PRACTICAL MANUAL

Soil Fertility and Nutrient Management

HNR 132 2(1+1)

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Syllabus: Soil Fertility and Nutrient Management HNR 132 2(1+1)

Analysis of soil for organic matter, available N,P,K and Micronutrients and interpretations. Gypsum requirement of saline and alkali soils. Lime requirement of acid soils. Estimation of organic carbon content in soil. Determination of Boron and chlorine content In soil. Determination of Calcium, Magnesium and Sulphur in soil. Sampling of organic manure and fertilizer for chemical analysis. Physical properties of organic manure and fertilizers. Total nitrogen in urea and farmyard manure. Estimation of ammonical nitrogen and nitrate nitrogen in ammonical fertilizer. Estimation of water soluble P2O5, Ca and S in SSP, Lime and Gypsum. Estimation of Potassium in MOP/SOP and Zinc in zinc sulphate. Visiting of fertilizer testing laboratory.

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CONTENTS

Experiment No. 1

Objective: To study sampling of organic manure, solid and liquid fertilizers

Organic manure: The organic materials derived from plant origin and animal waste which most commonly used to improve soil conditions and fertility include farm yard manure (FYM), animal wastes, crop residues, urban organic wastes (either as such or composted), green manures, bio-gas spent slurry, microbial preparations, vermicompost, Sewage sludge and some of the industrial wastes also find application in agriculture.

Material required: ……..

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Procedure of sample collection:

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How would you prepare manure sample for analysis: ……

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Solid fertilizer: Solid fertilizer is typically granulated or powdered form applied to crops. About 90% of fertilizers are applied as solids. The most widely used solid inorganic fertilizers are urea, diammonium phosphate and potassium chloride.

Material required for the sampling of solid fertilizer:

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Write the sampling procedure: ……..

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How prepare the sample for analysis: ……

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Liquid fertilizers: In India three liquid fertilizers are being used namely; ammonium polyphosphate (10-34-0),

urea ammonium nitrate (32% N) and super phosphoric acid (70 % P2O5). They are used mostly for fertigation. While the analytical procedure for the determination of N and P in them are the same as for the solid fertilizers, however, their sampling procedure is different and hence, described below:

Material required for the sampling of liquid fertilizer: ……

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Write the sampling procedure: ……..

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Write the precautions during sampling of fertilizers: ……..

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Experiment No. 2

Objective: To study physical properties of organic manure and fertilizer.

Organic manure: There are following physical characteristics of compost/ organic manure which used to evaluation for the maturity of organic manure.

1. Temperature and heat output:……...
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2. Color:……

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3. Odour/smell:……

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4. Structure:……

5. Moisture:……

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6. pH :……

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Write the standard which used for assessing the quality of compost

Criteria	Good quality	Poor quality
Color		
Odour (smell)		
pH		
C:N ratio		
Moisture		
Temperature		
Humus		
Nitrogen		

Fertilizers: The physical properties that directly affect the quality of spread of granular fertilizers and particle density are the most impactful factors influencing the deposition of granular fertilizers.

1. Particle size: ……..

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2. Particle density:

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3. Bulk density: ……..

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4. Particle shape: ……..

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5. Crusting strength:

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6. Flowability: ……..

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7. Coefficient of friction: ……..

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Experiment No. 3

Objective: To determine total nitrogen in organic manure by Kjeldahl method

Nitro -compounds formed by the reduction of salicylic acid with NO3- in acid medium are reduced to corresponding amino compounds by heating the mixture with sodium thiosulphate and zinc dust. The main product of nitration is 5-nitro salicylic acid and small amounts of 3-nitro salicylic acid

Reagents: ……..

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Write the procedure for total nitrogen estimation of Organic manure

Sample digestion: ……..

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Sample distillation: ……..……..

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Write the chemical reaction during digestion: ……..

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Observations and calculation:

(i) Weight of the sample = 1.0 g
(ii) Digestion volume = 100 ml
(iii) Aliquot taken = 5 ml
(iv) 1 ml of N/200 H_2SO_4 = 0.00007 g N.
(v) Titration value (TV) = Sample TV - Blank TV
= X value

$$\text{Nitrogen content (\%)} = \frac{X \times (iv) \times (ii) \times 100}{(i) \times (iii)}$$

$$= \frac{X \times 0.00007 \times 100 \times 100}{1 \times 5}$$

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Experiment No. 4

Objective: To determine total nitrogen in urea fertilizer

Organic nitrogenous materials when digested with H_2SO_4 are oxidized to CO_2 and H_2O and their inorganic N is released. During digestion part of H_2SO_4 is reduced to SO, which in turn reduces nitrogenous materials to ammonia (NH_3). Ammonia combines with H_2SO_4 and forms $(NH_4)_2\ SO_4$ at the end of digestion. The NH_3 is distilled in alkaline medium and absorbed in standard acid. The excess of un-reacted acid is back titrated with standard alkali and the amount of ammonia (as N) is calculated from the volume of standard acid consumed.

Which reagents require for nitrogen estimation in Urea

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Write the procedure for nitrogen determination in Urea

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Calculations

$$\text{Nitrogen content (\%)} = \frac{1.401 \text{ X } (V1\ N1 - V2\ N2) - (V3\ N1 - V4N2)}{W} \text{ X df}$$

Where,

V_1 = ml of standard acid (HCl) taken in receiving flask for samples.

V_2 = ml of standard NaOH used in titrating samples.

V_3 = ml of standard acid (HCl) taken to receiving flask for blank.

V_4 = ml of standard NaOH used in titrating blank.

N_1 = Normality of standard acid (HCl).

N_2 = Normality of standard NaOH.

W = Weight of sample taken.

df = Dilution factor of sample.

Hint - 1000 ml of 1N HCl = 14.01 g N

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What are the points should kept in mind during sample analysis:

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Experiment No. 5

Objective: To estimate ammonical and nitrate nitrogen in ammonical fertilizer

Devarda alloy (50% Cu, 45% Al, 5% Zn) reduces NO_3 into NH_4 in an alkali condition. The NH_3 formed is then distilled from alkaline solution and absorbed in a standard acid. In the alloy, Zn and Al are the reducing agents while Cu increases bitterness of the alloy and prevent bumping during distillation.

Reagents required: …….

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Write the procedure: ……

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Calculations

$$NH_4 - N + NO_3^- - N\ (\%) = \frac{1.401\ X\ (V1\ N1 - V2\ N2) - (V3\ N1 - V4N2)}{W}\ X\ df$$

Where,

V_1N_1 is volume and normality of acid (HCl) taken for sample respectively.
V_2N_2 is volume and normality of alkali (NaOH) used for sample.
V_3N_1, is volume and normality of acid (HCl) taken in blank.
V_4N_2 is volume and normality of alkali (NaOH) used in blank titration.
df is dilution factor.
W is weight of sample taken for analysis.

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Write the precautions: ……..

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Experiment No. 6

Objective: To estimate water soluble phosphate in single super phosphate (SSP)

By repeatedly leaching the fertilizer sample with water through a filter paper, the water soluble phosphate is obtained in the filtrate. The soluble orthophosphate is precipitated by quimociac reagent as quinolinium phosphomolybdate ($(C_9H_7N)_3$, $H_3PO_4.12MoO_3$), which is dried at 250°C and weighed. The method is applicable to all fertilizers including fertilizer mixtures.

Reagents required: ……..

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Write the preparation of sample from SSP fertilizer: ……..

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Write the procedure for Phosphorus estimation: ……..

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Calculation

Water soluble phosphate (%) = $\frac{3.207 \text{ X A}}{\text{W}} \text{ X df}$

Where,

A = Weight of precipitating (after substraction of reagent blank precipitate)
W = Weight of sample in g.
df = Dilution factor (in present case 250 / 25 = 10)

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Experiment No. 7

Objective: To determine potassium in potassic fertilizers

Potassium from the fertilizer sample is first extracted with ammonium oxalate. The K in solution in precipitated with an excess of STPB as potassium tetraphenyl boron. The excess of STPB is back titrated with Benzalkonium chloride (Trade name Zephiran chloride) or quaternary ammonium salt using Clayton yellow as indicator.

Reagent required: ……..

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Write the procedure for K_2O determination from K- fertilizers

Extraction of K_2O: ……..

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Analysis of K_2O: ……

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Calculation

$$F = \frac{34.61}{(43.0 - \text{ml Benzakonium})} = \% K_2O/\text{ml STPB reagent}$$

Note: This factor applies to all fertilizers if 2.5 g sample is diluted to 250 ml and 15 ml aliquot is taken for analysis. If results are to be expressed as K rather than K_2O, substitute 28.73 for 34.61 in calculating the value of F.

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K_2O in fertilizer (%) = (ml STPB - Benzalkonium used) x F
Where F = % K_2O/ml of STPB reagent

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Experiment No. 8

Objective: To estimate calcium and magnesium in fertilizers by EDTA method

Both calcium and magnesium can be titrated directly with EDTA using eriochrome black (T) as indicator. However certain metals like Co, Ni, Cu, Zn and Mn cause interference which has to be either removed or masked with the use of hydroxyl amine hydrochloride or cyanide. It is also envisaged that some amount of Mg will be present in the solution otherwise a small amount of $MgCl_2$ is to be added before EDTA titration. After the combined determination of Ca + Mg, the Ca of the sample can be titrated with EDTA with the use of selective indicator calcein and the Mg can then be calculated by difference.

