

Soil Science Department



**International Agriculture Program
Fundamentals of Organic Farming (316 ASS)
Practical**

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Lesson 1

Soil pH and Electrical Conductivity (EC)

Soil pH

The pH value is a measure of the hydrogen ion activity of the soil water system and expresses the acidity and alkalinity of soil. The pH is a very important property of soil as it determines the availability of nutrients, microbial activity and physical condition of the soil. The pH of a solution has been defined as the negative logarithm of hydrogen ion activity which in very dilute solutions.

$$\text{pH} = -\log \text{H}^+$$

Pure water, which is exactly neutral at 25°C, has a pH 7.0. As pH decreases from 7, the hydrogen ion concentration exceeds OH⁻ ion and the range is acidic. When the OH⁻ ion concentration is more than H⁺ ion pH value lies between 7 and 14 and the range is alkaline.

Determination of pH

The methods for the determination of pH of the soil solution are mainly classified into two groups:

- a. Electric pH meter method.
- b. Indicator method.

The electric pH meter method which is commonly used is being described here. Most digital pH meter now a days have single (combined) electrode assembly. The instrument being a potentiometer requires to be calibrated before use with buffer solutions of known pH values.

Principle

A glass surface of the electrode in contact with hydrogen ions of the solution under test acquires an electrical potential which depends on the concentration of H⁺ ions. A measure of the electrical potential is; therefore, give H⁺ ion concentration or pH of the solution. The pH value is measure in soil water suspension such as saturated soil paste and (1:2.5 or 1:5) soil water suspensions.

Procedure

1. Weigh 20 g air-dry soil (< 2mm) into a 100 ml glass beaker.
2. Add 50 ml Deionized water using a graduated cylinder or 50ml volumetric flask.
3. Mix well with a glass rod, and allow standing for 30 minutes.

4. Stir suspension every 10 minutes during this period.
5. After 1 hour, stir the suspension.
6. Put the Combined Electrode in suspension (about 3cm deep). Take the reading after 30 seconds.
7. Remove the Combined Electrode from the suspension, and rinse thoroughly with Deionized water (DI) in a separate beaker, and carefully dry excess water with a tissue.

Electrical conductivity (EC)

In case, excess salts are present in soil, the plant growth in such soil is affected adversely because excessive salts in soil solution cause high osmotic pressure which prevents absorption of moisture and nutrients by plant in adequate amount. These soils are designated as salt affected soils. On the basis of nature and quantity of salts present. Soils may be classified as saline, alkali and saline-alkali.

$$\begin{aligned}\text{Salts in meq/L} &= \text{EC (dSm-1)} \times 10 \\ \% \text{ salts in solution} &= \text{EC (dSm-1)} \times 0.064 \\ \text{Osmotic pressure} &= \text{EC (dSm-1)} \times 0.36 \\ \text{ppm} &= \text{EC (dSm-1)} \times 640\end{aligned}$$

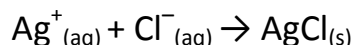
Procedure

1. Prepare 1:2.5 (soil:water) suspension, as for pH determination.
2. Filter the suspension using filter paper.
3. Transfer the clear filtrate into a 50 ml bottle, immerse the Conductivity Cell in the solution, and take the reading.
4. Remove the conductivity cell from the filtration, rinse thoroughly with Deionized water (DI), and carefully dry excess water with a tissue.

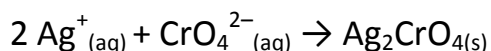
Lesson 2

Determination of Chloride (Mohr's Method)

This method determines the chloride ion concentration of a solution by titration with silver nitrate. As the silver nitrate solution is slowly added, a precipitate of silver chloride forms.



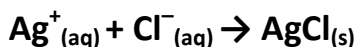
The end point of the titration occurs when all the chloride ions are precipitated. Then additional silver ions react with the chromate ions of the indicator, potassium chromate, to form a red-brown precipitate of silver chromate.



This method can be used to determine the chloride ion concentration of water samples from many sources such as seawater, stream water, river water and estuary water. Seawater is used as the example here. The pH of the sample solutions should be between 6.5 and 10. If the solutions are acidic, the gravimetric method or Volhard's method should be used.

