

Practical Manual

SOIL MINERALOGY, GENESIS AND CLASSIFICATION

APS-504, 3(2+1)



For

M.Sc. (Ag.) Soil Science



2023

Department of Soil Science

College of Agriculture

Rani Lakshmi Bai Central Agricultural University
Jhansi-284003

Practical manual

**SOIL MINERALOGY, GENESIS AND
CLASSIFICATION**

APS-504, 3(2+1)

M.Sc. (Ag.) Soil Science

DR. SUSHEEL KUMAR SINGH

DR. BHARAT LAL

DR. ANSUIYA PANDA

Department of Soil Science

College of Agriculture

Rani Lakshmi Bai Central Agricultural University

Jhansi-284003

CONTENT

Ex. No.	Practical's	Page No	Remarks/sign.
1	Separation of sand silt and clay fraction from soil	1-4	
2	Determination of specific surface by ethylene glycol method.	5-8	
3	Determination of cation exchange capacity of soil	9-10	
4	Pre treatment of sample for X-ray scanning	11-13	
5	X-ray scanning of the sample.	14-17	
6	Identification of minerals present in soil sample	18-20	
7	Identification of clay mineral by differential thermal analysis (DTA)	21-22	
8	Identification of clay mineral by infrared spectroscopy	23-25	
9	Identification of different rocks	26-30	
10	To study about the identification criteria of different minerals.	31-32	
11	To study about the description of soil profile	33-34	
12	Determination of soil color by using munsell color chart	35	
13	To study about the different chemical weathering indices for soil development	36-38	

Experiment No. 1

Objective: Separation of sand silt and clay fraction from soil

Principle

In soil, specific minerals tend to be more prevalent in specific size ranges of particles. Therefore, separate XRD analyses of soil separates of different sizes, i.e. sand (2-0 mm), silt (0.05-0.002 mm), and clay (< 0.002 mm) largely size separates before XRD analysis. The segregation of sand, silt and clay can be achieved based on their differential sedimentation velocity in water, but before dispersion of soil particles is necessary. The dispersion of soil particles can be attained by exclusion of flocculants, i.e. the soluble electrolytes and exchangeable polyvalent cat followed by the removal of cementing agents, e.g. carbonates of calcium or magnesium (CaCO_3 , MgCO_3 , CaCO_3 , MgCO_3), organic matter, colloidal iron oxides and hydro Fe_2O_3 , FeOOH , etc.), manganese oxide (MnO_2), etc; and saturating the exchange with a highly hydrated and easily dissociable cation, eg Na^+ . After the dispersion of sand can be separated by a 300-mesh (53 μm) sieve. After separating sand, the remaining suspension contains silt (0.05-0.002 mm) and clay (<0.002 mm) particles. The particles can be separated from silt by employing differential settling in a liquid (us water) based on the relationship between particle diameter and settling velocity particle in a given liquid by Stokes' law (Stokes 1851) as per Eq. 1

$$V=g(S_p-S_l)d^2/18\eta \text{Eq 1}$$

where is the terminal velocity (cm sec^{-1} the parle liquid medium, g is the acceleration due to gravity (980 cm sec^{-2}) S_p , and S_l are the density (gcm^{-3}) of the particle and the liquid, respectively, d is effective diameter of the particle (cm), and η is the viscosity of the liquid (poise or g cm sec^{-1}). Equation 1 shows that settling velocity is directly proportional to the gravitational force acting on the particle, density of the particle relative to the liquid, and particle diameter, and is inversely proportional to the of the liquid. If h is the depth of fall (cm) of the particle after time t (sec), then.

$$V=h/t$$

Eq 2

From Eq. 1 and Eq. 2 we get

$$t = \frac{18\eta h}{g(S_p-S_l)d^2}$$

Apparatus

- Balance
- pH meter
- Hot water bath
- Centrifuge

Reagents

- 1M Sodium acetate (NaOAc) of pH 5: Dissolve 82 g of NaOAc in approximately 600 mL of distilled water Add 27 mL of glacial acetic acid and dilute to about 980 ml. with distilled water Adjust the pH to 5 with dilute acetic acid (CH_3COOH) or dilute sodium hydroxide (NaOH) solution. Then make up the volume to 1 L.
- 30% Hydrogen peroxide (H_2O_2) .
- 1 M NaOAc of pH 7. Dissolve 82 g of NaOAc in approximately 800 mL of distilled water. Adjust

- the pH to 7 with acetic acid (CH_3COOH) or dilute sodium hydroxide (NaOH) solution. Then make up the volume to 1 L.
- iv. 95% Methanol which is brought to pH 3 with hydrochloric acid (HCl).
 - v. 0.3 M Sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$)-0.125 M sodium bicarbonate (NaHCO_3): Dissolve 88 g of $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ and 10.5 g NaHCO_3 in distilled water and dilute to 1-L in a volumetric flask.
 - vi. Sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$).
 - vii. 99% Methanol (CH_3OH).
 - viii. 1 M Sodium chloride (NaCl): Dissolve 58 g of NaCl in distilled water and dilute to 1-L in a volumetric flask.
 - ix. 1 M Sodium hydroxide (NaOH). Dissolve 10g of NaOH in distilled water and dilute to 250-mL in a volumetric flask.
 - x. Thymolphthalein indicator Dissolve 0.04 g of thymolphthalein in a mixture of 50 mL ethanol and 50 ml. distilled water.
 - xi. 0.5M Ammonium carbonate ($(\text{NH}_4)_2\text{CO}_3$)
 - xii. Solid sodium carbonate (Na_2CO_3)

Procedure

(a) Removal of Soluble Electrolytes and Calcium Carbonate

(i) For Non-calcareous Soil

- Take 20 g of air-dried soil (passed through a 2-mm sieve) in a 200 ml centrifuge tube (the required sample size varies with the nature of the soil or deposit being studied)
- Add 100 mL of 1 M NaOAc (pH 5) and shake for 30 min.
- Centrifuge the suspension and discard the supernatant liquid
- If large centrifuge machine is not available, follow the procedure Calcareous soil

(ii) For Calcareous Soil

- Take 20 g of air-dried soil (passed through a 2-mm sieve) in a 1-1 glass beaker.
- Add 100 mL of 1 M NaOAc (pH 5) and heat the suspension on a water bath at near- boiling temperature for 30 min with occasional stirring
- Transfer the contents to centrifuge tubes, centrifuge the suspension and discard the supernatant liquid (alternatively, allow the suspension in the beaker to settle and siphon out the supernatant with a thin capillary tube) steps 1 to 3 should be repeated until CO_2 bubbles cease to occur indicating complete decomposition of carbonates.

The next steps are similar for both types.

(b) Removal of Organic Matter

- After 1 M NaOAc (pH 5) treatment, transfer the soil with distilled water to a 1-L glass beaker (if supernatant was directly removed by siphoning from the beaker, then continue with the same beaker) and heat the content on a water bath to evaporate the excess water. The water content in sample should be kept at minimum (just above saturation, complete drying must be avoided) to avoid dilution of H_2O_2 , which is added in the next step. The H_2O_2 treatment is more effective in acid medium.
- Take the beaker out of the water bath, add 5 ml. of 30% H_2O_2 , and stir with a glass rod for 5-10 min or till the frothing subsides. Then cover the beaker with a cover glass, place it on a water

bath and heat the contents. This may cause further frothing which should be allowed to stop before proceeding to the next step. After this, scrub and rinse down the soil particles adhered to the inner walls of the beaker with a second instalment of 5 mL of 30% H₂O₂. Stir the contents add an additional 10 ml of 30% H₂O₂, followed again by stirring. Then cover the beaker with a cover glass and digest the contents for 2-4 h by heating the beaker on a water bath The H₂O₂, treatment should be continued until frothing becomes negligible, indicating that almost entire amount of organic matter has been oxidized.