Reagents required and their preparation: ……..……..

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Write the procedure for Ca and Mg estimation from fertilizers

Preparation of sample: ……

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Titration for Ca + Mg:

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Titration for Ca: ……

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Calculations:

$$\text{Calcium titre (mg/ml)} = \frac{\text{Volume taken of calcium standard solution (ml)}}{\text{Volume of EDTA solution used}}$$

From calcium titre, calculate magnesium titre value as follows:

Magnesium titre = Calcium titre x 0.6064

$$\text{Mg (\%)} = \frac{(V1-V2)\times \text{Mg titre}\times 100}{\text{aliquot taken}}$$

$$\text{Ca (\%)} = \frac{(V2)\times \text{Ca titre}\times 100}{\text{aliquot taken}}$$

Where,

V_1 = Volume of EDTA used for titration of Ca + Mg (ml)

V_2 = Volume of EDTA used for titration of Ca (ml)

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Experiment No. 9

Objective: To determine sulphur in fertilizers by EDTA gravimetric method

The non-sulphate inorganic S content can be converted into sulphate by oxidizing with bromine H_2O_2 or concentrate nitric acid. The sulphates can be precipitated out with barium chloride as $BaSO_4$ which can be weighed gravimetrically by heating it at 800°C. Certain cations like Na^+, K^+, Ca^{2+}, Al^{3+}, Cr^{2+} and Fe^{3+} cause interference, which has to be removed or masked before precipitation.

Reagents required: ……..

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Write the procedure for determination of S in fertilizers:

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Procedure for S determination in SSP:

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Calculations

$$\text{Sulphur (S) content in fertilizer (\%)} = \frac{137.4 \times M}{W}$$

Where, M = Weight of precipitate in g
W = Weight of the sample taken in g

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Experiment No. 10

Objective: To estimate Zn in zinc sulphate

EDTA (Ethylenediamine Tetra Acetate) forms complexes with polyvalent cations including Zn. The other interfering elements are either removed or masked and zinc is titrated with EDTA. In this method, Zn is first masked by adding sodium cyanide as tetracyano-sodium zincate Na_2 Zn $(CN)_4$) and free Mg is titrated with EDTA at pH 10. Later the Zn is again de-masked with formaldehyde acetic acid and titrated against EDTA.

Reagents required: ……..……..

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Write the procedure for estimation of Zn:

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Calculations

Zinc content (%) = $\frac{10 \times V2}{V1}$

Where,

V_1 = Volume of EDTA solution (ml) used for 10 ml of standard Zn solution.

V_2 = Volume of EDTA solution (ml) used for titration of sample solution

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Experiment No. 11

Objective: To estimate organic carbon content in soil sample

Walkley-Black rapid titration method (wet combustion) is extremely useful. Soil organic carbon is oxidized to CO_2 in presence of an excess of oxidizing agent such as a mixture of potassium dichromate and sulphuric acid. It is actually the chromic acid formed by the action of potassium dichromate and sulphuric acid which oxidizes the carbon. The excess of dichromate is determined by blank titration with ferrous ammonium sulphate (redox-titration).

Reagents required: …….

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Write the procedure for organic carbon determination in soil: ……..

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Write the chemical during the analysis of organic carbon in soil:

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Calculations:

$$2K_2Cr_2O_7 + 8H_2SO_4 = 2K_2SO_4 + Cr_2(SO_4)_3 + 8H_2O + 6O$$

$$3C + 6O = 3CO_2$$

$$2K\ Cr\ O \xrightarrow{\text{evolve}} 6O \xrightarrow{\text{oxidise}} 3C$$

(2×294) g $K_2Cr_2O_7 \equiv (3 \times 12)$ g C

Hence; 49 g K Cr O $\equiv \frac{3 \times 12}{2 \times 294} \times 49 = 3$ g C

Now 49 g $K_2Cr_2O_7$ dissolved in 1 litre gives 1(N) – solution

i.e. 1000 ml 1(N) $K_2Cr_2O_7 \equiv 3$ g C

1 ml 1(N) K2Cr2O7 $\equiv 0.003$ g C

Alternatively,

$$\textbf{Organic Carbon (\%)} = \frac{\mathbf{10\ (B-T)}}{\mathbf{B}} \times \mathbf{0.003} \times \frac{\mathbf{100}}{\mathbf{w}}$$

where,

B = Volume (ml) of ferrous ammonium sulphate solution required for blank titration.

T = Volume (ml) of ferrous ammonium sulphate (ml) needed for sample titration

W = weight of soil sample in g

More precisely, Organic carbon value considering recovery factor of 0.77 can be calculated using the formula.

$$= \frac{10\ (B-T)}{B} \times 0.003 \times \frac{100}{w} \times \frac{1}{0.77} \times \frac{(100+m)}{100}$$

Where, m = air dry moisture

% organic matter = % organic carbon x 1.724

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Interpretations:

Low Organic carbon	Medium Organic carbon	High Organic carbon
< 0.50 %	0.50 – 0.75 %	> 0.75 %

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Experiment No. 12

Objective: To determine available nitrogen in soil

In the alkaline permanganate method (Subbiah and Asija, 1956) nitrogen is released by the alkaline permanganate solution and estimated by the usual ammonia distillation procedure, the distillate being absorbed in standard acid and excess acid back titrated with standard alkali using methyl red indicator.

Reagents required: ……..

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Write the procedure for nitrogen estimation in soil: ……..

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Calculations:

$$\text{Available nitrogen in soil (Kg/ha)} = \frac{(S-B)\times 0.00028}{20} \times 10^6 \times 2.24$$

$$= (S- B) \text{ X } 31.36$$

Where, S is the titre value for soil sample
B is titre value for blank sample
The factor 0.00028 is arrived at by

$2NH_4OH + H_2SO_4 = (NH_4)_2SO_4 + 2H_2O$

Or 98 g of H_2SO_4 (or 1L of 2N H_2SO_4) ≡ 28g N

Or 1 ml of 0.02N H_2SO_4 ≡ 0.00028 g N

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Interpretations

Low Nitrogen	Medium Nitrogen	High Nitrogen
< 280 Kg/ha	280 – 560 Kg/ha	> 560 Kg/ha

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Experiment No. 13

Objective: To determine available phosphorus in soils by Olsen method

An alkaline (pH 8.5) bicarbonate solution can repress the concentration of calcium ions by precipitation as calcium carbonate and of aluminium and ferric ions as hydroxides. Thus phosphate ions concentrations are increased and available phosphate can be extracted from soil by shaking with alkaline $NaHCO_3$ and filtering. The 0.5 (M) sodium bicarbonate adjusted to pH 8.5 actually controls the ionic activity of calcium, through the solubility product of calcium carbonate, during the extraction of calcareous soils.

Reagents required: …….

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Write the working procedure for P estimation in soil sample

Phosphorus extraction from soil: ……

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Color development and phosphorus detection:

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Write the chemical reactions during phosphorus estimation

During phosphorus extraction: ……..

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During color development: ……..

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Calculations:

$$\text{Available P (Kg/ha)} = \frac{Q \times V \times 2.24 \times 10^6}{A \times S \times 10^6} = \frac{Q \times V \times 2.24}{A \times S}$$

$$= Q \times 8.96$$

Where, Q = quantity P (Sample reading), V = volume of olsen's reagent used

A = Volume of aliquot used for color development, S = weight of sample (g)

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Interpretations

Low Nitrogen	Medium Nitrogen	High Nitrogen
< 12.5 Kg/ha	12.5 - 25 Kg/ha	> 25 Kg/ha

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What are the precautions should kept in mind during P analysis: ……..

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Experiment No. 14

Objective: To estimate available potassium in soil by neutral normal ammonium acetate method

The readily exchangeable plus water soluble K^+ is determined in the neutral normal ammonium acetate extract of the soil. The NH_4^+ ion provide a sharp and quick separation from the exchange sites while it determine by using flame-photometer.

Reagents required: …….

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Write the procedure for potassium determination in soil: ……..

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Calculations

$$\text{Available K (Kg /ha)} = R - B \times \frac{25}{5} \times 2.24 = R \times 11.2$$

Where,

R = ppm of K in the extract, obtained from the standard curve (soil sample reading).

B = reading of blank sample (without soil)

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Interpretations

Low Potassium	Medium Potassium	High Potassium
< 135 Kg/ha	135 - 335 Kg/ha	> 335 Kg/ha

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Experiment No. 15

Objective: To determine available sulphur in soil

When the soil solution is shaken with $CaCl_2$ (0.15 %), the chloride ions displace the adsorbed sulphate during extraction. The filtrate is analyzed for sulphur by turbidimetry method as outlined by Chesin and Yien (1950), in which turbidity produced due to the precipitation of SO_4^{-2} as $BaSO_4$ is measured on a spectrophotometer at a wavelength of 420 nm or corresponding to blue filter.