Method

1. Pipette a 10 mL aliquot of extraction solution into a conical flask and add about 50ml distilled water and 1ml of chromate indicator.
2. Titrate the extraction solution with 0.1 mol l⁻¹ silver nitrate solution. Although the silver chloride that forms is a white precipitate, the chromate indicator initially gives the cloudy solution a faint lemon-yellow colour. The endpoint of the titration is identified as the first appearance of a red-brown colour of silver chromate.
3. Determine the average volume of silver nitrate used from your concordant titers.
4. Use the following reaction equation to determine the moles of chloride ions reacting.



Calculation:

$$\begin{aligned}\text{Cl}^- \text{ (mmoles /l)} &= M_{\text{AgNO}_3} \times V_{\text{AgNO}_3} \times 1000/10 \\ &= 0.1 \times V_{\text{AgNO}_3} \times 100\end{aligned}$$

Where:

V_{AgNO_3} (Volume of silver nitrate titrated sample).

M_{AgNO_3} (Molarity of silver nitrate = 0.1M).

Lesson 3

Determination of Calcium and Magnesium

The determination of water soluble salts in soil is of special importance in arid and semi-arid countries. In irrigated area it is very important to know the salt content of the soils, because these soils are in danger of becoming saline or alkali. The water soluble salts in soils are generally determined in two types of soil water extracts:

a. Saturation extract. b. 1:2.5 soil water extract.

If EC of the soil saturation extract is greater than 2 dS/m or greater than 1.0 dS/m for the 1:2.5 soil water extract, the water extract should be retained for determination of soluble ions. The saturation extract determination considered to be more reliable because it is directly related to the field-moisture range. However, determination in saturation extract is time consuming 1:2.5 soil water extract can be used for rapidity.

Principle

The method is based on the fact that Calcium and Magnesium ions form stable complexes with versene.

Method for determination of Ca⁺⁺

1. Take a suitable aliquot (5 or 10 ml) of the given solution of the soil extract (saturation or 1:2.5 soil water extract) and add 5 ml of 16% NaOH solution.
2. Add 40-50 mg of the murexid indicator powder.
3. Titrate it with 0.01N EDTA solution till the color gradually changes from orange red to reddish violet (purple).

Calculations

$$\text{Ca}^{2+} \text{ (meq/l)} = \frac{\text{Normality of EDTA} \times \text{Vol. of EDTA} \times 1000}{\text{ml. of aliquot taken extract}}$$

Method for determination of Mg⁺⁺

1. Pipette out 25 ml of the given solution.
2. Add 5 ml of ammonium chloride-ammonium hydroxide buffer, and add 3-4 drops of Eriochrome black T indicator.
3. Titrate this solution with 0.01N EDTA till the color changes to bright blue.

Calculations

$$[\text{Ca} + \text{Mg}] \text{ (meq/l)} = \frac{\text{Normality of EDTA} \times \text{vol. of EDTA} \times 1000}{\text{ml. of aliquot taken}}$$

$$\text{Mg}^{2+} \text{ (meq/l)} = \text{Ca} + \text{Mg} \text{ (meq/l)} - \text{Ca} \text{ (meq/l)}$$

Lesson 4

Total Calcium Carbonate "acid neutralization method"

Carbonate in the sample is dissolved in the excess of hydrochloric acid (HCl). The remainder of the acid is titrated against sodium hydroxide (NaOH). However, a given weight of soil is reacted with an excess of acid. In this reaction, CO_2 gas is released and the acid not used in the dissolution of carbonates is back-titrated with NaOH solution. In the titration method, two equivalents of acid are assumed to react with one mole of CaCO_3 . Hence, one equivalent of acid is assumed to be equivalent to one-half mole of CaCO_3 .

Active CaCO_3 is usually related to total CaCO_3 equivalent, being about 50% or so of the total value. Proponents of its use claim that this fraction is more closely related to nutrient behavior, such as involved with iron chlorosis.

Principle

The "rapid titration method" by Piper, also called "acid neutralization method", is used. The sample is treated with dilute acid and the residual acid (not consumed by carbonate) is titrated. The results are referred to as "calcium carbonate equivalent" since the dissolution is not selective for calcite, but also other carbonates such as dolomite will be dissolved to some extent.