(c) Removal of Iron Oxides

- Transfer the organic matter free soil to centrifuge tubes and wash thrice with 1N NaOAc (pH 5), once with 95% methanol, once with 99% methanol and finally twice with 1 N NaOAc (pH 7) to raise the pH near to 7.0. At every step, discard the liquid by centrifugation and decantation. (Alternately, the washings may be done by decantation or siphoning out of supernatant from the beaker after allowing the suspension to settle by keeping overnight.)
- Transfer the soil to a 1-L glass beaker (if the soil was transferred into centrifuge tubes in the previous step, otherwise continue with the same beaker), add 40 mL of 0.3 M sodium citrate-0.125 M sodium bicarbonate solution and place the beaker on a water bath. Heat the contents to bring the temperature to 75-80 °C (not more since this will form sulphide from dithionite and will cause aggregation; and not less as the reaction may remain incomplete), then add 1 g of solid sodium dithionite (Na₂S₂O₄). Stir the mixture continuously for 1 min and intermittently for 5 min. Again add 1 g Na₂S₂O₄, and repeat the stirring as before. Add the third portion of 1 g Na₂S₂O₄ and stir as before till the colour of the suspension becomes gray to bluish (in case of montmorillonitic soil) or whitish (in case of kaolinitic soil). In this treatment of citrate-bicarbonate-dithionite (CBD), sodium citrate acts as a chelating agent for ferrous iron which is released by the reduction of ferric iron by sodium dithionite. Sodium citrate can also chelate ferric iron present in solution. The sodium bicarbonate acts buffering agent to maintain the pH near 7.3.
- Transfer the contents into centrifuge tubes immediately after CBD treatment, and centrifuge for 1 min at 2000 RPM. Discard the supernatant liquid by decantation and wash the soil residue twice with 95% methanol. After each washing, centrifuge the content and discard the supernatant by decantation. Washing has to be given immediately after CBD treatment otherwise re-oxidation of iron will take place. Transfer the material to a 1-L beaker.
- Add 100 mL of distilled water and 1 N NaOH in small increments, alternated with vigorous agitation until a pH of 9.5 is obtained, as indicated by the blue colour with thymolphthalein indicator when small drops of the suspension are tested on a spot plate. This pH will help in dispersion of the suspension.

(d) Removal of Free Silica (optional)

- Transfer the sample to a 500-mL Ni dish, add solid Na₂CO₃ to make its 2% (pH 10.5) and boil the content for 5 min and then centrifuge. This will dissolve and remove free silica, which otherwise may affect sharpness of XRD peaks.

(e) Separation of Sand, Silt and Clay

- Transfer the suspension to a 2.5-L glass bottle (or settling bottle) and add distilled water up to the neck. After 8 h (It may slightly vary with temperature. At a higher temperature, viscosity decreases and therefore it will take less time. One should always take more time than that prescribed to avoid siphoning of bigger particles than clay.), siphon out the liquid from top 10

cm of the settling bottle and store in another 2.5-L glass bottle (or storage bottle) (Alternatively, siphon out the liquid from top 20 cm of the settling bottle after 16 h). Add distilled water to the content of settling bottle up to the neck, shake the content gently, and keep for 8 h (or 16 h), then again siphon out the liquid from top 10 cm (top 20 cm for 1 settling time) of the settling bottle and store in the same storage bottle as earlier. Repeat this exercise until the liquid in the top 10 cm of the settling bottle becomes clear after 8 h settling time (or top 20 cm after 16 h). The suspension in the storage bottle contains clay particles, whereas the suspension remaining in the settling bottle contains sand and silt particles.

- To separate the sand from the silt, place a large polypropylene funnel on a 1-L measuring cylinder (or 1-L tall form beaker) and put a 3- μ m (300-mesh) sieve on a Then, shake the contents of the settling bottle vigorously for 40 sec. Transfer (in installments) the entire suspension of the settling bottle to the sieve using jets of water from a wash bottle. Wash the solids through the sieve using jets of water from a wash bottle until the effluent is clear. In this way, the silt particles will pass through the sieve, and sand particles will remain on the sieve. Remove the sieve from the funnel and transfer the sand particles quantitatively to a 250-ml glass beaker using jets of distilled water.
- Dry the sand by keeping at 105 °C in a hot-air oven and then store in a container. Transfer the silt from the measuring cylinder (or beaker) to a labelled container The volume of the suspension in the storage bottle containing clay particles should be reduced for convenience in storing and further analysis. For this, add solid NaCl or concentrated solution of NaCl (say 1 M NaCl) to the suspension, shake the content and then leave it for a few hours for the clay particles to settle. A clear supernatant indicates complete settling of the clay particles: Siphon out the liquid to reduce the volume of the clay suspension

Determination of Clay Concentration

- For determination of clay concentration, the excess salt has to be removed from the clay suspension. Make up the volume of the clay suspension to a particular volume, say 250 ml. Take 50 mL suspension in centrifuge tubes and wash it five times with 0.5 M ammonium carbonate which will remove sodium chloride. Transfer the suspension in a pre-weighed beaker in an oven for drying at 110 °C. This will dry the suspension as well as decompose the excess ammonium carbonate into ammonia and carbon dioxide, which will escape. Weigh the beaker with dried clay and determine the amount of clay present in 50 ml. and thus determine the concentration, *ie*, amount present in 100 mL suspension. The volume of the clay suspension taken for determination of clay concentration can be varied as per convenience.

Experiment No. 2

Objective: Determination of specific surface by ethylene glycol method.

Physical and chemical properties of a substance are highly influenced by the extent of its surface area. Soils differ markedly in surface area as a result of differences in texture, type of clay mineral and amount of organic matter. Some important properties like surface charge densities of soil solids or clays, retention of water and cations by soil, cation exchange capacity of soils are highly correlated with the surface area of soils. The term specific surface may be defined as the total surface area per unit mass of dry soil (S_m) or the total surface area per unit volume of soil solid (S_{vs}) or the total surface area per unit volume of soil (S_{vt}). S_m is expressed as square centimetre of surface per gram of dry soil; S_{vs} is expressed as square centimetre of surface per cubic centimetre of soil solids and S_{vt} is expressed as square centimetre of surface per cubic centimetre of soil.

$$S_m = S_A/M_s$$

$$S_{vs} = S_A/V_s$$

$$S_{vt} = S_A/V_t$$

Where, S_A is the total surface area, M_s is the mass of soil solids or dry soil, V_s is the volume of soil solids and V_t is the total volume of soil.

Methods of determination

Specific surface may be measured by various methods. The most common methods are BET method and ethylene glycol method. Generally external surface area is determined by BET method and total (i.e. internal plus external) surface area is determined by ethylene glycol method. Only external surface can also be determined by ethylene glycol method for which the measurements are to be made after heating the soil sample or clay at 600°C for 2 hours to suppress interlayer swelling. As both total and external surface area can be measured by ethylene glycol method, this method is described here.

Principle

Ethylene glycol is a polar molecule and it adsorbs on the surface of clay minerals. The principle of this technique is that the soil samples are dried in the air and the dry surface of the soil is saturated with ethylene glycol in a vacuum desiccator. The excess of this polar liquid is removed under vacuum over either anhydrous CaCl_2 or CaCl_2 - ethylene glycol mixture. When the weight of the clay mineral glycol mixture reaches a constant value, the surface area is calculated from the following equation:

$$A = W_g / (W_s \times 0.00031)$$

Where A is the specific surface (m^2/gm),

W_g is the weight of the ethylene glycol in the sample,

W_s is the weight of the air dry sample and 0.00031 is the gram of glycol which are required to form a monolayer per square metre of surface [the amount of ethylene glycol that are required to form a monolayer on the absorbing surface is $3.1 \times 10^{-4} \text{ gm}/\text{m}^2$]. By this method total (internal + external) surface is determined.

When the expanding lattice type minerals are heated to 600°C, the inter lamellar layers collapse irreversibly and the ethylene glycol is adsorbed only on external surfaces. So, if specific surface is determined after heating the sample, only external surface is measured by this method.

The internal surface of expanding lattice type clays like montmorillonite and vermiculite can be determined by comparing ethylene glycol adsorption before and after heating the sample.