Reagents required: …….

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How would you prepare standard solution for S estimation in soil: ……..

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Write the working procedure for S estimation in soil: ……..

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Calculations:

Available S in soil (mg/Kg) = $S - B \times \frac{50}{10} \times \frac{1}{10}$

Where, S = Soil sample reading

B = Blank sample reading

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Interpretations:

Low Sulphur	Medium Sulphur	High Sulphur
< 11.25 mg/Kg	11.25 – 17.50 mg/Kg	> 17.50 mg/Kg

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Experiment No. 16

Objective: To determine available micronutrients (Fe, Mn, Zn and Cu) in soil

Lindsay and Norvill (1978) developed a method using DTPA (Diethylene triamine Penta Acetic Acid). This method has been adopted in all the laboratories engaged in the analyses of available Zn, Cu, Mn and Fe. Chelating agents offer great promise for assessing readily available micronutrient cations in soil. These agents combine with free metal ions in solution to from soluble complexes. DTPA is an efficient complexing agent for all the essential micronutrient cations, viz. Cu, Fe, Mn and Zn. With the advent of hollow cathode lamps along with precision detecting device all the estimation are now possible in a single extraction.

Reagents required: ……

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Prepare the extracting solution for Zn determination in soil: ……..

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Write the working procedure for Zn determination in soil sample: ……..

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Calculation

Micronutrients content in soil (mg/Kg or ppm) = R x 2
Where, R is the reading

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Interpretations:

Micronutrients	Deficient	Sufficient	High level
Available Fe (mg/Kg)	<4.5	4.5-9.0	> 9.0
Available Mn (mg/Kg)	<3.5	3.5- 7.0	> 7.0
Available Zn (mg/Kg)	<0.6	0.6- 1.2	> 1.2
Available Cu (mg/Kg)	<0.2	0.2- 0.4	> 0.4

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Experiment No. 17

Objective: To determine boron content in soil sample

In soil extract, B can be determined by azomethine- H method. The methods employs azomethine-H as the reagent to form a stable colored complex with H_3BO_3 at pH 5.1 in aqueous medium, which retains proportional absorbance – concentration properties for several hours independent of the presence of wide variety of salt. The maximum absorbance occurs at 420 nm.

Reagents required: …….

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Preparation of working standard solution: …….

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Write the procedure for B estimation in soil: ……..

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Calculations:

Boron content in soil (ug/g or ppm) = Sample (reading from the standard curve) × 10

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Interpretations:

Micronutrients	Deficient	Sufficient	High level
Available B (mg/Kg)	< 0.5	0.5 – 1.0	> 1.0

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Experiment No. 18

Objective: To determine chloride (Cl^-) content in soil sample

Chloride determination is based on the formation of nearly insoluble silver salts

$$Cl^- + Ag+ \rightarrow AgCl$$

(white spongy precipitate)

Silver nitrate in presence of potassium chromate indicator is used for precipitating Cl-

$$NaCl + AgNO_3 \rightarrow AgCl + NaNO_3$$

$$K_2CrO_4 + 2AgNO_3 \rightarrow Ag_2CrO_4 + 2KNO_3$$

Reddish brown ppt.

Reagents required: …….

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Write the procedure of Cl^- estimation in soil: ……

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Calculations;

meq of Cl^- per litre of soil extract = $(N_A \times T) \times (\frac{1000}{V})$

meq of Cl^- per 100 g soil = $(N_A \times T) \times (\frac{200}{V}) \times \frac{100}{40}$

Cl^- (mg /100 g soil) = $(N_A \times T) \times (\frac{200}{V}) \times \frac{100}{40} \times 35.5$

Note :1 ml of 0.02 N $AgNO_3$ (0.02 meq. $AgNO_3$) = 0.00071 g Cl.

Where,

Volume of aliquot taken (from soil extract or water sample) = V ml.

Volume of $AgNO_3$ solution used in titration = T ml

Normality of $AgNO_3$= 0.02 (N) or N_A

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APPENDICES

SAMPLING

MANURE:

Material required: Spade/ Khurpi, Marker, Kin box/ screw capped jars, Stainless steel sieve 2 mm, screw capped jars
Procedure:

- Any material such as pieces of broken glasses, stones, metal, plastics etc should be removed at the time of sampling.
- Five hundred grams each from seven to eight sites from different depth of the manure pile should be collected. These should be mixed together to form a composite sample.
- Sub-sampling is done by spreading the manure in a circular disc-like shape and divided into four equal parts by the method known as quartering. The material in the opposite quarters is discarded and that in the remaining quarters is mixed well. This process is repeated till the sample size is reduced to approximately 500 g.
- The collected sample should be stored in a polythene bag in a refrigerator before analysis.
- In case of oilcakes, the sample is to be air dried, then powdered and passed through a 2 mm sieve before analysis. The analysis is carried out in exactly the same way as in the case of FYM etc.
- Samples of press mud from sugar factories or press mud - spent wash compost can be drawn from 7-8 sites and for making the final sample.
- For sampling of Vermicompost, the living earthworms have to be separated from the compost. Vermicompost is heaped in the form of a cone under light or sunlight on a plastic sheet or on a clean surface. The earthworms move to the bottom of compost mass and can be removed.

Prepare sample for analysis

- Analyses for parameters such as pH, ammoniacal nitrogen, nitrate nitrogen, acid extractable phosphorus, micro-flora and fauna should be done on moist samples.
- Manure samples are usually partially air-dried at a temperature between 25°C to 35°C in the shade.
- The air-dry sample is ground to pass 2 mm sieve and stored in screw capped jars.
- Analysis should be carried out as soon as possible to avoid undue chemical changes subsequent to sampling.
- The microbiological analysis should be done as soon as possible using moist samples.

SOLID FERTILIZER

Material required: Glass or screwed hard polyethylene bottle, Single or double tube probe, Sampling cup**.**
Procedure: Appropriate procedure is required for sampling from the bulk material at discharge ports or at manufacturer's godowns at the production site, special care is also needed during sampling from bagged materials stored in large godowns of the producers/importers or small godowns of the traders.

- Number of bags to chosen from a lot depend on the lot size. For example: if the lot consists of 10 bags, one bag is chosen for sampling. The number of bags chosen increases as the size of lot increases as the size of lot increases reaching to 10 bags from a lot size of 1601-2000 bags as specified in the FCO.
- The identified sampling bags should be laid horizontally and mixed thoroughly.
- The sampling probe should be inserted diagonally from one corner to another keeping the slit down and rotated while withdrawing.
- Piercing at two places is required from each selected bag.

- All samples thus drawn are to be mixed together and reduced by the process of quartering or riffling to about 1.2 kg.
- Samples should be kept in clear air tight containers with identification marks.

Prepare the sample for analysis.

- The collected sample should be ground quickly to avoid any absorption of moisture and grinding and repeated sieving by <1 mm.
- However, fertilizers whose particle size is already less than 1 mm, only thorough mixing is needed to make a homogeneous mass.
- Sample should be ground in a porcelain pestle and mortar to pass through 0.425 mm IS sieve to make it more homogenous.
- The prepared sample should be kept in an air tight glass bottle for further analysis.

LIQUID FERTILIZERS

Material required: Glass or polyethylene bottles (capacity 500 ml), Nylon cord, Missouri type sampling bottles (500 ml)
Procedure:

- Mix fertilizer solution from storage tank or delivery tank thoroughly.
- Flush direct tap or delivery line and collect sample in a glass or polyethylene bottle.
- Alternatively, lower the sample bottle by a nylon cord from the top opening of the storage tank to the bottom of the tank.
- Raise slowly the sample bottle while filling full.
- Transfer to sample container and seal tightly.
- Repeat process to obtain such representative samples.

PRECAUTIONS DURING SAMPLING OF FERTILIZERS

- The samples should not be taken at a place exposed to the rain/sun.
- The sampling instrument should be clean and dry and the sampling container should be free from any contaminations.
- The contents of each bag selected for sampling should be mixed as thoroughly as possible by suitable means to make it homogenous.
- The samples should be kept in suitable, clean, dry and air tight glass or screwed hard polyethylene bottle of about 400 g capacity or in a thick gauge polyethylene bag.

PHYSICAL PROPERTIES OF ORGANIC MANURE AND FERTILIZER

ORGANIC MANURE

Temperature and heat output: The maturity of a manure / compost can be judged by recording the temperature of the finished compost. It should also be free of flies, pathogens and weed seeds.

Color: Mature compost should be dark brown to black in color irrespective of raw material used. Aerobic composting is characterized by progressive darkening of color while the decomposition of biomass under anaerobic conditions pale green which shows minor changes with progressive composting.

Odour/smell: Mature compost smells like forest soil (earthy smell). This is caused by two gases (geosmin and 2-methyl lisoborneol) which are produced by fungi and actinomycetes. However, color and odour are too subjective to provide accurate estimate of maturity. Any foul smell is not a good sign because it indicates that the composting process is not complete.

Structure: The material should be crumbly, moderately loose, neither compact nor lumpy.

