil (g)}}$$

C. Determination of Clay

- Mix suspension in the hydrometer jar with paddle, withdraw the paddle, with great care, leaving suspension undisturbed
- After 4 hours, insert the hydrometer, and take hydrometer reading **R_c**.

Percentage **Clay** in soil:

$$\% \text{ Clay (w/w)} = (R_c-R_b) \times \frac{100}{\text{Oven -dry soil (g)}}$$

Percentage **Silt** in soil:

$$\% \text{ Silt (w/w)} = \% \text{ [Silt +Clay (w/w)]} - [\% \text{ Clay (w/w)}]$$

D. Determination of Sand

- After taking readings required for clay and silt, pour suspension quantitatively through a 50µm sieve.
- Wash sieve until water passing the sieve is clear.
- Transfer the sand quantitatively from sieve a 50 mL beaker of known weight.
- Allow the sand in the beaker to settle, and decant excess water.
- Dry beaker with sand overnight at 105°C
- Cool in a desiccator, and re-weigh beaker with sand.

Percentage **Sand** in soil:

$$\% \text{ Sand (w/w)} = \text{Sand weight} \times \frac{100}{\text{Oven -dry soil (g)}}$$

Where: Weight of sand follows from

$$\text{Sand weight (g)} = [\text{Beaker + Sand (g)}] - [\text{Beaker (g)}]$$

Note:

1. If possible, all hydrometer jars should be placed in a water bath at constant temperature (20°C); in that case, temperature corrections are not needed.
2. For temperature correction, use a value of 0.4 for each degree temperature difference from 20°C. Add or subtract this factor if the temperature is more or less than 20°C, respectively.
3. All results of mechanical analysis should be expressed on the basis of oven-dry soil (24 hours drying at 105°C).
4. In the above procedure, carbonates and organic matter are not removed from the soil.
5. The Hydrometer method, as described in this section, cannot be applied to soils that contain free gypsum (gypsiferous soils). For gypsiferous soils, see Hesse (1971).
6. Sum of % silt and clay + % sand should be 100%. The magnitude of deviation from 100 is an indication for the degree in accuracy.

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Soil Texture

Once the percentage of sand, silt, and clay is measured, the soil may be assigned a textural class using the USDA textural triangle (Fig.4). Within the textural triangle are various soil textures which depend on the relative proportions of the soil fractions.

6. REFERENCE / RELATED DOCUMENTS:

Bouyouces, G. J. 1962. Hydrometer method improved for making particle size analysis of soils. Agron. J., 53:464-465.

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DETERMINATION OF EXTRACTABLE POTASSIUM IN SOIL

1. PURPOSE:

Extractable potassium determination in soil

2. EQUIPMENTS/APPARATUS REQUIRED:

Mechanical Shaker, reciprocating

Flamephotometer

Beaker, 50 ml

Pipette, 10 ml

Wash bottle

Volumetric flasks

3. REAGENTS/MEDIAS REQUIRED:

A. 1 N NH₄OAC: Dissolve 77.1 g ammonium acetate (F.W. 77.1, CH₃COONH₄) in 800 ml distilled water and make the volume to 1 litre. Adjust pH 7.0 using either NH₄OH or HCl.

B. Stock solution : Dissolve 1.907 g oven dried KCl in distilled water and make volume 1-2. This solution contains 1000ppm K.

ii. Prepare standard solutions from stock solution in 100ml volumetric flask.

4. METHOD: Wight 2.5 g air dried, ground, passed through 2 mm sieve soil sample into a 250ml conical flask, add 50 ml extracting reagent, shake on a flat bed recipro shaker for 30 minutes and filter the extract. Determine K by flame photometer in ppm using graph readings.

5. CALCULATIONS REQUIRED:

Extractable K (ppm) = reading (ppm) × 20

6. REFERENCE / RELATED DOCUMENTS:

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C. pp. 96-97.

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DETERMINATION OF EXTRACTABLE SOIL PHOSPHORUS (OLSEN'S METHOD)

1. PURPOSE:

Soil Extractable Phosphorus Determination (The Olsen Method)

2. EQUIPMENTS/APPARATUS REQUIRED:

Spectrophotometer

Standard laboratory glassware, Beaker, Volumetric flasks pipettes funnels

Extraction bottles

3. REAGENTS/MEDIAS REQUIRED:

A. Sodium bicarbonate solution, 0.5 M NaHCO₃: Dissolve 42.0 g NaHCO₃ in app. 700 ml distilled water, shake well and make the volume 1000 ml . Adjust pH 8.5 using 5 N NaOH.

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

B. Mixed reagent:

(a) Ammonium hepta molybdate 4.8 % (NH₄)₆MO₇O₂₄ 4HO₂ (f. wt. 1235.9)

Dissolve 12.0 g in distilled water and make volume 250 ml

(b) Potassium antimony tartrate (KsbO. C₄ H₂O₆)

Dissolve 0.291 g make volume to 100 ml with distilled water

(c) 5N H₂SO₄. Dilute 148 ml concentrated sulfuric acid (in fume hood) in distilled water, let it cool and make volume 1 L.