Materials required

Vacuum desiccator (25 cm in diameter) with porcelain plate,

vacuum pump capable of reducing the pressure to 0.025 mm of Hg,

spatula, hot-air oven, desiccator, aluminium moisture boxes (diameter 6-7 cm and height not exceeding 2 cm),

pipette (1 ml), balance (0.0001 gm sensitivity), culture chamber of glass dish having a diameter of 20 cm and ethylene glycol, CaCl₂ - glycol solvent buffer, phosphorus pentoxide (P₂O₅), calcium chloride (40 mesh anhydrous reagent grade), hydrogen peroxide.

Procedure

(i) Preparation of CaCl₂ - Glycol solvent :

- About 120 gm of 40 mesh CaCl₂ is dried in an oven at 210°C for 1 hour or more to remove all traces of water.
- 20 gm ethylene glycol is weighed in a 400 ml beaker. The CaCl₂ is removed from the oven. 100 gm hot CaCl₂ is weighed without cooling and is added to the beaker containing glycol.
- Immediately the contents of the beaker are mixed thoroughly with spatula.
- The heat of the CaCl₂ facilitates solvation. After cooling of the solvent, the mixture is transferred into a culture chamber and it is spread uniformly over the bottom.
- This is stored in a sealed desiccator.

(ii) Sample pre-treatment

- About 10 gm of air dry 2 mm sieved soil or about 2 gm clay is treated with H₂O₂ and placed on hot water bath at about 65 to 70°C to remove organic matter.
- The sample is saturated with calcium by leaching with an excess of 1.0 N CaCl₂. Excess CaCl₂ is removed by repeated washing with water.
- The sample is dried in the air, grinded if necessary and again passed through a 60 mesh (2 mm) sieve. If it is desired to measure the external surface, about 4 gm sample is heated at 600°C for 2 hours to suppress interlayer swelling.

(iii) Sorption of ethylene glycol

- 0.3 gm of airdry clay or 1.1 gm of air dry soil is weighed in two aluminium boxes separately and the sample is spread evenly over the bottom of the box. The lid of the box is removed and the box is placed on the porcelain plate of a vacuum desiccator containing about 250 gm of P₂O₅.
- The desiccator is evacuated by applying vacuum for 1 hour by a vacuum pump. The stopcock of the vacuum desiccator is closed and the sample is dried for a period of 5 to 6 hours till a constant weight is obtained.
- The dried samples are then weighed accurately. 1 ml of ethylene glycol is taken in a pipette and added dropwise over the entire surface of one sample till the entire sample becomes wet.

- The box is placed in an oven at a temperature of 50 to 60°C for few minutes to facilitate wetting. The boxes containing the wetted and non-wetted samples are placed in a culture chamber on a hardwar cloth over approximately 120 gm of CaCl₂ - glycol solvate to shorten the mean diffusion path of glycol vapour and enhance equilibrium.
- The chamber is covered and placed in a vacuum desiccator having temperature 25 ± 1°C. The desiccator is evacuated by a vacuum pump for 1 hour. The stopcock is closed and the sample is left in vacuum for 48 hours with 2 to 3 evacuations for about 30 minutes after about 16 hours to remove any foreign vapours which may have reduced the vacuum.
- The vacuum is released; the boxes are covered with lids and weighed accurately. The weights of the ethylene glycol retained by 1 gm of wetted and non wetted samples are calculated separately to obtain their specific surface.
- Then the corrected specific surface of clay or soil is calculated. This is total specific surface of clay or soil. If it is desired to measure the external specific surface, the sample heated at 600°C for 2 hours is taken instead of or air dry sample and the external specific surface is determined in the same way as stated above.
- The internal specific surface is calculated from the difference between total specific surface and external specific surface of the sample.

Observations and calculations

(A) Total specific surface: Air dry sample is taken for measurement

		I wetted sample	II Nonwetted sample
(a)	Wt. of m. boxgmgm
(b)	Wt. of m. box + dry sample =gmgm
(c)	Wt. of dry sample (b-a) =gmgm
(d)	Wt. of m. box +sample +ethylene glycol =gmgm
(e)	Wt. of glycol retained (d- c) =gmgm
(f)	Wt. of glycol retained by 1 gm dry sample (e/c) =gmgm
(g)	Specific surface of sample (f/ 0.00031)m ² /gmm ² /gm
(h)	Correlated specific surface of the sample = [g(I) - g(II)] m ² /gmm ² /gm

(B) External specific surface: sample heated at 600°C is taken for measurement

		I wetted sample	II Nonwetted sample
(a)	Wt. of m. boxgmgm
(b)	Wt. of m. box + dry sample =gmgm
(c)	Wt. of dry sample (b-a) =gmgm
(d)	Wt. of m. box +sample +ethylene glycol =gmgm
(e)	Wt. of glycol retained (d- c) =gmgm
(f)	Wt. of glycol retained by 1 gm dry sample (e/c) =gmgm
(g)	Specific surface of sample (f/ 0.00031)gm gm

(h)	Correlated specific surface of the sample = $[g(I) - g(II)]$m ² /gm m ² /gm
-----	--	-------------------------	--------------------------

(C) Internal specific surface = $[A(h) - B(h)] = \dots\dots\dots m^2/gm$

Precautions

- Care should be taken to minimize adsorption of atmospheric water during weighing of dried sample. The moisture boxes must be covered with lid when they are outside the desiccator. The sample is to be weighed quickly.
- The sample or the aluminium box should not be touched with hand. Tongs or clean paper strip should be used for taking the aluminium box inside or outside the desiccator.
- Sufficient care is to be taken to prevent contamination of ethylene glycol by water vapour. If large amounts of contamination are suspected, ethylene glycol is to be redistilled under reduced pressure.

Experiment No. 3

Objective: Determination of cation exchange capacity of soil.

Principle

When a sample of soil is placed in a solution of a salt, such as ammonium acetate, ammonium ions are adsorbed by the soil and an equivalent amount of cations is displaced from the soil into the solution. This reaction is termed as 'cation exchange', and the cations displaced from the soil are referred to as 'exchangeable'. The surface-active constituents of soils that have cation-exchange properties are collectively termed as 'exchange complex' and consists for the most part of various clay minerals and organic matter. Soil mineral and organic colloidal particles have negative valence charges that holds dissociable cations and are thus called 'colloidal electrolytes'. The cation exchange capacity determination involves measuring the total quantity of negative charges per unit weight of the material. Stated otherwise the total amount of exchangeable cations that a soil can retain is designated as the cation exchange capacity and is usually expressed as milliequivalents per 100 g soil or $[\text{cm.l. (p+)}\text{kg}^{-1}]$. The determination of CEC is of fundamental importance in soil chemistry research. Adsorption, desorption and leaching of fertilizers, thermodynamic study of ion exchange; retention and release of nutrients, agrochemicals, soil pollutants, all depends upon the exchange capacity of the soil; CEC is also found to be an important parameter for soil classification. The cation exchange capacity is usually measured by leaching the soil or colloid with neutral normal ammonium acetate. Then the excess salt is removed by washing with 95% ethanol. The ammonium ion (NH_4^+) is then determined by steam distillation with magnesium oxide in an alkaline medium. The ammonia evolved is adsorbed into a known quantity of the standard acid containing methyl red indicator and the excess acid back titrated with a standard alkali.

Reagents required:

- 1(N) NH_4OAc adjusted to $\text{pH} = 7$; Dilute 60 ml glacial acetic acid (99.5%) and 75 ml concentrated ammonia solution (sp.gr.0.91, 25% NH_3) to 1 litre. Mix well, cool and adjust the pH of the solution to 7.0 with dilute acetic acid or ammonia solution. Alternatively, weigh 77.08 g NH_4OAc and dissolve in one litre distilled water and adjust the pH to 7 carefully with dilute acetic acid or ammonia solution.
- Ethanol 60%
- Ammonium chloride (AR)
- Magnesium oxide-carbonate free, freshly ignited (ignite at 650°C for 2 hours and cool in a desiccator over KOH pellets, store in tightly stoppered bottle)
- Standard H_2SO_4 ; 0.1 (N)
- Standard NaOH ; 0.1 (N)
- Standard oxalic acid – 0.1 (N)
- Methyl red indicator
- NaOH; 45%
- Silver nitrate solution about 0.1 (M) : Dissolve 8.5 g AgNO_3 in 500 ml water. Add 2 ml concentrated HNO_3 and mix well.