Moisture status: The approximate moisture status of compost can be judged by inserting an iron rod at different depth in heaps or windrows. The rod would be quite moist if moisture is more than required. If it is pressed by hand, no water should drip from the sample.

pH: The pH of a good quality compost should be between 6.5 and 7.5. Nitrogen fixing and phosphate solubilizing bacteria can thrive well and multiply in this pH range.

Standards for assessing the quality of compost

Criteria	Good quality	Poor quality
Colour	Brown to black	varies
Odour (smell)	Earthy smell	Foul odour
pH	6.5- 7.5	Below 6 or above 8
C:N ratio	10:1 – 20: 1	>20:1
Moisture	About 20%	>30%
Temperature	30- 40 °C	>45 °C
Humus	6 – 8 %	<4%
Nitrogen	>1.25%	<1%

FERTILIZERS

Particle Size: Particle size has an exponential impact on spread width and to the risk of product segregation. In general, larger particles are thrown wider distance by a spreader than smaller particles. For an example, urea with a particle diameter of 4.7 mm can have a spread width of 65 ft whereas 1.7 mm has a spread width of only 33 ft. Fertilizer with very small particles, will be difficult to evenly spread with small particles (e.g. dust and fines).

Particle Density: Particle density indicates the mass to volume ratio of particles and is reported as kg/m^3. Beyond particle size, particle density of a fertilizer must be taken into consideration for spreader setup and evaluation of the risk of segregation for blended products.

Bulk Density: Bulk density represents the mass to volume ratio of a bulk sample including the space between individual particles and is reported as kg/m^3. However, bulk density which one to use when spreading fertilizer.

Particle Shape: Particle Shape can vary among fertilizers which can be classified as round (spherical or egg-shaped), cubic, rectangular and irregular. Urea and DAP are examples of spherically shaped fertilizers, whereas potash is irregularly shaped. Shape can influence behavior of material during conveyance and distribution.

Crushing Strength: Crushing strength is defined by the International Fertilizer Development Center as the resistance of granules to deform or fracture under pressure (IFDC, 1986). Crushing strength is especially helpful in gauging handling and storage properties of a granular material and determining the pressure limits applied during bag and bulk storage. Hardness or strength can govern the reaction of fertilizers to handling, transportation, storage and application. Crushing strength may be reported, but can also be reported in the form of particle hardness. A simple finger test can be used to evaluate hardness or strength at time of spreading. Granule crushed between thumb and forefinger is "soft"; spinner disc speed usually < 700 rpm. Granule crushed between forefinger and a hard surface is "medium hard"; spinner disc speed usually 700-800 rpm. Granule not crushed between forefinger and hard surface is "hard"; spinner disc speed usually > 800 rpm.

Flowability: Flowability refers to a material's ability to flow under humid conditions, so is an important property to consider during handling, metering and deposition of fertilizers. Flowability can affect the accuracy of metering and placement. More flowable materials can be metered at higher flow rates and their particles will tend not to stick together or bridge during conveyance. As humidity increases, less flowable materials will stick together, making them difficult to meter and evenly apply. Poor flow increases particle segregation and reduces spread width.

Coefficient of Friction: The coefficient of friction is the degree of friction experienced between a material and another surface such as spinning disc(s), ground surface and air etc. A higher degree of friction will result in longer contact with the spinner discs resulting in a larger departure angle and a more uneven spread. The coefficient of friction and particle shape are directly related to how and when a granular fertilizer particle will exit the spreader.

DETERMINATION OF TOTAL NITROGEN IN ORGANIC MANURE BY KJELDAHL METHOD

Reagents

- Sulphuric acid-salicylic acid mixture: 1 g of salicylic acid is added to 30 ml of concentrated H_2SO_4.
- Sodium thiosulphate.
- Sulphate mixture: 20 parts of K_2SO_4 + 1 part of catalyst mixture (or 20 parts $CuSO_4$ and 1 part selenium powder).
- 40% NaOH
- 4% boric acid

Procedure

- 1g sample of the organic manure is transferred to a 100 ml Kjeldahl flask.
- Add 20 ml sulphuric acid- salicylic acid mixture is added and swirled gently so as to bring the dry manure sample in contact with the acid.
- It is allowed to stand overnight. This treatment binds the nitrate nitrogen in organic combination.
- After reduction, next day, 5 g of sodium thiosulphate is added and heated gently for about 5 minutes. Precaution is taken to avoid frothing. This reduces the nitrate group to form amino-salicylic acid.
- The contents are then cooled, 10 g sulphate mixture is added to the flask and digested on the Kjeldahl apparatus at full heat. The digestion is continued for 1 hour after the solution is cleared. Bumping during digestion can be avoided by the addition of glass beads. All forms of nitrogen are converted to ammonium sulphate which when distilled with 40% sodium hydroxide solution gives ammonia which is absorbed in standard boric acid.

Reactions

(i) $2KNO_3 + H_2SO_4 \rightarrow K_2SO_4 + 2HNO_3$

(ii) $HNO_3 + HO\text{-}C_6H_4.COOH \rightarrow HO\text{-}C_6H_3NO_2.COOH + H_2O$
(Nitro-salicylic acid)

(iii) $Na_2S_2O_3 + H_2SO_4 \rightarrow Na_2SO_4 + H_2SO_3 + S$

(iv) $H_2SO_3 + HO\text{-}C_6H_3NO_2.COOH + H_2O \rightarrow 3H_2SO_4 + HO\text{-}C_6H_3\ NH_2.COOH$
(Amino-salicylic acid)

(v) $(NH_4)_2SO_4 + 2NaOH \rightarrow 2NH_3 + H_2O + Na_2SO_4$

Distillation

- After digestion, the digest is cooled and diluted with distilled water to make up the volume to 100 ml.
- 10 ml of the digest is transferred to a vacuum jacket of micro-Kjeldahl distillation.
- In a conical flask, 10 ml of 4 % boric acid containing bromocresol green and methyl red indicator to which the condenser

outlet of the flask is dipped.

- The aliquot is added and the funnel of the apparatus is washed with 2-3 ml of deionised water and 10 ml of 40 % NaOH solution is added.
- 5 ml aliquot is distilled to the flask containing 10 ml boric acid.
- After completion of distillation, the boric acid is titrated against N/200 H_2SO_4 until purple color starts appearing.
- A blank determination of nitrogen contained in all reagents used should be carried out simultaneously to the same end point as that of the sample

DETERMINATION OF TOTAL NITROGEN IN UREA FERTILIZER

Reagents

1. Sulphuric acid – H_2SO_4 (93-98%)
2. Copper sulphate - $CuSO_4.H_2O$, AR grade
3. Potassium sulphate or anhydrous sodium sulphate - AR grade
4. 45% sodium hydroxide (NaOH) solution: Dissolve 450 g solid NaOH in water and dilute to one liter.
5. 0.1N NaOH. Dissolving 4.0 g NaOH in water & make volume to 1 liter. Standardise against 0.1N potassium hydrogen pthalate.
6. Zinc granule - AR grade.
7. 0.1N HCl or 0.1N H_2SO_4, Prepare standard 0.1N solution and standardize against 0.1N sodium carbonate or borox.
8. Methyl red indicator solution.

Procedure

- Weigh 1 g sample and place in Kjeldahl flask.
- Add 0.7 g copper sulphate, 15 g K_2SO_4 or anhydrous Na_2SO_4 and 30 ml H_2SO_4
- Place flask in inclined position and heat gently until frothing ceases. If necessary, add small amount of paraffin to reduce frothing.
- Boil until solution is clear and then continue digestion for at least 30 more minutes.
- Remove from burner and cool, add 200 ml water and transfer to a 500 m volumetric flask. Cool and dilute to mark.
- Transfer a 25 ml aliquot to distilling flask and add about 300 ml of water.
- Take accurately 20-25 ml standard acid (0.1N HCl) in the receiving conical flask so that there will be an excess of at least 5 ml of 0.1N acid. Add 2-3 drops of methyl red indicator. Add enough water to cover the end of the condenser outlet tube.
- Add a few Zn granules to distillation flask to prevent bumping tilt the flask and add gently 30 ml of 45% NaOH.
- Immediately connect distillation flask to distillation unit and swirl to mix the content. Distill at moderately high heat till at least 150 ml of distillate has been collected. Test with red litmus paper if any NH_3 is still coming out.
- Remove receiving flask and rinse outlet tube into receiving flask with a small amount of distilled water.
- Titrate excess standard acid in distillate with 0.1 N NaOH. Determine blank on reagent using same quantity of standard acid in a receiving conical flask.

Precautions

- The material after digestion should not solidify.
- No NH_3 should be lost during distillation.
- If the indicator changes color during distillation, repeat the determination using either a smaller sample weight or a larger volume of standard acid.