(d) Add both the dissolved reagents (a + b) in 1000 ml 5 N H₂SO₄ and make volume 2000 ml with distilled water. Store in a Pyrex bottle in a dark, cool place

C. Colour developing reagent: Weigh 0.528g 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 (1000 ppm): Dissolve 4.3937 g potassium dihydrogen phosphate (KH₂PO₄) in distilled water and make volume to 1 liter.

STANDARDS (0.05-1.5ppm):

Making 5 ppm sub-stock solution

4. METHOD:

Weigh 2.5 g air dried and ground soil, add 50 ml extracting solution. Shake for 30 minutes and filter with Whatman No. 42. Pipette out 5 ml aliquot. Add 5 ml 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 color developed. Take reading on concentration mode at 880 nm wavelength on Spectrophotometer.

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5. CALCULATIONS:- Extractable phosphorous in soil (ppm)=ppm p*x $\frac{A}{wt} \times \frac{50}{v}$

A= Total volume of Extractant (ml)

Wt= Wight of air-dry soils (g)

V= Volume of extract used (ml)

6. REFERENCE / RELATED DOCUMENTS:

Olsen, S. R., C. V. Cole, F. S. Watanabe and L. A. Dean. 1954. Estimation of available phosphorus in soils by extraction with sodium bicarbonate. USDA Circ. 939. pp. 19.

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DETERMINATION OF SODIUM IN SOIL

1. PURPOSE:

Sodium Determination of Soil

2. EQUIPMENTS/APPARATUS REQUIRED:

Flame Photometer

Volumetric flask

Conical flask

3. REAGENTS/MEDIAS REQUIRED:

Preparation of stock solutions

Stock solution of sodium (1000 ppm) can be prepared by dissolving oven dried NaCl (2.5435 g) in distilled water and making the volume to exactly one litre. Store in a cool and dry place.

Preparation of working standards:

Stock solution so prepared will be used for the preparation of working standards by using the formula:

$$C_1V_1 = C_2V_2$$

Where

C_1 = Concentration of stock solution in ppm

V_1 = Volume to be taken of stock solution in ml

C_2 = Concentration of Na to be required in ppm

V_2 = Total volume to be required in ml

$$C_2 \text{ (ppm required)} = \frac{V_1(\text{ml of known solution}) \times C_1 \text{ (ppm of known solution)}}{V_2 \text{ (Total volume to be made)}}$$

Working standard solution can be prepared of 10,20,30,-----100 ppm concentration and for plant 0-10 ppm concentration or as desired.

4. METHOD:

All Flamephotometers have approximately the same procedure but this procedure is mainly based on PFP 7 Flamephotometer.

Steps are as follow

1. After ignition of flame photometer wait for 5 – 10 minutes to stabilize the instrument.
2. Adjust zero with extractant.
3. Run desired standards and draw calibration curve.
4. If the sample to be analysed have higher concentration (ppm)than the highest working standard solution concentration, dilute the sample solution and multiply the reading with dilution factor.

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5. After completion of analysis aspirate the distilled water for at least 15 minutes to remove all the elemental particles and to avoid the chance of chocking.
6. In case of chocking the instrument, aspirate semi hot distilled water or very dilute HCl in semi hot distilled water. However in case of still chocking very dilute HCl in semi hot distilled water should be used for aspirating.
7. Clean the table, wash all the apparatus and place at proper place before leaving the laboratory.

5. CALCULATIONS REQUIRED:

Na (ppm) = R × Dilution factor

Where

R = Flame photometer reading in concentration made

6. REFERENCE / RELATED DOCUMENTS:

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington.

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DETERMINATION OF SULPHATE IN SOIL

1. PURPOSE:

Soil Sulphate Determination

2. EQUIPMENTS/APPARATUS REQUIRED:

Spectrophotometer

Electronic balance

Pipette

3. REAGENTS/MEDIAS REQUIRED:

A. 0.001 M CaCl₂: 0.147 g CaCl₂ L⁻¹

B. Acid Mixture: 125 ml HNO₃ + 250 ml Acetic Acid + 100 ml H₃PO₄ (85 %) Dilute to one litre with deionized water.

C. Acid Sulphate solution (20ppm S): 86ml conc. HCl+100ml S solution (100ppm). Make volume upto 500 ml with Deionized water.

D. BaCl₂.2H₂O Crystal: 20-60 mesh crystal

E. Gum Acacia Solution: Dissolve 0.5 g gum acacia in 50 ml Deionized water. Mix and filter. Add 50 ml acetic acid. Filter if necessary.

F. Dissolve 0.5434 g potassium Sulphate in distilled water and bring to 1-L volume. This solution contains 100ppm SO₄-S (stock solution).

G. SO₄ – Standards from 100 ppm standard: 0, 5, 10, 15 and 20 ppm solutions

4. METHOD:

Weigh 25 g soil and add 50 ml CaCl₂ extracting solution. Shake for 30 minutes and filter through Whatman No. 42 filter paper. Take 5 ml sample in a 50 ml volumetric flask. Add 5 ml mixed acid reagent and 1 ml acid Sulphate solution and mix. Add 0.5 g BaCl₂.2H₂O crystals. Let stand undisturbed for 3 minutes and then mix.

Add 1 ml gum acacia reagent and mix. Make 50 ml volume with distilled water. Take readings of samples and standards at 420-450 nm between 3 to 8 minutes after final shaking. Run reagent blank.