Procedure

- Transfer without loss 10 g of air dry soil sample accurately weighed in a 250 ml beaker and add 50 ml of neutral normal ammonium acetate solution.
- Stir occasionally for an hour cover with watch glass and leave overnight.
- Filter the contents through Whatman No. 44 filter paper receiving the filtrate in a 250 ml

volumetric flask.

- Transfer the soil completely on to the filter paper and continue to leach the soil with 1(N) NH_4OAc (using 20 ml at a time), allowing the leachate to drain out completely before adding a fresh aliquot.
- Continue the process, until the flask is full to the mark.
- Preserve this for estimation of exchangeable bases (Na^+ , K^+ , Ca^{++} and Mg^{++}). The residue left on the filter paper is intended for determination of cation exchange capacity of the soils.
- Wash the residue left on the filter paper with 60% alcohol to remove excess ammonium acetate. To ensure this add a pinch of solid NH_4Cl to the residue on the filter paper and wash with alcohol till the filtrate is free from chloride (as tested with silver nitrate solution, the filtrate is perfectly clear when free from chloride). If the washing is to be interrupted such as for the night, attach a rubber tube to the tail of the funnel and pinch it tight with a clip when there is solution above the level of soil in the filter paper. i.e. in no case the soil should dry otherwise loss of ammonia may occur.
- Remove the soil with the filter paper into a 800 ml distillation flask and add about 200 ml of water and about 3 g MgO (one spoonful approximately).
- Add few glass beads and little liquid paraffin so as to avoid bumping and frothing during distillation.
- Pour 100 ml of 45% sodium hydroxide and immediately connect the distillation flask to the condenser and distill ammonia in a known excess of 0.1(N) H_2SO_4 (say 25 ml) to which a few drops of methyl red indicator is added. (Continue distillation to collect about 150 ml distillate).
- Back titrate the excess of acid with 0.1(N) NaOH . Standardize NaOH versus oxalic acid and H_2SO_4 versus standard NaOH .
- Perform a blank distillation without the soil on a similar volume of liquid.

Calculations

CEC is normally expressed in milliequivalents of the cation per 100 g soil, presently as $c \text{ mol}/(\text{pt}) \text{ kg}^{-1}$. Milliequivalent means the equivalent weight expressed in milligrams. For instance 20 g of Ca^{2+} represents 1 equivalent or 1000 milliequivalent Ca^{2+} . Likewise 18 mg NH_4^+ would represent 1 milliequivalent (meq) of NH_4^+ .

Since 1000 ml of 1(N) acid or alkali = 1.0 g equivalent of any cation.

It follows that 1000 ml of 1(N) acid or alkali = 1000 milliequivalents of any cation.

Therefore, 1 ml 1(N) acid or alkali = 1 milliequivalents of any cation

$$\text{Cation exchange capacity} = \left[(V_1 N_1 - V_2 N_2) \times \frac{100}{w} \right] c \text{ mol (pt) kg}^{-1}$$

where V_1 = ml of standard acid taken initially for ammonia absorption

N_1 = normality of standard acid

V_2 = ml of standard base used in back titrating of excess acid

N_2 = normality of standard base

w = weight of sample in g

Experiment No. 4

Objective: Pre treatment of sample for X-ray scanning

Principle

Sand and silt particles can be scanned as randomly oriented samples by X-rays after drying at 105 °C. The clay particles are compulsorily saturated with different cations before X-ray analysis to make them homo-ionic. For clay particles, both random and parallel orientation specimens should be prepared.

Apparatus

Centrifuge

Reagents

- (i) 1 N Calcium chloride (CaCl_2): Dissolve 73.5 g of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ in distilled water and make up the volume to 1 L volumetric flask.
- (ii) 1 N Calcium acetate ($\text{Ca}(\text{OAc})_2$) of pH 7.7: Dissolve 79 g of anhydrous $\text{Ca}(\text{OAc})_2$ in distilled water and dilute it to around 980 ml.. adjust the pH to 7.7 with dilute solution of $\text{Ca}(\text{OH})_2$ or CH_3COOH , the dilute to 1-1 in a volumetric flask.
- (iii) 1N Magnesium chloride (MgCl_2) Dissolve 101.65 g of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ in distilled water and make the volume up to 1-1 in a volumetric flask.
- (iv) 1N Magnesium acetate ($\text{Mg}(\text{OAc})_2$) of pH 7.0 Dissolve 107 g of $\text{Mg}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ in distilled water and dilute it to around 980 ml., adjust the pH to 7 with a dilute solution of $\text{Mg}(\text{OH})_2$ or CH_3COOH , then dilute to 1-1 in a volumetric flask.
- (v) 1N Potassium chloride (KCl) Dissolve 74 55 g of KCl in distilled water and dilute to 1-L in a volumetric flask.
- (vi) 50% Methanol (CH_3OH)
- (vii) 95% Methanol
- (viii) 80% Acetone ($\text{CH}_3)_2\text{CO}$
- (ix) Ethanol-benzene (1:10) solution. Add 100 mL of ethanol to 1L of benzene and mix thoroughly
- (x) Ethanol-benzene (1:200) solution: Add 10 ml. of ethanol to 2 L of benzene and mix thoroughly.
- (xi) Glycerol-ethanol-benzene (4.5:100-1000) Add 45 mL glycerol to 100 mL of ethanol, then add benzene in small increments followed by shaking until 1 L benzene is added, and mix the contents thoroughly.
- (xii) 0.02 N Silver nitrate solution (AgNO_3) Dissolve 0.34 g of AgNO_3 in distilled water and dilute it to 100 mL.
- (xiii) 10% Glycerol Take 10 mL of glycerol in a 100-mL volumetric flask, and make up the volume with 95% ethanol, then mix the contents thoroughly.

Procedure

(i) Preparation of random oriented clay specimen

- Take 20 mL of clay suspension in a centrifuge tube, centrifuge and discard the supernatant, then add 25 mL of 1 N CaCl_2 , and mix the contents thoroughly, shake for 10 min and centrifuge to discard the supernatant.
- Then give two washings with 25 mL of 1 N $\text{Ca}(\text{OAc})_2$ of pH 7.7 (in a similar way as with 1N CaCl_2) to effectively remove the exchangeable H^+ .
- Again wash once with 25 ml of N CaCl_2 , to remove the excess $\text{Ca}(\text{OAc})_2$ and facilitate Cl test.

- Remove the excess salts from the clay suspension by washing the content once with 50% methanol, once with 95% methanol, and three or more times with 80% acetone and addition of AgNO_3 to the supernatant creates no precipitation, indicating complete removal of Cl.
- Then wash the sample twice with ethanol-benzene (1:10) solution, and three times with glycerol ethanol benzene (4.5: 100: 1000) solution. The sample sorbs glycerol from this solution.
- Then wash the sample once with ethanol-benzene (1:200) solution to get rid of the excess glycerol.
- Re-suspend the sample in benzene and transfer it to a watch glass.
- After the clays are settled, discard the excess benzene and allow the sample to dry at room temperature
- Use the dried sample as random oriented clay specimen for X-ray scanning.