ESTIMATION OF AMMONIACAL AND NITRATE NITROGEN IN AMMONIACAL FERTILIZER

Reagents

- Standard 0.1N NaOH.
- Standard 0.1N HCl or H_2SO_4
- NaOH-45% solution.
- Methyl red indicator solution.
- Devarda alloy

Procedure

- Dissolve 1 g of prepared sample in water, filter if required and make the volume to 250 ml.
- Transfer 25 ml aliquot to a distilling flask, add 300 ml water, 3 g Devarda alloy and 5 ml 45% NaOH solution pouring the latter down the side of flask so that it does not mix at once with contents.
- Immediately connect with distillation unit immersing the outlet tube of condenser in 20-25 ml standard acid containing 2-3 drops methyl red. After 15 minutes, mix the contents of distilling flask by rotation.
- Heat slowly at first and then at a rate to yield 250 ml distillate in an hour. Collect distillate and titrate with standard alkali. Carry out the blank sample.

Precautions: Copper present in Devarda alloy itself works as an anti-bumping agent. However, 1-3 ml of tributyl citrate or 1 g of paraffin can be used as an anti-foaming agent, if required.

ESTIMATION OF WATER-SOLUBLE PHOSPHATES IN SINGLE SUPER PHOSPHATE (SSP)

Reagents: 1. Quimociac reagent: Dissolve 70 g of sodium molybdate dehydrate in 150 ml water. Dissolve 60 g citric acid in mixture of 85 ml HNO_3 and 150 ml water and cool. Gradually add sodium molybdate solution to citric acid-nitric acid mixture with stirring. Dissolve 5 ml synthetic quinoline in mixture of 35 ml HNO_3 and 100 ml water.

2. Nitric acid AR grade (Phosphorus free).

Preparation of sample solution: Place 1 g prepared sample on 9-cm filter paper No.1 or 5 in a funnel in a 250 ml volumetric flask. Using a fine stream of water directed in a circular path around the entire periphery of the filter paper, wash the sample with 10-15 ml portions until 240-250 ml filtrate has been collected within one hour. Use suction if washing would not otherwise be complete within one hour. Ensure that the water and sample are thoroughly mixed with each washing and allow each portion of water to pass through the filter before adding the next portion. If the filtrate is turbid, add 1-2 ml of HNO_3 and dilute to 250 ml and mix.

Procedure for P_2O_5 estimation

- Pipette an aliquot containing not more than 25 mg P_2O_5 (usually 25 ml) into a 500 ml beaker. Dilute if necessary, to 500 ml.
- Add 10 ml HNO_3 (1+1) and boil gently for 10 minutes to hydrolyze non orthophosphates. Cool and dilute to 100 ml with water.
- Add 50 ml quimociac reagent, shake well, cover with watch glass, place on hot plate and boil for 1 minute. (Caution: do not use open flame).
- Cool to room temperature swirl carefully 3-5 times during cooling.
- Allow the precipitate to settle and filter through sintered glass crucible no. 4 After complete transfer, wash the precipitate 5 times using 25 ml of water for each wash. Use vacuum pump, suck dry between washings.
- Place the crucible in a drying oven and dry at 250°C for 30 minutes. Cool in desiccator and weigh as $(C_9H_7N)_3$, $H_3PO_4.12MoO_3$.
- Carry out reagent blank and subtract the weight of reagent in blank precipitate

DETERMINATION OF POTASSIUM IN POTASSIC FERTILIZERS

Reagents

1. NaOH (20%). Dissolve 20 g NaOH in 100 ml distilled water.
2. Formaldehyde (37%) solution.
3. Sodium tetraphenyl boron (STPB) solution (1.2%). Dissolve 12 g sodium tetraphenyl boron in approximately 800 ml water. Add 20-25g Al $(OH)_3$, stir for 5 minutes and filter through Whatman No.42 paper or equivalent into a 1 liter volumetric flask. Rinse the beaker sparingly with water and add to filtrate. Collect entire filtrate; add 2 ml 20 % NaOH solution, dilute to volume with water and mix. Let it stand for 48 hours and standardize (so that 1 ml STPB = 1 % K_2O), prepared solution should be store at room temperature.
4. Benzalkonium chloride (BAC) or Quaternary ammonium chloride solution (approximately 0.625 %). Dilute 50 ml of 12.8 % BAC to 1 liter with water, mix and standardise. Cetyl trimethyl ammonium bromide may be substituted for BAC. If other concentration is used, adjust volume accordingly.
5. Clayton yellow, 0.04 %. Dissolve 40 mg in 100 ml water.
6. Ammonium oxalate solution $(NH_4)_2 C_2O_4$, 4%.

Standardization of solutions

1. **Benzalkonium chloride (BAC):** Take 1.0 ml STPB solution in 250 ml erlenmeyer flask, add 20-25 ml water, 1 ml 20% NaOH, 2.5 ml HCHO, 1.5 ml of 4 % ammonium oxalate and 6-8 drops of clayton yellow indicator. Titrate to pink end point with BAC solution so that 2.0 ml = 1.0 ml STPB solution.
2. **Sodium tetraphenyl boron solution:** Dissolve 2.5 g of KH_2PO_4 in water in 250 ml volumetric flask, add 50 ml 4% ammonium oxalate solution, dilute to volume with water and mix. Transfer 15 ml aliquot (51.92 mg K_2O or 43.10 mg K) in 100 ml volumetric flask add 2 ml of 20% NaOH, 5 ml HCHO and 43 ml STPB reagent. Dilute to volume with water and mix thoroughly. Let is stand for 5-10 minutes and pass through dry filter No. 42. Transfer 50 ml aliquot of filtrate in the 250 ml erlenmeyer flask, add 6-8 drops of indicator (clayton yellow) & titrate excess STPB with BAC solution to pink end point

$$F = \frac{34.61}{(43.0 - \text{ml Benzakonium})} = \% K_2O/\text{ml STPB reagent}$$

Note: This factor applies to all fertilizers if 2.5 g sample is diluted to 250 ml and 15 ml aliquot is taken for analysis. If results are to be expressed as K rather than K_2O, substitute 28.73 for 34.61 in calculating the value of F.

Procedure

Preparation of sample solution

- Straight potassium fertilizers (MOP, SOP, Potassium magnesium sulphate and Kainite). Dissolve 2.5 g prepared sample and dilute to 250 ml without adding NH_4OH and $(NH_4)_2C_2O_4$ When interfering substances such as NH_3, Ca, Al are

present, these have to be removed before precipitation.

- Place 2.5 g of prepared sample in 250 ml volumetric flask.
- Add 125 ml water and 50 ml of 4% $(NH_4)_2C_2O_4$ solution. Add 1 ml diglycol stearate to prevent foaming, if needed.
- Boil for 30 minutes, add slight excess of NH_4OH and after cooling dilute to 250 ml, mix and pass through dry filter No. 12 or equivalent.

Analysis

- Transfer 15 ml aliquot of sample solution in 100 ml volumetric flask and add 2 ml 20% NaOH and 5 ml HCHO.
- Add 1 ml standard STPB solution for each 1% K_2O expected in sample plus additional 8 ml excess to ensure complete precipitation
- Dilute to volume with water, mix thoroughly, let it stand for 5-10 minutes and pass it through dry filter paper Whatman No.12 or equivalent.
- Transfer 50 ml filtrate to 250 ml erlenmeyer flask, add 6-8 drops of indicator (clayton yellow) & titrate excess STPB with standard Benzalkonium solution to pink end point.

Suggestions

- Do not premix NaOH and formaldehyde solution as such mixtures are not stable and lose their power to complex NH_4^+.
- Acetone is a good solvent for the tetraphenyl boron precipitates. Therefore, its use also helps in cleaning glassware.
- While calculation the factor as % K_2O/ml of STPB, the figure of 34.61 is the actual percentage of K_2O present in standard KH_2PO_4

ESTIMATION OF CALCIUM AND MAGNESIUM IN FERTILIZERS BY EDTA METHOD

Reagents

1. Buffer solution (pH 10.0). Dissolve 67.5 g ammonium chloride in 200 ml of distilled water, add 570 ml ammonia solution and dilute to 1 liter.
2. Potassium hydroxide-potassium cyanide solution. Dissolve 280 g potassium hydroxide and 66 g potassium cyanide in 1 liter of distilled water.
3. Potassium cyanide solution (2%). Dissolve 2 g potassium cyanide in 100 ml of distilled water.
4. Eriochrome black T indicator solution. Dissolve 0.2 g of indicator in 50 ml of methyl alcohol containing 2 g of hydroxyamine hydrochloride.
5. Calcium standard solution (1 mg/ml). Dissolve 2.4973 g calcium carbonate (primary standard grade, previously dried for 2 hours at 285°C) in HCl (1+10). Dilute to 1 liter with double distilled water.
6. Calcein indicator mixture - Grind together 1 g calcein indicator with 10 g charcoal and 100 g potassium chloride.
7. Disodium dihydrogen ethylene diamine tetra acetic acid (EDTA) standard solution (0.4%). Dissolve 4 g Na_2H_2 EDTA in 1 liter of distilled water.
8. Triethanolamine
9. Potassium ferrocyanide solution (4%). Dissolve 4 g potassium ferrocyanide in 100 ml of distilled water.

Standardizations of the calcium solution

- Pipette 10 ml calcium standard solution into 250 ml Erlenmeyer flask. Add 100 ml of distilled water, 10 ml KOH-KCN solution, 2 drops of triethanolamine solution, 5 drops of potassium ferrocyanide solution and 15±1 mg of calcein indicator.
- Immediately place the flask on a magnetic stirrer in front of day light fluorescent light and white background. While stirring, titrate with EDTA solution to disappearance of all fluorescent green & until solution remains pink.