5. CALCULATIONS REQUIRED:

Sulphate concentration (ppm) = Sulphate conc. (ppm) × Dilution factor

7. REFERENCE / RELATED DOCUMENTS:

Bardsley, C. E. and J. D. Lancaster. 1965. Chapter 79. C. A. Black. Ed. Methods of soil analysis.

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DETERMINATION OF CALCIUM CARBONATE IN SOIL

1. PURPOSE:

Soil Calcium Carbonate Assessment

2. EQUIPMENTS/APPARATUS REQUIRED:

Pipette, 10 ml

Flask

Balance

3. REAGENTS/MEDIAS REQUIRED:

Hydrochloric Acid (1 + 3, Acid + Water)

4. METHOD:

Weigh the flask. Add 10 ml HCl 3 N (1:3 acid, water) and weigh it. Add 10 g soil and weigh again. Shake gently and stay for two hours. Note the final weight of flask after 2 hours.

5. CALCULATIONS REQUIRED:

Wt. of flask = A

Wt. of flask + acid = B

Wt. of flask + acid + soil = C

Wt. of flask + acid + soil after 2 hours = D

Wt. of CO₂ lost = Initial Wt. (C) – Final Wt. (D) = E

E × 227.4

% CaCO₃ = -----

Wt. of soil taken

7. REFERENCE / RELATED DOCUMENTS:

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C.

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GYPSUM REQUIREMENT OF SALINE SODIC AND SODIC SOIL

1. PURPOSE:

Assessment of Gypsum Requirement for Saline Sodic and Sodic Soil

2. EQUIPMENTS/APPARATUS REQUIRED:

Weighing balance

Volumetric flask, 1000 ml

Mechanical shaker

Conical flask, 250 ml

Plastic bottle, 250 ml

Funnel with stand

Pipette, 10 ml

Burette, 50 ml

3. REAGENTS/MEDIAS REQUIRED:

A. Saturated gypsum solution: Dissolve 5.0 g CaSO₄·2H₂O in 1 litre distilled water. Stopper the volumetric flask and shake by hand for one hour or for 10 minutes on reciprocating shaker. Filter and determine the calcium concentration of solution by versenate method. The calcium concentration should be at least 28 me L⁻¹.

B. EDTA 0.01 N. Dissolve 2.0g of EDTA and 0.05g MgCl₂·6H₂O in water and dilute to volume of 1-L.

C. Buffer solution: Ammonium chloride – ammonium hydroxide solution. Dissolve 67.5g of ammonium chloride in 570ml of concentration ammonium hydroxide and make volume to 1-L.

D. EBT indicator. Dissolve 0.5g of EBT and 4.5g of hydroxylamine hydrochloride in 100ml of 95% ethanol.

4. METHOD:

Weigh 5.0 g air dried soil in a 250 ml capacity plastic bottle, add 100 ml saturated gypsum solution, stopper the bottle, shake by hand several times for half an hour or for 5 minutes in a mechanical shaker. Filter the suspension and. Take 5 ml of aliquot, add 7 – 10 drops of C and D each. Titrate against B from wine red to bluish end point.

5. CALCULATIONS :

$$\text{Gypsum Req. (me100 g}^{-1}\text{)} = \frac{\text{Blank Reading} - \text{Sample Reading} \times 0.01 \times 1000 \times 2}{5.0}$$

5 g soil was added in 100 ml of gypsum saturated solution.

So, 1000 ml soil filtrate will correspond to 50 g air dry soil.

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Therefore, in 50 g soil, Ca conc. = Ca conc. (me L⁻¹) of gyp. sol. - Ca+Mg conc. (me L⁻¹) of filtrate.

Ca conc. in 100 g soil = 100/50 = 2

Conversions:

Say

100 g soil has gypsum requirement = M me

Then gyp. req. for

Tonnes per acre 15 cm of soil = M x 0.86

Tonnes per ha 15 cm of soil = M x 2.12

Tonnes per acre 30 cm of soil = M x 1.72

Tonnes per ha 30 cm of soil = M x 4.25

6. CAUTIONS / SAFETY REQUIREMENTS:

6.1 Never use oven dry soil sample as heating promotes the conversion of CaSO₄ 2H₂O to CaSO₄.1/2 H₂O (plaster of Paris) which is more soluble than that of CaSO₄. 2H₂O. Hence the gypsum requirement will be more than the actual.

7. REFERENCE / RELATED DOCUMENTS:

Schoonover, W. R. 1952. Examination of soil for alkali. Univ. of Calif. Ext. Service Berkely California. (Mimeographed).