(ii) **Preparation of parallel-oriented clay specimen**

For parallel orientation, Separate specimens of clay have to be saturated with Mg and K.

a. **Preparation of parallel-oriented Mg-saturated clay specimen**

- Take 20 ml of clay suspension in a centrifuge tube, centrifuge and discard the supernatant, then add 25 mL of 1 N MgCl_2 , mix the contents thoroughly, shake for 10 min and centrifuge to discard the supernatant. Repeat the washing 25 mL of 1N MgCl_2 once.
- Then give two washings with 25 ml. of 1 N $\text{Mg}(\text{OAc})_2$, of pH 7.0 (in a similar way as with 1N MgCl_2) to effectively remove the exchangeable H^+
- Wash once again with 25 mL of 1 N MgCl_2 , to remove the excess $\text{Mg}(\text{OAc})_2$, and facilitate Cl test .
- Then remove the excess salts from the clay suspension by washing the content once with 50% methanol, once with 95% methanol, and three or more times with 80% acetone until the addition of AgNO_3 to the supernatant creates no precipitation, indicating complete removal of Cl.
- Then suspend the clay in small amount (around 10 mL) of distilled water and determine the clay concentration by evaporation of a known volume of suspension.
- After determining the clay concentration of the suspension, prepare a suspension containing 2% clay by allowing necessary dilution with distilled water.
- Take a glass slide (2.6 cm x 4.6 cm) and rub one of its sides on a rough surface to make that side sufficiently rough so that it can hold the clay particles after the water is evaporated Wash the slide with distilled water and dry it.
- Label the smooth side of the glass slide, and place it on a smooth surface.
- Pipette out 2 mL of the 2% clay suspension and spread on the rough side of the labelled glass slide.
- Allow the suspension on the slide to dry at room temperature.
- The air-dried specimen can be scanned by X-ray to get the diffractogram of Mg saturated air-dried clay (Mg_{air}).
- Then add sufficient 10% glycerol (in ethanol) solution to the clay by a chromatographic sprayer and allow it to dry at room temperature.
- When excess glycerol evaporates to form a slightly moist clay specimen, again scan it under X-ray to get the diffractogram of Mg saturated glycerol solvated clay (Mg_{gly}).
- Alternatively, the Mg saturated clay can be dried out directly from a suspension of clay made with 10% glycerol (in ethanol) solution to get the Mg saturated glycerol solvated clay (Mg_{gly}) specimen.

b) **Preparation of parallel-oriented K-saturated clay specimen**

- Take 20 ml of clay suspension in a centrifuge tube, centrifuge and discard the supernatant
- Add 25 ml of 1 N KCl, mix the contents thoroughly, shake for 10 min and centrifuge to discard the supernatant. In this way, give five washings with 1N KCL.
- Then remove the excess salt from the clay suspension by washing the content once with distilled water, and five times with 99% methanol, once or more with 80% acetone until addition of AgNO₃ to the supernatant creates no precipitation, indicating complete removal of Cl
- Then suspend the clay in small amount (around 10 mL) of distilled water and determine the clay concentration by evaporation of a known volume of suspension.
- After determining the clay concentration of suspension, prepare a suspension containing 2% clay by allowing necessary dilution with distilled water.
- Take a glass slide (2.6 cm x 4.6 cm) and rub one of its sides on a rough surface to make that side sufficiently rough so that it can hold the clay particles after the water is evaporated. Wash the slide with distilled water and dry it.
- Label the smooth side of the glass slide, and place it on a smooth surface.
- Pipette out 2 mL of the 2% clay suspension and spread on the rough side of the labelled glass slide.
- Allow the suspension on the slide to dry at room temperature.
- The air-dried specimen can be scanned by X-ray to get the diffractogram of K saturated air-dried clay (K_{air})
- Then heat the slide in a muffle furnace at 300 °C temperature for 2 h. After heating. cool the sample in a desiccator and scan it under X-ray (as soon as it becomes cold enough to handle), to get the diffractogram of K-saturated clay heated at 300 °C (K₃₀₀).
- Then heat the slide in a muffle furnace at 550 °C temperature for 2 h. After heating. cool the sample in a desiccator and scan it under X-ray (as soon as it becomes cool enough to handle) to get the diffractogram of K-saturated clay heated at 550 °C (K₅₅₀)

Experiment No. 5

Objective: X-ray scanning of the sample.

Principle:

When a monochromatic and parallel beam of X-rays is incident upon a crystal surface at a particular angle, also known as the Bragg angle, some of it would get diffracted from the subsequent planes of the crystal and leave the crystal at the same angle (θ) with respect to the planes. The identification of minerals present in soil solids by XRD is based on the quantitative relationship of the Bragg angle with the distance (d) between the successive atomic planes of the crystal, as given by the Bragg law below equation.

$$n\lambda = 2d \sin\theta$$

where n is the order or degree of diffraction, and λ is the wavelength (in nm or Å) of the incident X-ray. The unit of d is same as the unit of λ

The parallel and monochromatic incident X-ray of wavelength λ strikes the crystal at point 'O' of the first atomic plane and point 'C' of the second atomic plane, making an angle θ with each of the atomic planes. After diffraction, the X-rays leave the crystal making the same angle θ with each of the atomic planes. The value of θ , that is for the diffraction of the X-ray of a given wavelength (λ), depends on the distance (d) between the successive atomic planes of the crystal lattice. The X-rays in the incident beam are in phase along the wave-front 'OA'. To prevent destructive interference of X-rays after interacting with the atomic planes, the outgoing X-rays must also be in phase along the wave front 'OB'. The X-ray which has travelled through the point 'C' on the second atomic plane must have travelled the additional path length of 'AC+CB' than the X-ray that has travelled through the point 'O' of first atomic plane. From geometry, we can find that

$$AC = OC \sin\theta$$

and

$$CB = OC \sin\theta$$

and

$$OC = d$$

and therefore

$$AC + CB = 2d \sin\theta$$

To keep the outgoing X-rays in phase, the additional path travelled must be equal to λ its integral multiple. Hence, the Bragg condition for X-ray diffraction from crystal planes can be written as

$$n\lambda = 2d \sin\theta$$

where, n is an integer number and indicates the order of diffraction.

The X-ray diffractograms of different samples can be obtained by

- (1) Debye-Scherrer powder camera method, and
- (2) Spectrometric method.

The working principles of powder camera and spectrometric method are discussed in the following sections:

(i) Principle of powder camera method

This method is also known as Hull-Debye-Scherrer method as it was independently devised by Hull in USA and Debye and Scherrer in Germany between 1915 and 1917. Let AB be a crystal plane, XY and YL are the incident and diffracted beams, respectively, making an angle θ with the plane AB. The YX is the direction of the original X-ray beam. Thus, the angle between diffracted and original X-ray beam is 2θ . Since this is a powder sample, this plane must be present in the sample in all possible orientations which will produce a cone LYM on the surface of which lie the diffracted beams. Thus, by geometry this cone is having an apical angle of 4θ (in radians). This cone when intercepted by a cylindrical X-ray sensitive film, two curved lines, GLH () and QMP (re produced on the film by diffracted beams. The length of the film, S, between the 1-points of the two curved lines, i.e. between L and M forms an arc of a circle of radi, which is related to the apical angle by

$$S = R \times 4\theta$$

where, 4θ is the apical angle in radian, i.e. the angle subtended by the arc at the centre of the circle. Here, R is known for a particular camera, S is measured after exposure of the sample to X-rays, and from S, θ is calculated. Then d is obtained from θ using Bragg equation.

The camera used in this technique is made up of metal, and is cylindrical. It has two openings on diametrically opposite sides through which are cone-shaped tubes called collimators. The X-ray beam after passing through the mal fiber passes through one collimator which directs the X-ray beam to the cylindrical wire shaped sample protruding out of the cylindrical sample holder at the centre of the camera. The other collimator directs the original beam to an absorber. Due to these collimators scattering of X-ray by air molecules is reduced and hence the background radiation diminished. There is provision of rotating the sample holder slowly and asymmetrically so that all the atomic planes, of the crystals present in the sample, in all possible orientations get enough chances to be exposed to the X-ray beam. An X-ray sensitive film strip, which captures the diffracted X-ray beam, is placed inside the whole periphery of the camera and fixed with a clip. There are two different sizes of camera. The larger one has a diameter of 114.6 mm and the smaller one has a diameter of 57.26 mm so that half the circumferences of these two cameras are 180 mm and 90 mm, respectively.

