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SOIL TESTING MANUAL

Do-It-Yourself Manual

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1. INTRODUCTION

Vigyan Ashram has developed a “**Soil manual**” for laboratories and colleges/institutes. Users of the manual will be able to set up their laboratory using this manual.

All the standard operating procedures are mentioned in this manual .We have provided safety instructions while performing any experiment, with this manual. Users are suggested to read the manual carefully along and assess the site conditions before performing the experiment.

2. PURPOSE OF MANUAL

- To share Standard Operating Procedures to laboratories and institutes.
- To reduce cost of each procedure.
- Manual to be made available online as open source.

3. SAFETY INSTRUCTIONS

During performing experiment we should use safety equipment such as hand gloves, shoes and glasses.

(And as appropriate)

SOP FOR ORGANIC CARBON:-

Introduction: -

Estimates of organic carbon are used to assess the amount of organic matter in soil. The method measures the amount of organic carbon in plant and animal remains including soil humus. Higher soil organic carbon promotes soil structure or tilth meaning there is greater physical stability. This improves soil aeration (oxygen in the soil) and water drainage and retention, and reduces the risk of erosion and nutrient leaching. The method used for organic carbon estimation from soil sample is Walkey – Black method.

Equipment:-

1. 500-mL Erlenmeyer flasks.
2. 10-mL pipette.
3. 10-and 20-mL dispensers.
4. 50-mL burette.
5. Analytical balance.
6. Magnetic stirrer.

Reagents:-

1. **H_3PO_4, 85%.**

2. **H_2SO_4, concentrated (96%)**

3. **NaCl**, solid.

4. **Standard 0.167M $K_2Cr_2O_7$**:- Dissolve 49.04 g of dried (105°C) $K_2Cr_2O_7$ in water and dilute to 1L.

5. **0.5M Fe^{2+} solution**: Dissolve 196.1g of Fe $(NH_4)_2(SO_4)$ •$6H_2O$ in 800 mL of water containing 20 mL of concentrated H_2SO_4 and dilute to 1 L. The Fe^{2+} in this solution oxidizes slowly on exposure to air so it must be standardized against the dichromate.

6. **Ferroine indicator:** Slowly dissolve 3.71 g of ophenanthroline and 1.74 g of $FeSO_4$•$7H_2O$ in 250 mL of water.

Procedure:

1. Weigh out 0.10 to 2.00g dried soil (ground to <60 mesh) and transfer to a 500-mL Erlenmeyer flask. The sample should contain 10 to 25mg of organic C (17 to 43mg organic matter). For a 1g soil sample, this would be 1.2 to 4.3% organic matter. Use up to 2.0g of sample for light colored soils and 0.1g for organic soils.

2. Add 10 ml $K_2Cr_2O_7$ solution by means of pipette.

3. Add 20 mL. H_2SO_4 of concentrated by means of a dispenser and swirl gently to mix of the flask out of the solution. Avoid excessive swirling that would result in organic particles adhering to the sides of the flask out of the solution.

4. Allow to stand for 30 min. The flasks should be placed on an insulation pad during this time to avoid rapid heat loss.

5. Dilute the suspension with about 200 mL of water to provide a clearer suspension for viewing the endpoint.

6. Add 10 mL of 85% H_3PO_4, using a suitable dispenser, and 0.2g of NaCl. The H_3PO_4 and NaCl is added to complex Fe^{3+} which would interfere with the titration endpoint.

7. Add 10 drops of Ferro in indicator. The indicator should be added just prior to titration to avoid deactivation by adsorption onto clay surfaces.

8. Titrate with 0.5 M Fe^{2+} to a burgundy endpoint. The color of the solution at the beginning is yellow-orange to dark green, depending on the amount of unreacted $Cr_2O_7^{2-}$ remaining, which shifts to a turbid grey before the endpoint and then changes sharply to a wine red at the

endpoint . Use of a magnetic stirrer with an incandescent light makes the endpoint easier to see in the turbid system (fluorescent lighting gives a different endpoint color). Alternatively use a Pt. electrode to determine the endpoint after step 5 above. This will eliminate uncertainty in determining the endpoint by color change.