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DETERMINATION OF TOTAL NITROGEN IN SOIL

1. PURPOSE:

Total Nitrogen Evaluation in Soil

2. EQUIPMENTS/APPARATUS REQUIRED:

- Kjeldhal distillation unit
- Balance
- Volumetric flasks, 100, 1000 & 2000ml
- Pipette, 10 ml
- Conical flask, 250 ml
- Burette, 50 ml
- Beaker, 50 & 1000 ml
- Wash bottle
- Digestion Block

3. REAGENTS/MEDIAS REQUIRED:

- A. Sulphuric acid Conc. (reagent grade N free).
- B. Digestion mixture: K₂SO₄+CuSO₄ in 9:1
- C. Sodium hydroxide solution (40%): Dissolve 400g sodium hydroxide in one litre distilled water.
- D. Bromocresol green and Methyl red indicator : Dissolve 0.5 g BCG and 0.1 g methyl red in 100 ml of 95 % ethanol
- E. Boric acid solution (4%): Dissolve 40 g of boric acid in 1.0 litres of CO₂ free distilled water.
- F. 0.1 N H₂SO₄ solution
- G. Phenolphthalein indicator

STANDARDISATION OF NaOH 0.1 N:

REAGENTS:

- A Potassium acid phthalate 0.1 N: Weigh 20.423 g of oven dried potassium acid phthalate, dissolve in distilled water and make the volume 1.0 litre.
- B. Phenolphthalein indicator. Dissolve 1 g of phenolphthalein in 100 ml 50% ethanol (50 ml of ethanol and 50 ml distilled water) with constant stirring. Filter if precipitates are formed.
- C. Sodium hydroxide 0.1 N: 4.0 g of sodium hydroxide is dissolved in distilled water and make volume one litre.

Pipette 10 ml of 0.1 N potassium acid phthalate into a titration flask and add 1-2 drops of phenolphthalein, titrate against solution of sodium hydroxide to light pink colour end point. Note the reading and adjust the normality of the base by

$$N_a \times V_a = N_b \times V_b$$

Standardisation of H₂SO₄ 0.1 N:

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Pipette out 10 ml of 0.1 N standardised base in a titration flask, add 1-2 drops of phenolphthalein indicator titrate against the acid to be standardised, to colourless endpoint. Note the volume of acid used. By using the above formula adjust the normality i.e. 0.1N. If less volume of acid to be standardised is used for 10 ml of standardised base then dilute the acid or vice versa.

4. METHOD:

Weigh 5 g air dried, passed through 2.0 mm sieve soil sample and 1.0 g digestion mixture into clean and dry digestion tube. Add 10-15 ml concentrated commercial sulphuric acid and heat at 420°C. Continue to heat until the colour of the material changes from black to white/greenish white. Solidification should be avoided.

Distill digested material in same flask on the distillation unit. Collect distillate in 20-25 ml 4% boric acid in the receiver. Add few drops of indicator. Purple color will form and will be changed to golden yellow on distillation. Which is then titrated against 0.1 N H₂SO₄ for soil from golden yellow to a purple end point.

5. CALCULATIONS REQUIRED:

$$\%N = \frac{14.1 \times \text{ml of titrant for sample} - \text{ml of titrant for blank} \times N \text{ of acid}}{\text{weight of sample (g)} \times 10}$$

$$= \frac{0.00141 \times R \times 100}{5}$$

Factor for soil if weight of soil is 5.0 g

$$\% N = R - \text{blank} \times 0.0282$$

6. CAUTIONS / SAFETY REQUIREMENTS:

6.1 Always run blank along with samples.

7. REFERENCE / RELATED DOCUMENTS:

Bremner, M. 1960. Kjeldhal Method. Agronomy – 9, 2nd Ed.

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DETERMINATION OF CATION EXCHANGE CAPACITY OF SOIL

1. PURPOSE:

Determination of the Capacity of the Cation Exchange of Soil

2. EQUIPMENTS/APPARATUS REQUIRED:

Flame photometer

Centrifuge machine

Volumetric flask, 100 ml

Cylinder, 50 ml

Weighing balance

Funnel with stand

Burette, 50 ml

3. REAGENTS/MEDIAS REQUIRED:

A. Sodium acetate solution (saturating solution), 1.0 N: Dissolve 136 g sodium acetatetrihydrate in distilled water and make volume upto 1 litre. Adjust pH approximately 8.2 using NaOH or CH₃COOH.

B. Ethanol 95%

C. Ammonium acetate solution (replacing solution) 1.0 N: Dissolve 77.08g in distilled water and make volume up to 1L and adjust pH at 7.0 with Ammonium Sulphate.

D. **Sodium Stock Solution (1000 ppm)** Dissolve dried 2.5418 g of NaCl in 1 L deionized water (Desired working standards be prepared from it).

4. METHOD:

Weigh 5.0 g soil sample in a centrifuge tube. Add 33 ml of reagent A, stopper the tube, shake for 5-7 minutes, centrifuge for 5 minutes or till the clear supernatant solution in centrifuge at 1200-2000 rpm. Decant and discard. Repeat same process further two times with 33 ml of reagent A. Centrifuge, decant and discard the supernatant solution. Now add 33ml of B reagent to the tube, stopper, shake for 5-7 minutes. Decant and discard supernatant solution. Repeat the same procedure further two times with 33 ml of reagent B. Now add 33 ml of reagent C to the same tube, stopper, shake and centrifuge at 2000 rpm for 5-7 minutes. Decant the supernatant solution in the 100 ml volumetric flask. Repeat the same procedure for further two times with 33 ml of reagent C. Make the volume 100 ml with distilled water. This solution is ready for the determination of replaced Na by flame photometer.