Procedure

1. Weigh 1 g air-dry soil into a 250-ml Erlenmeyer flask.
2. Add 10 mL 1 N hydrochloric acid solution to the flask with a volumetric pipette.
3. Swirl and leave the flask overnight, or heat to 50-60°C, and let the flask cool.
4. Add 50-100 mL Deionized water (DI) water using a graduated cylinder, and add 2-3 drops phenolphthalein indicator.
5. Titrate with 1 N sodium hydroxide solution while swirling the flask.
6. Continue the titration until a faint pink color develops, and take the reading, R.

Calculation

Percentage Calcium Carbonate in soil

$$\% \text{CaCO}_3 = [(10 \times N_{\text{HCl}}) - (R \times N_{\text{NaOH}})] \times 0.05 \times 100 / W_t$$

Where:

N_{HCl} = Normality of HCl solution.

N_{NaOH} = Normality of NaOH solution.

R = Volume of NaOH solution used (ml).

W_t = Weight of air-dry soil (g).

0.05 = (Eq.W. for $\text{CaCO}_3 \div 1000$) = (50 \div 1000) = 0.05

Lesson 5

Carbon dioxide production (Soil Respiration)

Definitions

Soil Respiration: is used to mean the uptake of O₂ and /or the release of CO₂ by living, metabolizing entities in the soil.

Soil Aeration: is used to mean the exchange of O₂ and CO₂ between the atmosphere and the soil.

Principals

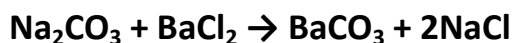
The measurement of the quantity and rate of carbon dioxide evolution from soils has been one of the best laboratory methods for measuring activity of soil micro flora as a whole. The method has its widest application in the study of organic matter decomposition in soils.

Method

1. Weigh 20g air-dries soil into a milk bottle and mix 2g ground Alfalfa with the dry soil.
2. Add about 10ml of distilled water.
3. Add 10ml from NaOH (1N) into a test tube then insert this tube vertically into the milk bottle.
4. Cover the beaker and incubate at room temperature for one week in this stage (Incubation interval) we observed this reaction:



5. In the end week, transfer the content of test tube into flask, then add 20ml from BaCl₂ (2N) solution.



6. Take about 10ml from residual (NaOH) solution into a volumetric flask.
7. Add 10 drops phenolphthalein indicator.
8. Titrate with (0.1N) HCl solution for residual (NaOH) until the color changes from (Pink) to (White).

Calculation

$$\text{CO}_2 \text{ (meq /100g soil)} = (\text{N}_{\text{NaOH}} \times \text{V}_{\text{NaOH}} - \text{N}_{\text{HCl}} \times \text{V}_{\text{HCl}}) \times 100/20$$

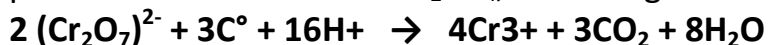
Lesson 6

Soil Organic Matter "Walkley-Black method"

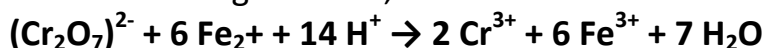
The organic matter in soil consists of a mixture of plant and animal residues in various stages of decomposition, of substances synthesized chemically and biologically from the breakdown products, and of microorganisms and small animals and their decomposing remains. Soil organic matter is usually partitioned into non-humic and humic substances. The major portion of the organic matter in most soil consists of humic substances. These are amorphous, Dark-colored, hydrophilic, acidic, partly aromatic, chemically complex organic substances that range in molecular weight from a few hundred to several thousands.

Principle

Determination of the content organic matter is a routine procedure carried out in soil analysis and testing labs because of the importance of organic matter to soil and plant. Many quantitative methods have been proposed for the determination of organic matter. However, the most common method called "Walkley-Black method" in which the organic matter in soil is oxidized by the treatment with dichromate ($K_2Cr_2O_7$) in the presence of concentrated H_2SO_4 , according to the following reaction.



After the reaction, the excess $(Cr_2O_7)^{2-}$ is titrated with $Fe(NH_4)_2(SO_4)2 \cdot 6H_2O$, according to the following reaction;



The amount of $(Cr_2O_7)^{2-}$ used in reducing organic carbon present in the soil sample could be obtained by subtracting the excess $(Cr_2O_7)^{2-}$ from that added at the beginning.