(ii) Principle of spectrometric technique: Bragg-Brentano Diffractometer or X-ray Diffractometer

In this technique, instead of camera a cylindrical radiation shield is used. From one side of this shield, X-ray beam is allowed to enter after passing successively through a filter, collimator slits and a divergence slit. The X-ray then falls on a flat specimen at the centre of the shield. The diffracted beam from the sample is received in a proportional or GM

counter after passing successively through an anti-scatter slit, collimator slits and a receiving diffracted beam produces electric pulse after falling on a detector. The current of the is proportional to the intensity of the diffracted beam, and is plotted on a recorder or transferred to a computer. The proportional counter is fixed on a goniometer which keeps track of the angle 2θ between the direction of diffracted beam and the original X-ray beam. The angle 2θ is varied continuously or step by step by

moving the goniometer. This process is called scanning. For the angles of incidence (θ), where Bragg condition is satisfied, diffraction maxima (ie peaks in X-ray diffractogram) are produced. The order of diffraction (n), for a particular d -value, increases with θ .

the flat sample is tangent to the focusing circle of radius R . The sample to source distance (FS) and sample to receiver distance (SG) are the same and equal to the radius of the circle described by the goniometric point 'G' revolving about the sample around point 'S' (Centre of Goniometer circle). The triangle SGG" is an axial section through a diffraction cone of angle 4θ (in radians) diverging from 'S'; obviously this triangle is the intersection of this cone with the plane of the goniometer arc. The diffracted rays come to an approximate focus at 'G', the position of the receiving slit and diverge again as they enter the counter tube. The focusing action is not perfect because of the use of flat specimen rather than the one with a continuously changing curvature. There are also other factors such as penetration of X-ray beam into the specimen, instrumental aberration, and the size and wavelength distribution of the source which make the lines being displaced slightly from their correct positions causing slight distortions in the profiles. During scanning, the sample surface is rotated with one-half the angular speed with which the goniometer or receiver revolves, so that the sample surface remains tangent to the focusing circle at all times. This is known as the ' $\theta:2\theta$ ' type arrangement. During scanning, the goniometer keeps track of angle of incidence in terms of 2θ , i.e., the angle between the diffracted beam and original beam. As the goniometer moves about point 'S' on the sample towards larger angles of incidence, the radius of the focusing circle decreases. It can be shown that the radius (R) of the focusing circle depends on the angle of incidence θ as per equation

$$R = \frac{FS}{2 \sin\theta}$$

For $2\theta = 0$, $R = \infty$, whereas at $2\theta = 180^\circ$, R reaches a minimum value of $FS/2$. It can also be shown that the base of the diffracted cone intercepted by the receiving slit is a circle of radius $R = FS \sin 2\theta$. Recent X-ray diffractometers mostly have ' $\theta: \theta$ ' arrangement, where the sample holder remains fixed while the X-ray source and the detector on goniometer move at same angular speed.

Apparatus

- Powder camera
- X-ray spectrometer or X-ray diffractometer

Procedure

(a) Procedure for Powder camera

- Take some of powdered clay sample, and add a little amount of collodion or gum acacia to form a paste and roll it to form a cylindrical shape.
- Take a capillary tube of 0.3 mm internal diameter, and push the cylindrical paste sample through the tube hole using a metal wire, so that a portion of it extrudes outside
- Place a film of known length on the camera and attach the collimators at both sides.
- Close the camera and attach the camera with an X-ray tube.
- Switch on the X-ray generator, maintain a potential difference of 40 kV and current of 20 mA and expose the sample to the X-ray for 30 min, while rotating the sample holder asymmetrically to ensure sufficient exposure of all possible orientations of the crystal planes present in the sample.
- After completion of exposure, take out the film and measure the length of the film.

- Measure the lengths of arcs impressed on the film caused by X-rays diffracted by the sample.
- Calculate the film-length correction factor (CF) by formula

$$CF = \frac{\text{Length of the film before exposure}}{\text{Length of the film after exposure}}$$

- Calculate the actual lengths of the arcs by

$$\text{Actual arc length (S}_A\text{)} = \text{Measured arc length (S}_M\text{)} \times CF$$

- From S_A calculate the Bragg angle (θ) by the formula

$$\theta \text{ (in radian)} = \frac{S}{4R}$$

Where R is the radius of the camera

Since π radian is equal to 180 degree, so above equation with θ in degree becomes,

$$\theta^\circ = \frac{S}{4R} \times \frac{180}{\pi}$$

$$\text{For larger camera, } R = \frac{180}{\pi} = 57.26$$

Therefore, above equation becomes

$$\theta^\circ = \frac{S}{4R} \times \frac{\pi}{180} \times \frac{180}{\pi} = \frac{S}{4}$$

$$\text{or, } \theta \text{ (in degree)} = S/4$$

- Calculate separation distance of subsequent atomic planes in the crystal (d) by Eq (Bragg law):

$$d = \frac{n\lambda}{2 \sin\theta}$$

where, n is the order of diffraction and λ is the wavelength of the X-ray used. The unit of d would be same as the unit of λ used in the equation, θ (in degree) = S/4

- From d, the minerals present can be identified.

(b) Procedure for X-ray Diffractometer

- Switch on the instrument and adjust the goniometer at a particular angle.
- Switch on the X-ray generator and adjust the potential difference and current as per the requirement of the X-ray diffractometer available.
- Place the random oriented powder specimen of sand, silt or clay or the parallel oriented specimen of clay mounted on glass slides on the sample holder, and close the section with the lid provided.
- Set the scanning range and scanning speed at desired values.
- Run the instruments to get the diffractogram of the sample.

Objective: Identification of minerals present in soil sample

In sand and silt

Mainly minerals are present in sand and silt particles, which can be identified by the d-spacing from below Table.

Table 1. Minerals present in sand and silt and their d spacings

Mineral	d spacing (nm)
Quartz	0.421, 0.335, 0.181
K-feldspar	0.321 – 0.328
Plagioclase feldspars	0.312 – 0.323
Mica	1.0, 0.5, 0.333
Hornblende	0.31- 0.325, 0.144

In clay

Based on random oriented specimens

From random oriented clay specimens, information on di-octahedral and tri-octahedral phyllosilicates can be separately obtained. Peaks (060) between 0.148 and 0.152 nm indicate di-octahedral phyllosilicate, while the peaks (060) between 0.1515 and 0.156 nm indicate tri-octahedral phyllosilicate.

Table 2. Basal spacing (nm) of clay minerals after undergoing different treatments

Mineral	Mg _{air}	Mg _{gly}	K _{air}	K ₃₀₀	K ₅₅₀
Smectite	1.4-1.5	1.77	1.23	1.00	1.00
Vermiculite	1.4-1.5	1.44	1.01	1.00	1.00
Chlorite	1.4-1.5	1.43	1.43	1.43	1.38
Mica or Illite	0.99-1.01	1.01	1.00	1.00	1.00
Kaolinite	0.715	0.715	0.715	0.715	No peak as it gets decomposed

Table 3. Identification of clay minerals with random interstratification among 2:1 mineral, and 2:1 and 2:1:1 minerals

Component mineral	Mica or illite	Vermiculite	Smectite
Chlorite	Between 1.0 and 1.4 nm in all treatments	1.4 nm in Mg _{gly} ; reduces to a value in between 1.4 and 1.0 nm in K ₅₅₀	Between 1.78 and 1.4 nm for Mg _{gly} ; reduces to a value in between 1.4 and 1.0 nm in K ₅₅₀
Smectite	Between 1.78 and 1.0 nm in Mg _{gly} ; between	Between 1.78 and 1.4 nm in Mg _{gly} ; between	

	1.23 and 1.0 nm in K_{air} ; reduces to 1.0 nm in K_{550} ; between 1.0 and 0.89 nm (2 nd order of smectite) in Mg_{gly}	1.23 and 1.0 nm in K_{air} ; reduces to 1.0 nm in K_{550}	
Vermiculite	Between 1.4 and 1.0 nm in Mg_{gly} ; reduces to 1.0 nm in K_{air} and K_{550}		

Table 4. Identification of clay minerals with regular interstratification among 2:1 minerals, and 2:1:1 minerals

Component mineral	Mica or illite	Vermiculite	Smectite
Chlorite	2.4 nm (1.0 nm + 1.4 nm) in all treatments	2.8 nm (1.4nm +1.4 nm) in Mg_{gly} ; 2.4 nm (1.0 nm + 1.4 nm) in K_{air} and K_{550}	3.18 nm (1.4 nm + 1.78 nm) in Mg_{gly} ; 2.63 nm (1.4 nm + 1.23nm) in K_{air} ; 2.4 nm (1.0 nm + 1.4 nm) in K_{550}
Smectite	2.78 nm (1.0 nm + 1.78 nm) in Mg_{gly} ; 2.23nm (1.0 nm + 1.23 nm) in K_{air} ; 1.0 nm in K_{550}	3.18 nm (1.4 nm + 1.78 nm) in Mg_{gly} ; 2.23 nm (1.0 nm + 1.23 nm) in K_{air} ; 1.0 nm in K_{550}	
Vermiculite	2.4 nm (1.0 nm + 1.4 nm) in Mg_{gly} ; 1.0 nm in K_{air} and K_{550}		

Based on parallel oriented specimens

The parallel oriented clay specimens provide information of c-axis spacing phyllosilicates as given in Table 2

The identification of interstratified minerals can be done using Tables 3 and 4.