5. CALCULATIONS:

$$\text{Cation exchange capacity (CEC) C mol (+) Kg}^{-1} = \frac{\text{Na conc. of ext. (me L}^{-1})}{\text{wt. of sample (g)}} \times 10$$

7. REFERENCE / RELATED DOCUMENTS:

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United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C. pp. 101.

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DETERMINATION OF EXTRACTABLE MICRONUTRIENTS IN SOIL

1. PURPOSE:

Determination of Plant Available Micronutrients in Soil

2. EQUIPMENTS/APPARATUS REQUIRED:

Atomic Absorption Spectrophotometer

Plastic beaker

Mechanical Shaker

Filter paper

Funnel

Test tubes

3. REAGENTS/MEDIAS REQUIRED:

0.005 M DTPA, 0.01 M CaCl₂, and 0.1 M TEA (tri-ethanol amine, adjusted to pH 7.3 with dilute HCl. Dissolve 3.934 g of DTPA and 2.94 g of CaCl₂ and 25.3 ml of TEA in approximately 200 ml of distilled water. After sufficient time for DTPA to dissolve, make the volume 2 litre. Adjust the pH 7.3 with 1:1 HCl while stirring.

4. METHOD:

Weigh 20 g of soil and add 40 ml of DTPA solution. Shake continuously for 2 hours on horizontal shaker and filter. A blank solution (0 ppm) containing all reagents except soil should be run with samples as blank. Read on concentration mode by atomic absorption spectrophotometer Prepare at least 4 standard using DTPA as matrix for each element with a range as following

For Zn and Cu 0.05,1.0,1.5,2.0 ppm

For Fe and Mn 0,5,10,20, 30, ppm

5. CALCULATIONS:

Micronutrients (ppm) = reading × 2

6. CAUTIONS / SAFETY REQUIREMENTS:

Always run blank along with samples.

7. REFERENCE / RELATED DOCUMENTS:

Lindsay and Norvell. 1978.

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DETERMINATION OF BORON IN SOIL

1. PURPOSE:

Boron Assessment in Soil

2. EQUIPMENTS/APPARATUS REQUIRED:

Spectrophotometer.

Weighing balance

Poly propylene volumetric flask

Pipette

Funnel with stand

Wash bottle

Water bath

Burner

Beaker, 50 ml

3. REAGENTS/MEDIAS REQUIRED:

A. Buffer solution: Dissolve 250 g ammonium acetate and 15 g of ethylenediamine 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 (H₃BO₃) in distilled water (for hot water extraction) or in 0.05 M HCl for HCl extraction and adjust the volume 1000 ml . This will be of 1000 ppm of boron solution. Make sub-stock solution to 100 ml of 100 ppm B. Then make working standard as desired.

$$C1V1 = C2V2$$

$$100 \times V1 = 0.5 \times 100$$

$$5 \times 100$$

$$V1 = \frac{5 \times 100}{1000} = 0.5 \text{ ml of sub-stock solution to make 0.5 ppm B working standard.}$$

D. 0.05 M HCl

4. METHOD:

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.

HCl EXTRACTION

Ten grams soil was shaken with 20 ml 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, 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 standard curve prepared Boron standards are prepared in

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0.05 M HCl for HCl extraction and in distilled water for hot water extraction. Using 420 nm wavelength determine boron (ppm) by spectrophotometer using color developing method.

5. CALCULATIONS:

$$B \text{ (ppm)} = R \times 100^*$$

Where

R = spectrophotometer reading

100* = total dilution factor.

6. CAUTIONS / SAFETY REQUIREMENTS:

6.1 Always run blank for accuracy.

7. REFERENCE / RELATED DOCUMENTS:

Bingham. 1982. Methods of soil analysis. Part II. Amer. Soc. Agron. USA.

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CHAPTER-3

WATER ANALYSIS

ELECTRICAL CONDUCTIVITY (EC) OF WATER

PURPOSE:

Determination of Electrical Conductivity (EC)

2. EQUIPMENTS/APPARATUS REQUIRED:

Conductivity Meter

3. REAGENTS/MEDIAS REQUIRED:

0.01 N KCl: Dissolve 0.7456 g KCl in 1 litre of distilled water.

4. METHOD:

The instrument must be standardised with 0.01 N KCl solution. The EC of this standard solution is 1.413 dS m⁻¹ at 25 °C. The cell constant (K) can be calculated by the formula.

$$1.413 \text{ dS m}^{-1}$$

$$K = \text{-----}$$

$$\text{Observed reading in dS m}^{-1}$$

The cell constant is necessary when 0.01 N KCl solution gives an EC values different from 1.413 dS m⁻¹. In this case, samples readings are multiplied by the cell constant.

5. CALCULATIONS REQUIRED:

$$\text{EC of water} = \text{Observed EC} \times K$$

6. CAUTIONS / SAFETY REQUIREMENTS:

The conductivity meter should be standardised daily or as desired depending upon the number of samples being tested.

7. REFERENCE / RELATED DOCUMENTS:

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C. pp. 89-91.