Estimation of Ca and Mg in sample solution

- Prepare the sample solution by weighing 1 g fertilizer sample into 250 ml volumetric flask. Add 200 ml distilled water and boil for 30 minutes. Cool and dilute to volume with water and mix.

Titration for Ca + Mg

- Pipette 25 ml of aliquot in a 250 ml Erlenmeyer flask.
- Dilute with 100 ml of distilled water. Add 5 ml of buffer solution (pH 10), 2 ml potassium cyanide solution, 2 drops of triethanolamine solution, 5 drops of potassium ferrocyanide solution 8 drops of Eriochrome black T indicator solution.
- Titrate immediately with EDTA solution, stirring and lighting as standardization. Color changes are wine red, purple, dark blue to clear blue to end point. It becomes green if over-titrated. Note the volume of EDTA used in ml (V_1).

Titration for Ca

- Pipette 25 ml of aliquot in an 250 ml Erlenmeyer flask.
- Dilute with 100 ml of water. Add 10 ml KOH-KCN solution, 2 drops of triethanolamine solution, 5 drops of potassium ferrocyanide solution and ± 1 mg of calcein indicator.
- Titrate immediately with EDTA solution as in standardization. Note the volume of EDTA used in ml (V_2).

DETERMINATION OF SULPHUR IN FERTILIZERS BY EDTA METHOD

Reagents

1. Concentrated Hydrochloric acid
2. Concentrated Nitric acid
3. Barium chloride 2% solution in water.
4. Silver nitrate solution 5%

Procedure: (for nitrate free samples such as ammonium sulphate, potassium sulphate, zinc sulphate, copper sulphate, ferrous sulphate, manganese sulphate, NP an NPK complexes and mixtures).

- Weigh about 2.5 g sample and transfer to 250 ml capacity volumetric flask and make up the volume with dilute HCl. Shake well and filter through Whatman filter paper No. 40 or equivalent paper in a dry beaker if the solution is not clear and transparent.
- Take 25 ml of filtered aliquot in a beaker of 250 ml capacity. Add 100 ml water and heat to boil. While stirring, add in a slow stream 1 ml of hot barium chloride solution for each one percent of S expected in the sample plus additional 10 ml in excess to ensure complete precipitation of S as barium sulphate. Boil for one minute.
- Digest the precipitate on a hot plate or water bath for 2 hours at low temperature such that the solution does not boil. Ensure that the supernatant liquid is clear and transparent. Cool to room temperature. Filter into a 30 ml capacity G4 grade sintered Gooch crucible previously dried at 250°C cooled and weighed. Wash the precipitate 10-12 times with hot water to ensure that the precipitate is free from barium chloride.
- Dry the crucible and its content at 250°C for two hours in a furnace. Cool to room temperature in a desiccator to a constant weight.

Procedure for sulphur in SSP

- Weigh about 2.5 g of sample and transfer to a beaker of 250 ml capacity. Add 250 ml of concentrated hydrocloric acid and 25 ml of water. Heat to boil the solution gently.
- Boil for 5 minutes and cool. Add 75 ml of water and transfer quantitatively into a 250 ml volumetric flask with dilute HCl and make up the volume.
- Shake well & filter about 50 ml through Whatman filter paper no. 40 or equivalent. Then proceed as previous sample.

ESTIMATION OF ZN IN ZINC SULPHATE

Reagents

1. EDTA solution: Dissolve 7.44 g of disodium ethylenediamine tetra acetate dihydrate in water and make up the volume to 1 liter in a volumetric flask.
2. Standard zinc solution
3. Ammonium hydroxide (20%) .
4. Eriochrome black T indicator. Mix thoroughly 1 g of Eriochrome black T with 100 g of sodium chloride.
5. Ammonium chloride.
6. Sodium cyanide
7. Formaldehyde acetic acid solution (4%): Dissolve 100 ml of formaldehyde (37-40%) in about 100 ml of distilled water. Add 40 ml glacial acetic acid and make the volume to 1 liter with distilled water.
8. Hydroxylamine hydrochloride: AR Grade solid salt or ascorbic acid.

Standardizations of the EDTA solution: Take 10 ml of standard zinc solution in 500 ml beaker. Add about 0.1 g of ammonium chloride and 30 ml of ammonium hydroxide solution 20% and 30 ml distilled water. Add a pinch of Eriochrome black (T) indicator. Titrate it with EDTA solution to obtain clear blue end point. Note the volume of EDTA used in ml (V_1).

Procedure for estimation of Zn

- Dissolve accurately weighed 1.0 g sample in 100 ml of distilled water in a volumetric flask.
- Take 10 ml of aliquot in a beaker. Add 0.1 g of hydroxylamine hydrochloride or ascorbic acid and 0.1 g of ammonium chloride
- Carefully add small quantity of sodium cyanide. A white precipitate will if zinc is present. Continue adding sodium cyanide till white precipitate disappears while swirling the beaker. Add about 0.5 g excess of sodium cyanide.
- Add about 20 ml of ammonium hydroxide (20%) and about 30 ml of distilled water. Add a pinch of eriochrome black (T) indicator. It will give red color.
- Titrate with EDTA solution till there is a sharp change to violet color,
- Add 20 ml of formaldehyde acetic acid solution into the above titrated solution and mix. Red color will reappear. Titrate it with EDTA solution to get blue end point without a red tinge. record the volume of EDTA used in titration in ml (V_2).

ESTIMATION OF ORGANIC CARBON CONTENT IN SOIL SAMPLE

Reactions

Oxidation of Carbon

$$K_2Cr_2O_7 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3O \quad (\times 2)$$

$$3C + 60 \rightarrow 3CO2$$

$$2K_2Cr_2O_7 + 8H_2SO_4 + 3C \rightarrow 2K_2SO_4 + 2Cr_2(SO_4)_3 + 8H_2O + 3CO_2$$

Titration

$$FeSO_4(NH_4)_2\ SO_4 . 6H_2O \rightarrow FeSO_4 + (NH_4)_2\ SO_4 + 6H_2O \quad (\times 2)$$

$$2FeSO_4 + H_2SO_4 + O \rightarrow Fe_2(SO_4)_3 + H_2O$$

$$2FeSO_4(NH_4)_2\ SO_4 . 12H_2O + H_2SO_4 + O \rightarrow 2(NH_4)_2SO_4 + Fe_2(SO_4)_3 + 13H_2O$$

Action of diphenylamine indicator

$$2C_6H_5NHC_6H_5 \xrightarrow[-H2O]{[O]} 2(C_6H_5 . NHC_6H_4) \xrightarrow[-H2O]{[O]} C_6H_5N\text{-}C_6H_4C_6H_4N\text{-}C_6H_5$$

Diphenylamine — diphenyl benzidine (violet)

Reagents

- Standard potassium dichromate solution 1(N). Heat, $K_2Cr_2O_7$ in an air oven for 4 hours at 105°C. Dissolve 49.04 of pure $K_2Cr_2O_7$ in distilled water and dilute to one litre.
- Ferrous ammonium sulphate (N/2). Dissolve 196 g ferrous ammonium sulphate, $FeSO_4(NH_4)_2\ SO_4.6H_2O$ in water. Add 25 ml of concentrated H_2SO_4 and dilute to one litre.
- Redox indicator. Use any one of the following:
  a) Diphenylamine indicator: Dissolve 0.5 g of diphenylamine in a mixture of 100 ml conc. sulphuric acid and 20 ml distilled water and store in a colored bottle.
  b) Ferroin indicator: Dissolve 1.485 g 1,10 phenanthroline monohydrate in about 80 ml water (warm if necessary and then cool) and added 0.69 g ferrous sulphate heptahydrate. Stir to dissolve and dilute to 100 ml.
- Concentrated sulphuric acid (sp.gr.1.84), 96% concentration or more

- Orthophosphoric acid (85%).

Procedure

- Take 1.0 g of the soil sample (weighed to the nearest milligram) into a clear and dry 500 ml conical flask.
- Add 10 ml of 1(N) $K_2Cr_2O_7$ by means of a pipette and swirl gently.
- Then add 20 ml of concentrated H_2SO_4 rapidly into the solution and immediately mix by swirling gently at first and then vigorously for a total of one minute.
- Keep the flask on an asbestos pad for 30 minutes.
- Add 200 ml of distilled water and add 10 ml of orthophosphoric acid also add 1ml of diphenylamine indicator. A blue violet color will appear.
- Titrate with ferrous ammonium sulphate solution till color changes from blue violet to green (If ferroin indicator is used the colour change at the endpoint is from blue to red).
- If more than 8 ml of the dichromate solution is consumed repeat the estimation with a smaller quantity (0.25 – 0.50g) of the soil sample.
- Simultaneously carry out a blank determination using all the reagents similarly but no soil sample.