Method

1. Weight out a portion of 5g of tested soil and transfer it to a dry 500ml conical flask.
2. Using a pipette, add 10 ml of 1N $K_2Cr_2O_7$ to the soil sample, and swirl slowly to mix soil sample with dichromate.
3. Add 20 ml of concentrated H_2SO_4 and swirl gently again.
4. Leave the flask to stand for 30 minutes and then add 200 ml of distilled water, and then add 10 ml of O-phosphoric acid and 1 ml of di- phenyl amine.

5. Titrate the contents of the conical flask with 0.5 N ferrous ammonium sulfate till the color changes from dark brown to blue-violet, then to green (C).
6. Simultaneously, a blank is run without soil (B).

Calculations

$$\text{O.M\%} = [10 (B - C)/B] \times 0.003 \times (100/W_s) \times 1.72$$

Or:

$$\text{O.M} = N (B - C) \times 0.003 \times (100/W_s) \times 1.72$$

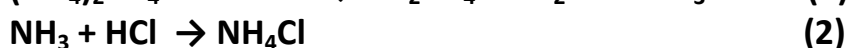
Lesson 7

Determination of Nitrogen

The nitrogen in ammonical nitrogenous fertilizer is determined by the Distillation with an alkali (such as NaOH) methods.

Principle

Nitrogen in any ammonical salt can be determined by distillation with sodium hydroxide. The solution of a known amount of salt is distilled with a known volume and normality of sodium hydroxide solution (the volume of NaOH is enough and more to expelled NH_3) by titration of the residual NaOH with a known acid we can calculate the nitrogen per cent in fertilizer.



Method

- 1- Take 20 mL of fertilizer solution (by pipette) to conical flask.
- 2- Add 40 mL of sodium hydroxide solution (0.5 N) to the conical flask which contain fertilizer solution.
- 3- Light the gas burner and start heating till 15 minute.
- 4- Test for ammonia gas using phenolphthalein paper.
- 5- Cool the conical flask and add 4 drops of indicator.
- 6- Titrate the solution (residual NaOH) using HCl acid (0.3N) and record the volume of acid.

Calculation

$$\text{Nitrogen percent\%} = [(V_k \times N_k) - (V_a \times N_a)] \times \frac{V_t}{V_s} \times \frac{14}{1000} \times \frac{100}{\text{wt.}}$$

Where:

V_t (Total volume of fertilizer solution = 1000ml).

V_s (Volume of fertilizer titrated sample = 20ml).

V_k (Total volume of NaOH).

N_k (Normality of NaOH).

V_a (Recorded volume of acid).

N_a (Normality of acid).

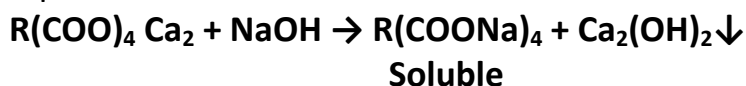
Lesson 8

Extraction of soil organic matter (Humus)

Soil organic matter (SOM) is considered an important part of soil for its high contribution to soil productivity. Generally, SOM contains two main fractions: humic substances and labile soil organic matter.

Humic substances have been found to be a stable material, specific to each soil and not markedly changing over decades of soil use (Siewert 1989, Stevenson 1994).

The interaction between organic matter and NaOH we can summarize by the following equation:



Characteristics of Humus:

1. Humus is the active fraction of the soil organic matter.
2. Humus is highly colloidal like clay, but is amorphous and non-crystalline.
3. The surface area and adsorptive capacity of humus are much greater than those of clay.
4. Humus has a cation exchange capacity between 150 to 300 meq, while from clay between 8 to 100 meq.
5. Humus absorbs 80 or 90% water from a saturated atmosphere.
6. Humus is black in color.

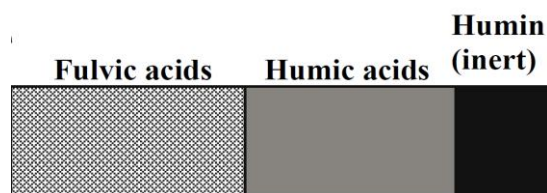


Fig. 1. Fractionation of soil organic matter by classical methods.

-
- Increase of colour intensity.
 - Increase of polymerization and humification.
 - Increase of C content.
 - Decrease of acidity.
 - Decrease of O content.