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DETERMINATION OF CARBONATES AND BICARBONATES IN WATER

PURPOSE:

Determination of the concentration of carbonates and bicarbonates in water

2. EQUIPMENTS/APPARATUS REQUIRED:

- Pipette, 50ml
- Conical flask, 100ml
- Cylinder, 50ml
- Wash bottle
- Burette, 50ml

3. REAGENTS/MEDIAS REQUIRED:

- A. Phenolphthalein 1%: Dissolve 1.0 g phenolphthalein in 100 ml ethanol with constant stirring, Filter if needed.
- B. Mehtyl Orange 0.1%: Dissolve 0.1g methyl orange in 100ml distilled water.
- C. Sulphuric Acid 0.1 N (standardized)

4. METHOD:

Pipette 50ml aliquot in conical flask, add 1-3 drops of reagent A. If no colour appears, carbonates are absent. If pink colour appears, carbonates are present. Titrate it against 0.1 N H₂SO₄ to colorless end point. Take three readings. Reserve the flask along with its contents for bicarbonates determination.

To the same conical flask (after colorless end point), add 1-2 drops of methyl orange, titrate against 0.1 N H₂SO₄ from golden yellow to a light pink or light orange colour end point. Take three readings. Reserve the flask for chloride determination.

5. CALCULATIONS REQUIRED:

$$\text{CO}_3^{-2} \text{ (me L}^{-1}\text{)} = \frac{2R_1 \times \text{Normality of H}_2\text{SO}_4}{\text{Aliquot (ml)}} \times 1000$$

Or

Volume of aliquot taken = 50 ml

Volume of 0.1 N H₂SO₄ used = Final Reading - Blank Reading
 = R₁

Actual Volume of 0.1 N H₂ SO₄ used = 2 x R₁ = 2R₁

$$\text{Carbonates (me L}^{-1}\text{)} = \frac{2R_1 \times 0.1 \text{ N H}_2\text{SO}_4}{50} \times 1000 = 4 R_1$$

$$\text{Bicarbonates (me L}^{-1}\text{)} = \frac{(R_2 - R_1) \times \text{Normality of H}_2\text{SO}_4}{\text{Aliquot (ml)}} \times 1000$$

Or

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DETERMINATION OF CHLORIDE IN WATER

1. PURPOSE:

Determination of Chloride in Water

2. EQUIPMENTS/APPARATUS REQUIRED:

Pipette, 50ml
 Conical flask, 100ml
 Cylinder, 50ml
 Wash bottle
 Burette, 50ml

3. REAGENTS/MEDIAS REQUIRED:

A. Potassium Chromate (K_2CrO_4), 5 % solution: Dissolve 5.0 g of potassium chromate in 50 ml distilled water and add 1 N (saturated solution) $AgNO_3$ drop wise until red precipitate is produced. The solution is filtered and filtrate is diluted to 100 ml.

B. Silver Nitrate ($AgNO_3$), 0.05 N. Dissolve 8.494 g silver nitrate in distilled water and dilute to 1 litre. Keep in a brown bottle to avoid photolysis.

4. METHOD:

To the same conical flask after light orange yellow end point, add 3-4 drops of reagents A. While stirring, titrate under bright light with reagent B., to a brick red precipitate./permanent reddish brown color.

5. CALCULATIONS REQUIRED:

$$Cl \text{ (me L}^{-1}\text{)} = \frac{\text{(ml of } AgNO_3 \text{ for sample -ml of } AgNO_3 \text{ for blank)} \times N}{\text{Aliquot (ml)}} \times 1000$$

Or

Volume of aliquot taken = 50 ml

Volume of 0.05 N $AgNO_3$ used = Final reading - initial reading
 = R_3

$$Cl \text{ (me L}^{-1}\text{)} = R_3 \times 0.05 \text{ N } AgNO_3 \times 1000/50$$

$$= R_3$$

Note: Always run a blank along with actual titration.

Q. Why we first neutralize CO_3 & HCO_3 before Cl titration?

If we don't neutralize CO_3 & HCO_3 , $AgNO_3$ will react with CO_3 & HCO_3 to form $AgHCO_3$. So more $AgNO_3$ will be utilize for chloride titration.

6. CAUTIONS / SAFETY REQUIREMENTS:

Always run blank for accuracy.

7. REFERENCE / RELATED DOCUMENTS:

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DETERMINATION OF CALCIUM AND MAGNESIUM IN WATER

PURPOSE:

Determination of Calcium and Magnesium in Water

2. EQUIPMENTS/APPARATUS REQUIRED:

- Burette
- China dish
- Pipette, 10 ml
- Cylinder, 50 ml
- Glass rod
- Wash bottle

3. REAGENTS/MEDIAS REQUIRED:

- A. NH₄Cl -NH₄ OH buffer solution: Dissolve 67.5 g NH₄Cl in 570 ml concentrated NH₄OH, make the volume up to 1 litre.
- B Sodium hydroxide (NaOH), 4 N: Dissolve 160 g NaOH in distilled water, make the volume of the solution to 1 litre .
- C. Standard calcium chloride solution (CaCl₂) , 0.01 N: Dissolve 0.5 g CaCl₂ pure (calcite crystals) in 10 ml of approx. 3 N (1+3) HCl and dilute to a volume of 1 litre.
- D. Eriochrome black-T indicator (EBT): Dissolve 0.5 g of EBT and 4.5 g hydroxyl amine hydrochloride (NH₂O.H. HCl, H₃NO.HCl) in 100 ml of 95 % ethanol.
- E. Ammonium purpurate indicator (C₈H₄N₅O₆. NH₄): Thoroughly mix 0.5 g ammonium purpurate with 100 g of potassium sulphate (K₂SO₄).
- F. EDTA (Versenate) solution, 0.01 N: Dissolve 2.0 g of EDTA and 0.05 g magnesium chloride hexahydrate (MgCl₂.6H₂O) in distilled water and dilute to a volume of 1 litre . Standardized the solution against reagent C using given titration procedure.