9. Run a reagent blank using the above procedure without soil. The blank is used to standardize the Fe^{2+} solution daily.

Calculations:-

$$O.C. = \frac{(B - S) \times M\ of\ Fe^{2+} \times 12 \times 100 g\ soil}{4000}$$

Where,

B = mL of Fe^{2+} solution used to titrate blank

S = mL of Fe^{2+} solution used to titrate sample

12/4000 = Mili equivalent weight of Carbon.

SOP FOR NITROGEN ESTIMATION

Introduction: -

Nitrogen is a macronutrient and is very important for plant growth. But too much nitrogen is just as dangerous. Nitrogen can also cause a lot of environmental damage in groundwater and in oceans. Hence it is necessary to estimate nitrogen content in soil. The process used for estimation of nitrogen from sample is Kjeldahls method.

Reagents:-

1. **Sulphuric acid**, 95%, reagent grade
2. **Catalyst** (copper sulphate and potassium sulphate):- Add 5g of potassium sulphate and 2g copper sulphate
3. **Sodium hydroxide 50%** :- Dissolve 50g sodium hydroxide in 50 ml DW
4. **Sulphuric acid solution 0.5 M**: - Dissolve 2.4 ml of 98% Sulphuric acid in 10 ml distilled water and make volume 100 ml.
5. **Hydrochloric acid 0.25 N :-**Dissolve 2.17 ml of HCl in 100 ml DW

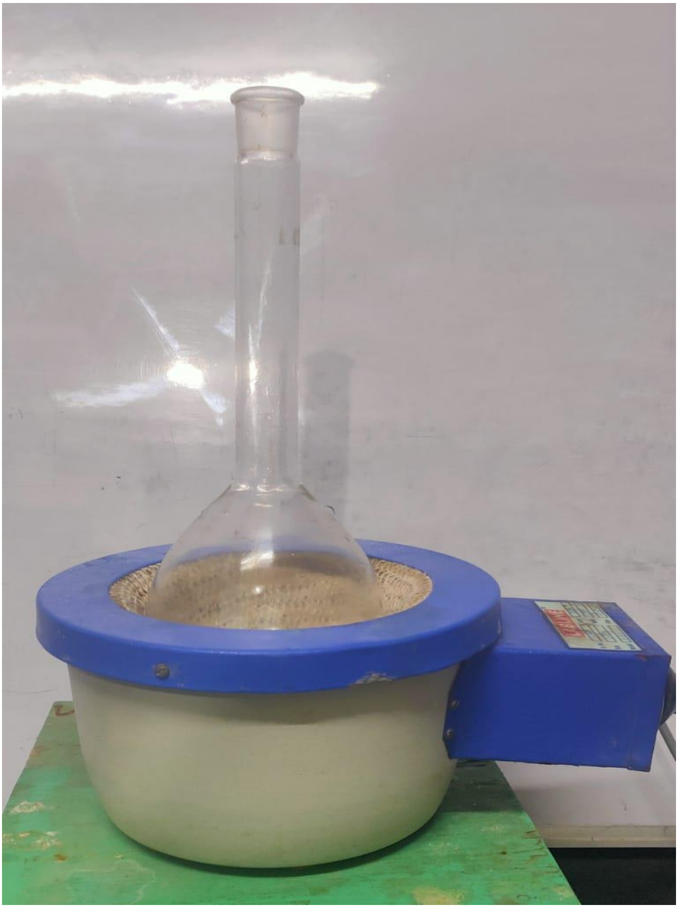
6. **Sodium carbonate solution :-** For standardization of HCl solution

PART A: - DIGESTION

1. Weigh around 1 grams of dry glycine in the digestion flask and add 2g of catalyst and add 20 ml sulphuric acid (95%).Add glass beads in the flask to avoid excess heating.
2. Digest the content in flask for 30-40 mines until the temp raises up to 160 degree Celsius.
3. Color changes from blue to colorless.