DETERMINATION OF AVAILABLE NITROGEN IN SOIL

Reagents

- 0.32% potassium permanganate solution–freshly prepared
- 2.5% sodium hydroxide solution–freshly prepared
- Standard sulphuric acid 0.02(N)
- Standard sodium hydroxide 0.02(N).
- Methyl red indicator (Dissolve 1 gm methyl red in 200 ml of rectified spirit)
- Liquid paraffin (extra pure)
- Glass beads

Procedure

- Weigh accurately 20 g of the soil sample in a distillation flask.
- Add 20 ml of distilled water, 100 ml of potassium permanganate solution and 100 ml of 2.5 percent sodium hydroxide solution (the frothing during boiling is prevented by adding liquid paraffin (1 ml) and bumping by adding a few glass beads.
- Immediately after alkali addition connect to the distillation apparatus and distill the contents in Kjeldahl assembly at a steady rate.
- Pipette out 25ml of standard sulphuric acid (0.02N) in a conical flask.
- Add methyl red indicator and dip the end of delivery tube in it.
- Distil the ammonia gas from the distillation flask for about 30–40 minutes until distillation is completed and collect about 100 ml of the distillate.
- Back titrate the excess acid with standard alkali i.e. 0.02 N NaOH. (Color change at end point is usually pink to faint yellow or straw).
- Perform a blank without sample.

DETERMINATION OF AVAILABLE PHOSPHORUS IN SOILS BY OLSEN METHOD

Reagents

- Olsen's reagent; 0.5 M Sodium bicarbonate solution (pH 8.5); Dissolve 42.0 g $NaHCO_3$ (L.R.) in double distilled water to give one litre of the solution. Adjust the pH to 8.5 with small amounts of 10% NaOH.
- Activated charcoal (free of P) or Darco G-60
- Dickman and Bray's chloromolybdic acid reagent; Weigh 15g of ammonium molybdate (AR) and dissolve in 300 ml of warm water (50°C), cool and filter if necessary. To this, add 400 ml of 10N HCl and make up the volume to one litre. Mix thoroughly and store in an amber glass stopper bottle.
- Stannous chloride solution; Stannous chloride solution; Dissolve 10g of crystalline stannous chloride by warming and store in an amber coloured bottle, carefully avoiding contact with air. This is 40% stock solution of $SnCl_2$. Just before use prepare freshly diluted stannous chloride. Pipette 1 ml 40% $SnCl_2$ into 330 ml of double distilled water (A piece of tin metal (AR) added to the stock solution will preserve the stock solution for a long time).

Procedure

- Accurately weigh 2.5 g of the soil sample in a 100 ml conical flask and to it add 50 ml of Olsen's reagent.
- Add 1 teaspoon of phosphorus free charcoal.
- Shake the suspension for 30 minutes on a platform type shaker.
- Filter the solution through Whatman 40 dry filter paper into clean and dry beakers. Perform a blank without soil (if the filtrate is not clear, return it to the conical flask containing the sample, add more charcoal, shake quickly and filter again).

Color Development

- Pipette 5 ml of the soil extract into a 25 ml volumetric flask.
- To it add 5 ml of Dickman and Bray's chloromolybdic acid reagent must be added drop by drop with constant shaking till the effervescence due to CO_2 evaluation ceases.
- Wash the neck of the flask with double distilled water until the contents are diluted to about 22 ml. Add 0.25 ml (5 drops) of the 0.1 M stannous chloride.
- Make the volume upto the mark with double distilled water.
- Measure the intensity of the blue color spectrophotometrically at 660 nm after 5 minutes. Determine the concentration of P from the standard curve perform a blank.

Reactions

Exchange reaction

Exchange complex] Phosphate + HCO_3^- → Exchange complex] HCO3 + Phosphate

Chemical reaction

$$Ca_3(PO_4)_2 + 6NaHCO_3 \rightarrow 3Ca(HCO_3)_2 + 2Na_3PO_4$$

$$3Ca(HCO_3)_2 \rightarrow 3CaCO_3 + 3H_2CO_3$$

$$2Na_3PO_4 + 3H_2CO_3 \rightarrow 2H_3PO_4 + 3Na_2CO_3$$

$$Ca_3(PO_4)_2 + 6NaHCO_3 \rightarrow 3CaCO_3 + 2H_3PO_4 + 3Na_2CO_3$$

Color development

$$(NH_4)_6Mo_7O_{24} \cdot 4H_2O + 6HCl \rightarrow 7H_2MoO_4 + 6NH_4Cl$$

(Ammonium molybdate) (Molybdenic acid)

$$H_3PO_4 + 12H_2MoO_4 \rightarrow H_3P(Mo_3O_{10})_4 + 12H_2O$$

(Phosphate) (Phosphomolybdate yellow coloured complex)

$$\text{Phosphomolybdate + SnCl} \xrightarrow{\text{reduction}} \text{Reduced phosphomolybdate}$$

(molybdenum-blue complex of approximate composition)

Standard Curve for Phosphorus

- Primary phosphate standard; 50 ppm of phosphorus; Dry (AR) grade potassium dihydrogen phosphate (KH_2PO_4) in an air oven at 40° – 50°C for one hour and cool in a desiccator. Weigh accurately 0.2195 gm of KH_2PO4 and dissolve in about 400 ml double distilled water in a 1 litre volumetric flask. Then add 25 ml of 7(N) H_2SO_4 (approx) and make up the volume to 1000 ml with double distilled water. This gives 50 ppm 'P' solution (Addition of H_2SO_4 preserves the solution indefinitely but should be stored in soft glass bottle rather than one of pyrex to minimize contamination with arsenic)
- Prepare 2 ppm standard (secondary/working solution) from the prepared 50 ppm by proper dilution (20 ml of 50 ppm stock diluted exactly to 500 ml for preparing 2 ppm standard).
- From this 2 ml stock prepare different concentrations of P in 25 ml volumetric flask viz. 0.2, 0.4, 0.6, 0.8, 1.0 ppm by pipetting requisite volume of 2 ml stock.
- Add 5 ml of extracting reagent (Olsen's) and the color is developed by adding 5 ml of chloromolybdic acid reagent and stannous chloride (1 ml or less).
- Make up the volume with double distilled water and take the readings after 4–5 minutes at 660 nm wavelength after properly adjusting the blank (0.00 ppm) to 100% transmittance or 0.00% absorbance. All reagents including the extraction solution and sample processing chemicals must be included in each of the standard solutions and in the blank employed for preparing the calibration curve. The standard curve is plotted by taking the spectrophotometer readings along the Y axis (ordinate) and the concentration of P (ppm) along the x-axis (abscissa).

Precautions for P estimation

- For a satisfactory phosphorus procedure constant conditions must be maintained in the blank, standard and test solutions.
- Alkaline washing powders (which often contains) phosphates must not be used for glassware cleaning. All glass wares should be cleaned with chromic acid and thoroughly washed with double distilled water. For final clearing the glassware may be dipped in or rinsed with 6 N HCl after apparently clean, then thoroughly washed with double distilled water.
- Double distilled water must be used for all purposes in P estimation.
- The reagents and filter papers should be as free of phosphorus as possible.
- Pyrex glass apparatus, particularly new ones are to be avoided to minimize contamination with arsenic, which interferes in the analysis.
- Molybdenum blue solutions must not be kept in the volumetric flask after completion of experiment. The volumetric flasks and the spectrophotometer cuvettes must be washed immediately after use.

ESTIMATION OF AVAILABLE POTASSIUM IN SOIL BY NEUTRAL NORMAL AMMONIUM ACETATE METHOD

Reagents

- Neutral normal ammonium acetate solution; Dilute 60 ml glacial acetic acid (99.5%) and 75 ml concentrated ammonia solution (sp. gr. 0.91, 25% NH_3) to one litre. Mix well, cool and adjust the pH to 7.0 with dilute acetic acid or ammonia solution.
- Potassium chloride solution: 1000 ppm stock solution; Dissolve 1.907 g of AR grade potassium chloride (dried at 60°C for 1 hr.) in distilled water and make up the volume to 1 litre.

Procedure

- Weigh 5 g soil sample in a 25 ml conical flask.
- Add 25 ml of neutral normal ammonium acetate (pH = 7) and shake for 25 minutes.
- Filter immediately through a dry filter paper (Whatman No.1).
- Reject first few ml of the filtrate.
- Determine the potassium concentration in the extract flame-photometrically after necessary setting and calibration of the instrument.

Standard curve for potassium

- From the mother stock solution (1000 ppm K), prepare 2, 5, 10, 15 and 20 ppm K solutions in 50 ml volumetric flask by proper dilution.
- Construct the standard curve by plotting the flamephotometer readings along Y-axis and the different concentrations (ppm) along X-axis.

DETERMINATION OF AVAILABLE SULPHUR IN SOIL

Reagent

- CaCl2 (0.15%). Dissolve 1.5 g of $CaCl_2$ dihydrate in distilled water and make the vol. to 1 lit.
- Stabilizing agent or conditioning agent: Dissolve 75 g NaCl in 250 ml of distilled water in a 500 ml volumetric flask and add 30 ml of concentrated HCl followed by 100 ml ethanol and 50 ml glycerol with constant stirring. Make the volume to 500 ml.
- BaCl2.2 H2O
- **Standard sulphate solution:** Dissolve 0.5434 g of AR grade K_2SO_4 in distilled water and dilute to 1 L. This is 100 ppm S solution.