4. METHOD:

Calcium: Pipette 10 ml of pre-treated aliquot in a conical flask. Add 5 drops of reagent B and add 50 mg of E. Titrate against F from orange red to lavender/purple end point. When closed to the endpoint, reagent F should be added drop wise (each drop after 5-10 second) as the colour change is not instantaneous.

Calcium + Magnesium: Pipette 10 ml of aliquot in a conical flask. Add 10 drops of reagent A, 3-4 drops of reagent D. Titrate against reagent F to a change in colour from wine red to bluish green.

5. CALCULATIONS REQUIRED:

$$\text{Ca}^{++} \text{ or } \text{Ca}^{++} + \text{Mg}^{++} \text{ (me L}^{-1}\text{)} = \frac{\text{ml of EDTA sol. for sample -ml of EDTA for blank} \times \text{N}}{\text{Aliquot in ml}} \times 1000$$

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$$= \frac{R_1 - R_2 \times 0.01 \times 1000}{10}$$

$$= (R_1 - R_2) = R \text{ me L}^{-1}$$

$$\text{Mg}^{++} (\text{me L}^{-1}) = \text{Ca}^{++} + \text{Mg}^{++} - \text{Ca}^{++} \text{ (All expressed as meL}^{-1}\text{)}$$

6. CAUTIONS / SAFETY REQUIREMENTS:

Always run a blank.

7. REFERENCE / RELATED DOCUMENTS:

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C.

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DETERMINATION OF SULPHATE IN WATER

1. PURPOSE:

Determination of Sulphate in Water

2. EQUIPMENTS/APPARATUS

Spectrophotometer

Electric balance

Volumetric flask

Pipette

3. REAGENTS/MEDIAS:

A. Acid Mixture: 125 ml HNO₃ + 250 ml Acetic Acid + 100 ml H₃PO₄ (85 %) Dilute to one litre with deionized water.

B. Acid Sulphate solution (20 ppm S): 86 ml conc. HCl + 100 ml S solution (100 ppm). Make volume upto 500 ml with deionized water.

C. BaCl₂.2H₂O Crystal: 20-60 mesh crystal

E. Gum Acacia Solution: Dissolve 0.5 g gum acacia in 50 ml deionized water. Mix and filter. Add 50 ml acetic acid. Filter if necessary.

F. SO₄ – Standards: Standards of 5, 10, 15 and 20 ppm from 100 ppm stock solution

4. METHOD:

Take 5 ml sample in a 50 ml volumetric flask. Add 5 ml mixed acid reagent and 1 ml acid sulphate solution and mix. Add 0.5 g BaCl₂.2H₂O crystals. Let stand undisturbed for 3 minutes and then mix. Add 1 ml gum acacia reagent and mix. Make total volume 50 ml. Take readings of samples and standards at 420-450 nm between 3 to 8 minutes after final shaking. Run reagent blank.

5. CALCULATIONS:

Sulphate concentration (ppm) = Sulphate conc. (ppm) × Dilution factor

6. REFERENCE / RELATED DOCUMENTS:

Verma, B. C. 1977.

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DETERMINATION OF SODIUM (Na) AND POTASSIUM (K) IN WATER

1. PURPOSE:

Determination of Sodium (Na) and Potassium (K) in water

2. EQUIPMENTS/APPARATUS REQUIRED:

Flame Photometer

3. REAGENTS/MEDIAS REQUIRED:

Preparation of stock solutions

Sodium: Dissolve 2.5435g dry NaCl in 1-L, it will be 1000ppm Na.

Potassium: Dissolve 1.9103 g dry KCl in 1-L, it will be 1000ppm K.

However, both the stock solutions in combined form can easily be prepared by dissolving the said amounts of both the salts in exactly one litre distilled water. Store in a cool and dry place.

Preparation of working standards:

Stock solutions so prepared will be used for the preparation of working standards by using the formula:

$$C_1V_1 = C_2V_2$$

Where

C₁ = Concentration of stock solution in ppm

V₁ = Volume to be taken of stock solution in ml

C₂ = Concentration of Na or K to be required in ppm

V₂ = Total volume to be required in ml

$$C_2 \text{ (ppm required)} = \frac{V_1 \text{ (ml of known solution)} \times C_1 \text{ (ppm of known solution)}}{V_2 \text{ (Total volume to be made)}}$$

Working standard solution can be prepared of 10, 20, 30,-----100 ppm concentration and for plant 0-10 ppm concentration or as desired.

4. METHOD:

CALIBRATION AND OPERATION:

1. "On" the fuel (gas).
2. Ignition
3. During ignition rotate the fuel knob clockwise for more gas supply to make ignition easy.
4. After the ignition rotate the knob anticlockwise and adjust the fuel in such a way that small cones should be formed in flame (blue flame).
5. Aspirate the distilled water for at least 10-15 minutes before aspirating the working standard solutions.
6. Be sure that you have adjusted the required filter i.e., sodium filter for Na or potassium filter

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for K by the knob provided.