Humus Extract Methods

1. Place 10 g air-dry of compost sample on a filter paper in a funnel then put them on a volumetric flask.
2. Add 100 ml of extraction 0.1N NaOH to the compost sample.
3. Take 10 ml from this supernatant solution into a test tube, and then add 10 ml of 0.1N HCl solution.
4. Record the deferent colors between the products.

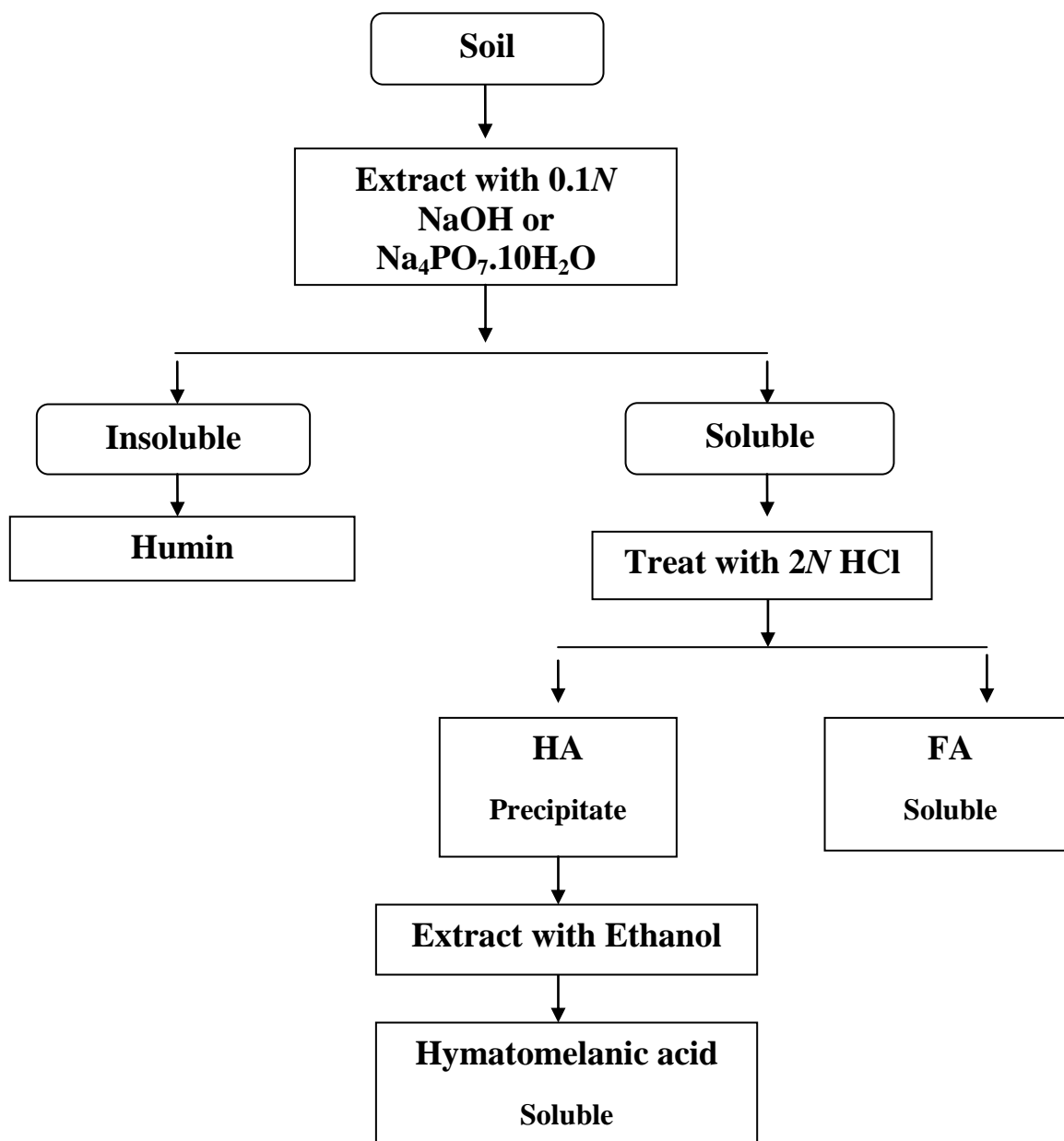


Fig. 2. Extraction and Fractionation of humic substances.