Semiquantitative Estimation of Clay minerals

The following steps are followed:

- (1) Calculate the areas under different first order peaks of the Mg saturated and glycerol solvated diffractogram after suitably placing a background curve below the diffractogram
- (2) Correct the peak area by dividing with the following correction factors:

S No.	d-spacing (nm) of peak	Correction factor
(i)	1.78 and above	4 (for smectite and interstratified smectite)
(ii)	≥ 1.43 to < 1.78	3 (for vermiculite)
(iii)	≈ 1.43	1 (for Chlorite in the Mg -sat /550° C diffractogram)*
(iv)	>1.0 to <1.43	2 (for interstratified minerals)
(v)	≤ 1.0	1 (for illite , kaolinite)

*In the presence of chlorite (as confirmed by the presence of 1.38 nm peak in K_{550}), if area (iii) is less than area (ii), then vermiculite area = area (ii) - area (iii) and has to be divided by 3, and area (iii) is chlorite area which has to be divided by 1.

If the area (iii) is higher than area (ii), then there is no vermiculite, all is chlorite and divide the area (iii) by 1

(3) Add the corrected areas, say Δ

(4) Calculate the factor as, $F = 100/\Delta$

(5) Multiply the corrected area of individual minerals by factor F to get the percentage of minerals

Precautions

- After starting the dispersion process, drying of the sample must be avoided; otherwise exchangeable K^+ and NH_4 would get fixed in the interlayers of expanding 2:1 clay minerals and collapse the layers irreversibly upon removal of water of hydration.
- The initial washing with 1 N NaOAc (pH 5) removes the soluble electrolytes and exchangeable polyvalent cations which act as flocculants. Moreover, Ca^{2+} if present in excess, leads to the development of resistant gel-like form of soil humus, which slows down the oxidation of soil organic matter during H_2O_2 treatment. Such phenomena can be avoided by initial washing with 1 N NaOAc (pH 5) as it removes most of the Ca^{2+} . In case of calcareous soil, boiling with 1 N NaOAc (pH 5) dissolves the Ca and Mg carbonates, which are then removed with the discarded supernatant. Washing the soil with this acid buffer makes the soil suspension slightly acid and non-calcareous, which is very important for proper oxidation of soil organic matter, and also reduction and subsequent dissolution of MnO_2 by H_2O_2 .
- Contact of skin with H_2O_2 should be avoided since this reagent may cause severe skin burns. If, H_2O_2 comes in contact with skin or eye, immediately wash the contact-site with ample amount of cold tap water.
- One should watch the reaction of H_2O_2 with organic matter closely, as vigorous frothing may spill out some soil from the beaker. In case of vigorous frothing, add a jet of distilled water through a wash bottle to cut the froth and avoid spilling out of soil.
- The washing with 1 N NaOAc (pH 5) after H_2O_2 treatment is given to remove the cations released from organic matter during its decomposition by H_2O_2 . The final two washings with 1 N NaOAc (pH 7) are given to raise the pH so as to facilitate the next step, i.e. removal of iron oxides by CBD treatment.
- The X-ray diffraction analysis of clay is more sensitive to alumino-silicates free from iron oxides than when large amounts of iron oxides are present. The exclusion of free iron oxides and coatings greatly enhances the extent of parallel orientation of the crystalline alumino-silicate layers, and hence the basal diffraction intensity. The electron microscopic examinations and the differential thermal and infrared analyses are also aided by the exclusion of iron oxides. For the removal of iron oxides by CBD treatment, dithionite reduces the ferric to ferrous, citrate chelates the ferric and the ferrous (and prevents the formation of FeS), and bicarbonate buffers the pH of the system near pH 7.3. During CBD treatment, heating above 80 °C should be avoided, otherwise FeS may form at temperatures > 80 °C. If a brown or red colours persists, the treatment is repeated once more.

Experiment No. 7

Objective: Identification of clay mineral by differential thermal analysis (DTA)

The differential thermal analysis method, commonly referred to as DTA, is a widely used technique and is particularly useful especially in the identification of amorphous material when x-ray diffraction analysis yields only featureless curves. It found application first in geology and later was extended to analysis in ceramics, glass, polymer, cement, plaster, and in studies of organic matter, explosives, and radioisotopes.

Principle

Differential thermal analysis measures the differences in temperature developed between an unknown and a reference sample, as the two are heated side by side at a controlled heating rate from 0 to 1000°C. The reference material, also called standard material, is a substance that is thermally inert over the temperature range of the analysis. Heating rates may vary from 0.1 to 2000°C/min. For most purposes, a heating rate of 20°C is used. During the heating process, the unknown sample undergoes a thermal reaction and transformation. The latter is reflected by a difference in temperature between the unknown and reference samples. This difference in temperature is recorded by a set of thermocouples and plotted in a graph, usually against the temperature at which the difference occurs.

If the temperature of the unknown sample becomes lower than that of the reference material, ΔT is negative, and an endothermic peak is produced. When the temperature of the sample becomes higher than that of the reference material, ΔT is positive, and an exothermic peak develops. The portion of the curve for which $\Delta T = 0$ (no difference in temperature between unknown and reference sample) is considered the baseline. Ideally, the baseline is a straight line. Upon analysis by DTA, the mineral may undergo several thermal reactions, culminating in one or a series of endo- and exothermic peaks. The curve with the peaks serves as a fingerprint, and the specific temperatures at which the peaks develop are diagnostic for the identification of the mineral. In addition, the peak height or peak area of the main endothermic reaction can be used for quantitative determination.

- Generally, DTA can be performed with liquid or solid samples. With soil samples, whole soil, sand, silt, or clay fractions can be used.
- When whole soils are analyzed, the <2 mm fraction should be treated first with 30% H₂O₂ to remove organic matter, which may interfere by giving strong exothermic reactions. In general, analysis of whole soils gives only peaks of low intensities. These same peaks are very large and intense if the clay fractions are analyzed.
- However, the quartz inversion peak at 573°C (864 K) is often absent in DTA curves of clays.
- The sand can be analyzed using the total sand fraction (2 to 0.05 mm) or one of the following sand fractions:

Very coarse sand:	2.00 to 1.00 mm
Coarse sand:	1.00 to 0.50 mm
Medium sand:	0.50 to 0.25 mm
Fine sand:	0.25 to 0.10 mm
Very fine sand:	0.10 to 0.05 mm