PART B: - DISTILLATION

1. Add 100 ml distilled water in the digestion flask and let it cool.
2. Add 50 ml 50% sodium hydroxide solution to the flask and check pH
3. Attach the flask to the distillation unit and collect the distillate in 0.5 M Sulphuric acid solution.



KJELDAHL APPARATUS

Note: - Back Titration:-

1. Prepare 0.5 molar Noah by adding 2gm Noah in 98 ml D/W.
2. Then titrate it with trapping solution in which ammonia is trapped to neutralize excess acid present in trapping solution
3. If you used boric acid as a trapping solution then there is no need to back titrate it.

PART C: - TITRATION

1. Add 6-7 drops of tashiro indicator to the distillate containing flask and titrate it with 0.25M HCl solution
2. End point is bluish to a slight violet color.

CALCULATIONS:-

$$\%N = \frac{(ml\ standard\ acid - ml\ of\ blank) \times N\ of\ acid \times 1.4007}{Weight\ of\ sample\ in\ grams}$$

Where,

N = Normality of acid used

Blank = NaOH required to back titrate excess H_2SO_4.

NOTE: - FOR STANDARDISATION OF HCl:-

1. Weigh approximately previously heated 1.4g of sodium carbonate solution and make it volume to 100 ml.
2. Add 2-3 drops of methyl red indicator to it.
3. Titrate it with 0.25 M HCl solution
4. End point is faint pink

SOP FOR CALCIUM AND MAGNESIUM

Introduction–

Calcium and Magnesium are micronutrients which are required in trace amounts to plants. Calcium is necessary for plants for synthesis of cell wall, and magnesium is necessary cofactor for metabolism of plants. Hence it is necessary to estimate Calcium and Magnesium content in soil.

Reagents:-

A. **Buffer Solution (NH_4Cl-NH_4OH)**:- Dissolve 67.5g ammonium chloride in 570 mL concentrated ammonium hydroxide, and transfer the solution to a 1-L volumetric flask, let it cool, and bring to volume with DI water.

B. **Eriochrome Black Indicator**:- Dissolve 0.5g eriochrome black with 4.5g hydroxylamine hydrochloride in 100 mL ethyl alcohol (95%). Prepare a fresh batch every month.

C. **Ethylenediaminetetraacetic Acid Solution: -** (EDTA), 0.01 N Dissolve 2g ethylene diaminetetraacetic acid, and 0.05 g magnesium chloride ($MgCl_2$) in DI water, and bring to 1-L volume with DI water.

D. **Sodium Hydroxide Solution (NaOH)**:- 2 N Dissolve 80g sodium hydroxide in about 800 mL DI water, transfer the solution to a 1-L volume, cool, and bring to volume with DI water.

E. **Ammonium Purpurate Indicator** ($C_8H_8N_6O_6$):- Mix 0.5g ammonium purpurate (Murexid) with 100g potassium sulphate (K_2SO_4).

F. **Standard Stock Calcium Chloride Solution ($CaCl_2.2H_2O$)** :- (0.01N) Dissolve 0.5g pure calcium carbonate ($CaCO_3$ dried for 3 hrs. at 100°C), 31 UAE University in 10 mL 3N hydrochloric acid and bring to 1-L volume with DI water. This can also be prepared by dissolving 0.735g calcium chloride dehydrate ($CaCl_2.2H_2O$) in 1-L volume with DI water.

G. **Ammonium acetate solution**: - Dissolve 77g of ammonium acetate in 80 ml of distilled water and bring volume up to 100 ml.

Procedure:-

Preparation of soil extract:-

1. Weigh 50gms of soil and add 200ml of 40% ethanol to it.
2. Filter the mixture and discard the filtrate and add ammonium acetate solution to it
3. Incubate the mixture overnight and use it as soil extract.