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Laboratory Experiments

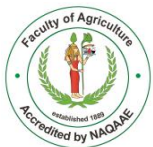


Name:

Reg. No. :

Determination of Soil pH and EC

Samples	pH Extract (1 : 2.5)	EC (dS/m)
1		
2		



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Laboratory Experimentals



Name:

Reg. No. :

Determination of Chloride Ion Concentration by Titration (Mohr's Method)

$$\begin{aligned}\text{Cl}^- (\text{mmoles /l}) &= M_{\text{AgNO}_3} \times V_{\text{AgNO}_3} \times 1000/10 \\ &= 0.1 \times V_{\text{AgNO}_3} \times 100\end{aligned}$$

Where:

V_{AgNO_3} (Volume of silver nitrate titrated sample).

M_{AgNO_3} (Molarity of silver nitrate = 0.1M).



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Laboratory Experiments



Name:

Reg. No. :

Determination of Calcium and Magnesium

Sample No.	Aliquot taken (ml)	Volume of EDTA used (ml)
1		
2		

Calculations:

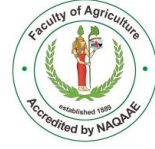
$$\text{Ca}^{2+} \text{ (meq/l)} = \frac{\text{Normality of EDTA} \times \text{Vol. of EDTA} \times 1000}{\text{ml. of aliquot taken extract}}$$

$$[\text{Ca} + \text{Mg}] \text{ (meq/l)} = \frac{\text{Normality of EDTA} \times \text{vol. of EDTA} \times 1000}{\text{ml. of aliquot taken}}$$

$$\text{Mg}^{2+} \text{ (meq/l)} = [\text{Ca} + \text{Mg (meq/l)}] - \text{Ca (meq /l)}$$



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Laboratory Experimentals



Name:

Reg. No. :

**Determination of Total Calcium Carbonate
by "acid neutralization method"**

CALCULATION:

Percentage Calcium Carbonate in soil:

$$\% \text{CaCO}_3 = [(10 \times N_{\text{HCl}}) - (R \times N_{\text{NaOH}})] \times 0.05 \times 100 / W_t$$

Where:

N_{HCl} = Normality of HCl solution.

N_{NaOH} = Normality of NaOH solution.

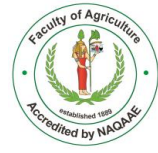
R = Volume of NaOH solution used (ml).

W_t = Weight of air-dry soil (g).

0.05 = (Eq.W. for $\text{CaCO}_3 \div 1000$) = (50 \div 1000) = 0.05



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Name:

Reg. No. :

**Determination of Carbon dioxide production
(Soil Respiration)**

Samples	Reading of Burette (V_{HCl}) ml
1	
2	

Calculation:

$$CO_2 \text{ (meq /100g soil)} = (N_{NaOH} \times V_{NaOH} - N_{HCl} \times V_{HCl}) \times 100/20$$

Where:

$N_{NaOH} = 1N.$

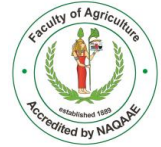
$V_{NaOH} = 10ml.$

$N_{HCl} = 0.1N.$

$V_{HCl} = \text{Reading of Burette.}$



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Name:

Reg. No. :

**Determination of Soil Organic Matter
"Walkley-Black method"**

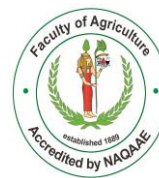
Sample No.	Volume of $\text{Fe}(\text{NH}_4)_2\text{SO}_4$ (ml)
1	
2	

Calculations

$$\text{O.M}\% = [10 (B - C)/B] \times 0.003 \times (100/W_s) \times 1.72$$

Or:

$$\text{O.M} = N (B - C) \times 0.003 \times (100/W_s) \times 1.72$$



Name: _____

Determination of Nitrogen

Samples	Reading of Burette (V_{HCl}) ml
1	
2	

Calculation:

$$\text{Nitrogen percent \%} = [(V_k \times N_k) - (V_a \times N_a)] \times \frac{V_t}{V_s} \times \frac{14}{1000} \times \frac{100}{\text{wt.}}$$

Where:

V_t (Total volume of fertilizer solution = 1000ml).

V_s (Volume of fertilizer titrated sample = 10ml).

V_k (Total volume of NaOH).

N_k (Normality of NaOH).

V_a (Reading of Burette for acid).

N_a (Normality of acid).