Standard preparation: Pipette out 0, 0.5, 1.0, 1.5, 2.0, 2.5 ml of 100 ppm sulphur solution into 50 ml volumetric flask and to this add 5 ml of conditioning agent and a pinch of $BaCl_2$. Make the volume to the mark to prepare the working standard of 0, 1, 2, 3, 4 and 5 ppm respectively. After 10 minutes, the turbidity developed in the standards is measured in a spectrophotometer at a wave length of 420 nm.

Procedure

- Take 10 g of soil into a 250 ml of conical flask.
- To this add 50 ml of $CaCl_2$ (0.15 %) solution and shake for 30 min.
- Filter through Whatman No. 1 filter paper.
- Pipette out 10 ml of extract into a 25 ml volumetric flask, add little amount of distilled water followed by 2.5 ml stabilizing agent and a pinch of $BaCl_2$.
- Shake the contents and make up the volume to the mark with distilled water.
- Measured the sulphur content in a spectrophotometer at a wave length of 420 nm.

DETERMINATION OF AVAILABLE MICRONUTRIENTS (FE, MN, ZN AND CU) IN SOIL

Reagents

- DTPA= 0.0005 M (formula weight 393.35)
- $CaCl_2.H_2O$= 0.01 M solution
- TEA = 0.1 M solution.

Extracting solution: Dissolve 13.1 ml reagent grade TEA, 1.967g DTPA (AR grade) and 1.47g of $CaCl_2$ in 100 of glass distilled water. Allow DTPA to dissolve and then dilute it to approximately 900 ml. Adjust the pH 7.3 ±0.05 with 1;1 HCl (while stirring) and dilute to 1litre (while stirring) and dilute to 1 litre.

Procedure

- Weight 10 g of air-dried soil in a 125 ml conical flask or polypropylene storage bottle.
- Then add 20 ml of the DTPA extracting solution.
- Cork the bottles or flasks and place them upright on a horizontal shaker. Shaker for 2 hours with a speed of 120 cycles per minute. Filter the suspension with Whatman 42 filter paper.
- Keep the filtrate in polypropylene bottles to be analyzed for Zn, Cu, Mn and Fe with atomic absorption

spectrophotometer.

- When the sample need dilution before measurement, they should be diluted with DTPA solution to maintain a constant matrix.

DETERMINATION OF BORON CONTENT IN SOIL SAMPLE

Reagents

- **Buffer solution:** Dissolve 250g of ammonium acetate and 15 g EDTA disodium salt in 400 ml distilled water and slowly add 125 ml of glacial acetic acid and mix.
- **Azomethine- H reagent:** Dissolve 0.45 g of azomethine –H in 100 ml of 1% L – ascorbic acid solution fresh reagent should be prepared each week and stored in a refrigerator.
- **Standard solution:** To prepare the standard stock solution, dissolve 0.570 g boric acid (H_3BO_3) AR grade in 1 litre of distilled water to obtain a stock solution in a 100 ml volumetric flask and dilute up to the mark. This solution contains 5 mg B/ml.

Working Standards and Standard Curve: To a series of 25 ml volumetric flasks add 0, 0.25, 0.5, 1.2 and 4 ml of 5 mg B /ml solution. To each volumetric flask add 2 ml of buffer solution and mix, add 2 ml of Azomethine – H reagent solution. Allow to stand for at room temperature for 30 minutes. Make the volume with distilled water and measure the absorbance at 420 nm on spectrophotometer. The concentration of B in working standard would be 0, 0.10, 0.20, 0.40, 0.80 mg B/ml. Over a concentration range of 0.5 to 10 mg B/ml, Azomethine-H solution from a stable complex with H_3BO_3 at pH 5.1. Maximum absorbance occurs at 420 nm with little or no interference from a wide variety of salts.

Procedure

- A 25 g soil sample and 50 ml of water and about 0.5 g of activated charcoal is boiled for 5 minutes in a quartz flask and filtered immediately through Whatman filter paper No. 42.
- 5 ml of the extract is taken in a 25 ml volumetric flask and 4 ml of buffer solution and 4 ml of azomethine –H reagent solution is added. The color is allowed to develop for 1 hour and the volume is made up to the mark.
- Intensity of color is measured by spectrophotometrically at 420 nm and the boron concentration read.

DETERMINATION OF CHLORIDE (Cl^-) CONTENT IN SOIL SAMPLE

Reagents

- Potassium chromate indicator, 5% aqueous solution of pure K_2CrO_4
- 0.02 (N) $AgNO_3$ Solution: Dissolve 3.4 g of $AgNO_3$(AR) in double distilled water and make up the volume to one litre. Standardize this solution against a standard NaCl solution and keep in amber colored bottle away from light.

Procedure

- Weigh 40 g of soil sample in a 500 ml of conical flask
- Add 200 ml double distilled water and shake for one hour in a shaking machine for equilibration.
- Filter the suspension
- Pipette out the 5 ml of extract or 5 ml of water sample in conical flask
- Add 5-6 drops K_2CrO_4 indicator and titrate the solution with 0.02 (N) $AgNO_3$ solution with stirring until the first reddish brown tinge appears. The ml of $AgNO_3$ required corresponds to the amount of chloride present.

DO'S AND DON'T'S IN THE LABORATORY DURING CHEMICAL ANALYSIS

Dos

- Periodical calibration of equipments, weighing balances and glass wares.
- Proper labeling of chemicals and reagents in the laboratory.
- Proper cleaning of glass wares, rinsing with distilled water and drying.
- Use locally available detergent liquids like Teepol for general glass washing and chromic acid (prepared by dissolving $K_2Cr_2O_7$ in concentrated H_2SO_4) for dirty and greasy glass wares.
- Use safety spectacles while digesting samples with acid.
- Keep first aid box ready and at hand. Display list of safety precautions and use of antidotes in a prominent place in laboratory.
- Use great care in evaporating perchloric acid as it is very explosive. Use fume cupboard for evaporation.
- Handle dangerous chemicals like NaCN or KCN and inflamatory substances like organic solvents very carefully.
- Use suction pumps to suck dangerous chemicals.
- Use non-silicone containing lubricants for stop cocks in burettes and delivery glass wares.
- Store solutions which are unaffected by exposure to air in glass bottles.
- Store oxidizing chemicals like iodine and silver nitrate only in amber color glass bottles.
- Store EDTA solution in hard polythene containers.
- Use distilled water or mineralized water for routine analysis and only double distilled water for micronutrient analysis.
- Carry our standardization of all reagents daily.
- Always carry out duplicate analysis of each sample.
- Always carry out blank determination along with the sample.

- Use TEFLON (Polytetrafluoro ethylene) beakers for fluoride estimation instead of borosilicate glass and plastic beaker for boron estimation.
- Always test for complete precipitation in gravimetric analysis.
- Use proper grade of Gooch crucible and filter paper to avoid leaching on colloidal precipitates. Use G3 Grade of Gooch crucible for precipitates of moderate particle size and G4 for finer precipitates such as of $BaSO_4$.
- Ensure proper wave length for spectrophotometric and colorimetric analysis.
- Periodically check absorbance scale with standard solutions.
- Ensure proper rinsing of pipettes before sucking next sample solution.
- Always keep similar conditions and use same reagents for both standard curve and sample analysis in spectrophotometric and atomic absorption (AAS) analysis.
- Keep concentration within specific sensitive range of particular element through suitable dilution in spectrophotometer and AAS.
- Ensure optimum temperature, air-gas mixture and pre-heating time as per instruction manual in case of flame photometer and AAS.
- Take adequate precaution to check ingress of moisture during sample preparation and weighing.
- Immediately connect the distillation tube with receiver flask after adding NaOH in distillation flask to avoid loss of released NH_3.

Donts

- Do not heat glass wares directly over the flame without wire gauge.
- Do not heat inflammable chemicals directly.
- Do not suck dangerous chemicals through mouth. Use suction pump.
- Do not keep AR grade chemicals open for longer time.
- Do not insert pipette into reference or analytical grade reagent bottle as it can introduce errors.
- Do not return the liquid reagents after they have been taken out from the bottle as it can be a source of contamination and introduce errors.
- Do not hold stopper between fingers while pouring liquid from bottle nor put it on shelf or working bench. Put it on a clean watch glass.
- Do not transfer readily-soluble substances from weighing bottle into volumetric flask directly, but transfer it to a beaker, dissolve and then quantitatively transfer into volumetric flask.
- Do not use glass or silica crucible for fusion of alkali metals or evaporation with HF but use platinum crucible (melting point 1773°C).
- Do not pour large volume of washing solution at a time to wash precipitate but use a number of small portions of washing solution and allow complete draining before addition of fresh portion,
- Do not weigh hot or cool precipitate directly as it may absorb moisture but cool it in a desiccator for at least half an hour before weighing.
- Do not touch the nebulizer or sucking tube in flame-photometer/AAS analysis with naked finger as it can introduce errors.