A. Calcium

1. Pipette 10-20 mL soil saturation extract, having not more than 1.0 meq Calcium, into 250-mL Erlenmeyer flask.

2. Dilute to 20-30 mL with DI water, add 2-3 mL 2N sodium hydroxide solution, and about 50 mg ammonium purpurate indicator.

3. Titrate with 0.01N EDTA. The color changes from red to lavender or purple. Near the end point, EDTA should be added one drop every 10 seconds since the color change is not instantaneous.

4. Always run a blank containing all reagents but no soil, and treat it in exactly the same way as the samples; and subtract the blank titration reading from the readings for all samples

B. Calcium plus Magnesium:-

1. Pipette 10-20 mL soil saturation extract into a 250mL flask, dilute to 20-30 mL with DI water. Then add 3-5 mL buffer solution. And a few drops of eriochrome black indicator.

2. Titrate with 0.01N EDTA until the color changes from red to blue.

Calculations:-

$$\frac{Ca\ or\ Ca + Mg\ meq/L = (V - B) \times N \times R \times 1000}{Weight}$$

$$Mg\ (meq/L) = (Ca + Mg\ (meq/L) - Ca\ (meq/L)$$

Where:-

V = Volume of EDTA titrated for the sample (mL)

B = Blank titration volume (mL)

R = Ratio between total volume of the extract and extract volume used for titration.

N = Normality of EDTA solution

SOP FOR POTASSIUM

Introduction:

Potassium is an essential plant nutrient and is required in large amounts for proper growth and reproduction of plant**s**. . It affects the plant shape, size, color, taste and other measurements attributed to healthy produce. Plants absorb potassium in its ionic form, K^+. The production of ATP can regulate the rate of photosynthesis. Potassium also helps regulate the opening and closing of the stomata, which regulates the exchange of water vapor, oxygen and carbon dioxide. . Other roles of K include: Increases root growth and improves drought resistance. Potassium is associated with the movement of water, nutrients and carbohydrates in plant tissue. It's involved with enzyme activation within the plant, which affects protein, starch and adenosine triphosphate (ATP) production. The production of ATP can regulate the rate of photosynthesis.

Reagents:-

1. **Ammonium acetate solution: -** Dissolve 77g in 900 ml in distilled water and to adjust pH 7.0 add 3N sodium hydroxide solution. And adjust volume up to 1 litter.

2. **Standard potassium chloride solution (1000 ppm)**:- Dissolve 1.908g potassium chloride solution in 1 litter distilled water. We get 1000 ppm solution. , then pipette out 10 ml, 5 ml, 1 ml respectively and prepare 10, 50, 100 ppm solution. This solutions are used for calibration of flame photometer.

Procedure:-

1. First, add 5 grams of soil in conical flask which is to be tested.
2. Add 25 ml ammonium acetate solution to it, and by using rotary shaker, shake it for 10 minutes.
3. Then filter the solution by using whatmann filter paper, take 5 ml filtrate and add 20 ml distilled water to it.
4. Take reading by using flame photometer, take dilution factor 5.

Calculations:-

$$K\ (mg)\% = \frac{x}{1000} \times \frac{100}{5} \times \frac{25}{1}$$

$$= x\ \times 0.5$$

$$K_2O\ (Mgm)\% = x \times 0.5 \times 1.2$$

$$= x \times 0.5$$

$$K_2O(Mgm)\% = x \times 0.5 \times 1.2$$

Whereas,

X = Reading

1000 = Microorganisms in milligrams

25 = Ammonium acetate solution

5 = Weight of soil

1.20 = Conversion factor for K to K_2O

DISCLAIMER

The content in this DIY manual is the developed by Vigyan Ashram. All instructions are merely for educational purpose and to create a sharable open source D-I-Y document. While the information in this document has been verified to the best of our abilities, we cannot guarantee the performance.

All the observation and data are taken from various experiments on system at Vigyan Ashram. We reserve the right to change the experiment. Please contact our website or our expert team for any clarification.