7. Now adjust the "ZERO" on the scale by rotating the knob "BLANK".
8. Aspirate the standard working solution and record the reading in ppm e.g. when you feed 100 ppm, standard solution the reading on the scale should be 100. If it is not 100 then use "COARSE" or "FINE" adjustment to make the reading 100. Use desired maximum standard to adjust 100.
9. Without disturbing the blank, check whether there is "ZERO" on the scale when distilled water is aspirated after aspirating working standard solutions. If so instrument is standardised according to the given Na or K filter. If not so then adjust "ZERO" by adjustment "BLANK" and repeat all the above procedure with standard working solution.
10. Before aspirating the next sample, always aspirate the distilled water until reading becomes ZERO
11. If the sample to be analyzed have higher concentration (ppm) than the highest working standard solution concentration, dilute the sample solution and multiply the reading with dilution factor.
12. After finishing the work aspirate the distilled water for at least 15 minutes for cleaning to avoid the chance of chocking.

5. CALCULATIONS REQUIRED:

K or Na (ppm) = ppm from calibration curve of standards.

6. REFERENCE / RELATED DOCUMENTS:

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C. pp. 96-97.

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<i>SFRI-STM-01</i>	Standard Test Methods	Issue on	15.01.2021	Revision	0.0
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DETERMINATION OF SODIUM ADSORPTION RATIO (SAR) AND RESIDUAL SODIUM CARBONATE (RSC) OF WATER

PURPOSE:

Determination of Sodium Adsorption Ratio (SAR) and Residual Sodium Carbonate (RSC) of Water

2. CALCULATIONS REQUIRED:

$$\text{SAR} = \text{Na}^+ / [(\text{Ca}^{++} + \text{Mg}^{++})/2]^{1/2}$$

All the cations are expressed as me L⁻¹.

Residual sodium carbonate can be calculated by using following equation

$$\text{RSC, me L}^{-1} = (\text{CO}_3^{-} + \text{HCO}_3^{-}) - (\text{Ca}^{++} + \text{Mg}^{++})$$

All expressed in me L⁻¹.

3. REFERENCE / RELATED DOCUMENTS:

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C.

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DETERMINATION OF HEAVY METALS IN WATER

PURPOSE:

Determination of Heavy Metals in Water

2. EQUIPMENTS/APPARATUS REQUIRED:

Atomic Absorption Spectrophotometer / ICP-OES

3. REAGENTS/MEDIAS REQUIRED:

Standard solutions for the respective heavy metals

4. METHOD:

Determine the heavy metals concentration using atomic absorption spectrophotometer / ICP-OES after standardizing. Dilute the sample if necessary.

5. CALCULATIONS REQUIRED:

Direct concentration is given on these instruments

7. REFERENCE / RELATED DOCUMENTS:

Standard method for the examination of water and waste water. (APHA, AWWA WEF) 20th Edition, 1998.

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DETERMINATION OF BORON IN WATER

1. PURPOSE:

Determination of Boron in Water

2. EQUIPMENTS/APPARATUS REQUIRED:

Poly propylene volumetric flask, 50 ml

Pipette, 2 ml

Weighing balance

Funnel with stand

Wash bottle

Beaker, 50 ml

Spectrophotometer.

3. REAGENTS/MEDIAS REQUIRED:

A. Buffer solution: Dissolve 250 g ammonium acetate and 15 g of ethylenediamine 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 (H₃BO₃) in distilled water (for hot water extraction) and in 0.05 M HCl for HCl extraction and adjust the volume 1000 ml . This will be of 1000 ppm of boron solution. Make sub-stock solution to 100 ml of 100 ppm B. Then make working standard as desired.

$$C_1V_1 = C_2V_2$$

$$100 \times V_1 = 0.5 \times 100$$

$$5 \times 100$$

$$V_1 = \frac{5 \times 100}{1000} = 0.5 \text{ ml of sub-stock solution to make 0.5 ppm B working standard.}$$

4. METHOD:

Filter water sample in boron free apparatus (Polypropylene). If boron free apparatus is not available then pyrex glass apparatus should be dipped in concentrated HCl for a week and then used.

Boron standards (0.5, 1.0, 1.5, 2.0 and 2.5 ppm) were prepared in distilled water. At 420 nm wavelength determine boron (ppm) by spectrophotometer after developing color using azomethine – H.

Transfer 1 ml sample in 50 ml polypropylene volumetric flask, add 2 ml buffer solution, add 2 ml azomethine- H reagent and mix. After 30 minutes read absorbance by specrophotometer directly at concentration mode or by graph using standard curve prepared from standards readings.

5. CALCULATIONS REQUIRED:

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$B \text{ (ppm)} = R \times \text{dilution factor}$

Where

R = spectrophotometer reading

6. CAUTIONS / SAFETY REQUIREMENTS:

6.1 Always run blank for accuracy.

7. REFERENCE / RELATED DOCUMENTS:

Gaines, T. P. and G. A. Mitchell. 1979. Commun. Soil Sci. Plan Anal. 10:1099-1108.

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