- DTA of sand shows mostly a strong endothermic peak of quartz at 573°C (846 K), and of primary minerals. Therefore, it is of importance only in the investigation of primary minerals or iron-manganese concretions.
- DTA of silt yields curves with peak resolutions between those of sand and whole soils and clays. Usually, the thermogram of silt shows more details or complexity than that of sand.
- The clay fraction of soils (<2 μm) can be used directly or can be separated first, before analysis, into coarse clay (2 to 0.2 μm) and fine clay (<0.2 μm) fractions. For general purposes, the clay fraction <2 μm gives satisfactory results for qualitative and quantitative interpretations. The amount of clay to be used depends on the instruments used. Instruments equipped with well holders need approximately 10 to 100 mg,
- whereas those equipped with Pt cups placed on ring-type thermocouples need only 1 to 10 mg.
- In qualitative analysis, it is often unnecessary to weigh the sample for DTA, although comparison of curves should be made with curves obtained from identical amounts of samples. On the other hand, in quantitative analysis, the amount of sample must be weighed accurately, because the height or area of the main endothermic peak increases or decreases proportionally with sample size.
- Qualitative identification of minerals can be achieved by using the DTA curves as fingerprints and comparing or matching them with DTA curves of standard minerals or with curves of well-known established minerals.
- Each mineral exhibit's specific thermal reaction features. The DTA curve of kaolinite is characterized by a strong endothermic peak at 450 to 600°C and by a strong exothermic peak at 900 to 1000°C. The endothermic peak is caused by dehydroxylation, whereas the exothermic peak is attributed to formation of γ-alumina or mullite or both.
- The curve of halloysite is almost similar to that of kaolinite but has, in addition, a low-temperature (100 to 200°C) endothermic peak of medium to strong intensity for loss of adsorbed interlayer water.
- Smectite (montmorillonite) exhibits a DTA curve characterized by a low-temperature (100 to 200°C) endothermic peak, an endothermic peak between 600 and 750°C, and a small dip between 800 and 900°C, followed by a weak exothermic peak between 900 and 1000°C.
- Gibbsite and goethite are usually characterized by a strong endothermic peak only between 290 and 350°C. Often goethite and the other iron oxide minerals have their endothermic reactions at a higher temperature than gibbsite.
- Allophane exhibits DTA features with a strong low-temperature (50 to 150°C) endothermic peak, and a strong exothermic peak at 900 to 1000°C. The low-temperature endothermic reaction is attributed to loss of adsorbed water, whereas the main exothermic reaction is caused by γ-alumina formation.

Objective: Identification of clay mineral by infrared spectroscopy

Recently, infrared spectroscopy has found extensive application in clay mineralogical studies. Amorphous as well as crystalline clays absorb infrared radiation, and the method can be used when x-ray analysis makes identification difficult. The infrared absorption spectrum of a mineral has a characteristic pattern that not only permits the identification of the mineral, but also reveals the presence of major functional groups within the structure of the particular compound under investigation.

Principle

Infrared absorption is related to molecular or atomic vibrations, and only radiation with a similar frequency as that of the vibration will be absorbed. Atoms and molecules within a compound oscillate or vibrate with frequencies of approximately 10^{13} to 10^{14} cps. These frequencies correspond to the frequencies of infrared radiation, and infrared radiation can, therefore, be absorbed by molecular vibrations when the interaction is accompanied by a change in dipole moment. A rapid vibration of atoms yields a rapid change in dipole moment, and absorption of infrared radiation is intense. On the other hand, a weak vibration of atoms produces a slow change in dipole, and consequently, absorption of infrared radiation is relatively weak. Symmetric molecules will also often not absorb infrared radiation. Molecular or atomic vibrations cause the interatomic distance to change, because the atomic movement called oscillation, subjects the atoms to a periodic displacement relative to one another.

The frequency of vibration obeys the law of simple harmonic motion, which is formulated as follows:

$$V = \frac{1}{2\pi c} \sqrt{k/m}$$

Where,

V is the frequency of vibration in cm^{-1} ,

c is the velocity of light in vacuum,

m is the reduced mass of the vibrating atoms, and

k is the force constant in dynes/cm.

Two types of vibrations are distinguished

(1) stretching vibrations or deformation, in which the atoms are oscillating in the direction of the bond axis without changing bond angles; and

(2) bending vibrations, in which the movement of atoms produces a change in bond angles.

The restoring force acting on stretching vibrations is usually greater than that required to restore bending vibrations. Therefore, stretching vibrations occur at higher frequencies than bending vibrations.

- The highest frequencies observed in minerals are those stretching vibrations of hydroxyl, OH, groups that occur between 3700 and 2000 cm^{-1} .
- Bending vibrations occur at lower frequencies, from 1630 to 400 cm^{-1} .
- Liquid, gas, and solid samples can be used in infrared spectroscopy. Liquid samples are usually pipetted or injected into infrared cells provided with a NaCl or KBr crystal window.

- Gas samples are also introduced into cells, similar to the cells mentioned above. Infrared gas cells are larger than cells for liquid samples and ensure better interaction between infrared radiation and the gas by providing a longer path length. Both NaCl and KBr are infrared inactive and will not interfere in the analysis.
- Solid samples should be ground to approximately $<2 \mu\text{m}$, because coarse particles tend to produce scattering of infrared radiation. Clay fractions ($<2 \mu\text{m}$) separated by particle size distribution analysis can be used directly or can be separated first by centrifugation into coarse (2 to $0.2 \mu\text{m}$) and fine ($<0.2 \mu\text{m}$) clay fractions.
- If grinding is necessary, it should be carried out with care, because vigorous grinding tends to destroy the mineral structures (become amorphous) and tends to increase the hygroscopic nature of the sample.
- Several methods were proposed for mounting solid samples in infrared analysis: the (1) mull method, and (2) KBr pellet technique, by which a weighted sample (1 to 10 mg) is carefully ground with 100 mg KBr and pressed into a transparent pellet. Sodium chloride also appears to be suitable for use in the pellet method. The use of clay films has recently attracted considerable attention.
- However, one disadvantage of the clay film technique is that it takes more time to prepare a clay film than to make a pellet.
- In the clay film mounting technique, clay or soil samples are made into a suspension by sonification. They are then pipetted onto Irtran-II window cells, so that 1 mg/cm^2 or 5 mg/cm^2 are transferred onto the cells.
- After air drying at room temperature, the cells are scanned from 4000 to 600 cm^{-1} or lower. Band positions in infrared analysis are indicated in units of frequencies, expressed in terms of centimeters.
- The frequency V , also known as the wave number, is defined as the number of waves, or wavelengths, per centimeter (cm^{-1}). It is related to the wavelength λ as follows:

$$V = 10^4/\lambda$$

- In which V is expressed per centimeter (cm^{-1}) and λ in micrometers (μm). The region often analyzed by infrared spectroscopy is in the range of 4000 to 600 cm^{-1} (equivalent to 2.5 to $25 \mu\text{m}$) or lower.
- In many instances, the results of infrared analysis are recorded in the transmittance mode. The latter yields curves that have an upside-down appearance when compared with absorption curves.
- Two groups of frequency regions usually characterize the infrared curves of most clay minerals: 1. Region between 4000 and 3000 cm^{-1} attributed to stretching vibrations of adsorbed water or octahedral OH groups, called the functional group region. 2. Region between 1400 and 800 cm^{-1} , attributed to Al-OH or Si-O vibrations, called the fingerprint region.
- The infrared curve of kaolinite is usually characterized by two strong bands for octahedral OH-stretching vibrations between 3800 and 3600 cm^{-1} when the sample is mounted by the KBr pellet technique. An additional third and very sharp band is present at 3670 cm^{-1} when the samples are mounted as films on Irtran-II cells. The fingerprint region, or lower-frequency region, exhibits sharp bands for kaolinite at 1150 , and 1080 cm^{-1} for O-Al-OH vibrations using clay films on Irtran windows. In addition, a sharp 1020 cm^{-1} band for Si-O and sharp bands at 910 to 920 cm^{-1} for Al-OH vibrations are present. When using KBr pellets, the bands at 1080

and 1020 cm^{-1} appear only as a weakly segregated doublet in most analyses reported for kaolinite.

- Smectite also exhibits a better-resolved curve with the clay film technique on Irtran-II. The KBr curve is characterized by one broad band followed by a water band, and one additional dominant broad band at 3640 , 3420 , and 1050 cm^{-1} , respectively. However, if one uses clay films on Irtran-II, the bands at 3640 cm^{-1} for OH-stretching vibrations and at 1050 cm^{-1} for Si-O vibration become very strong and sharp. In addition, bands at 1150 , 910 , 880 , and 850 cm^{-1} increase sharply in intensity in clay film samples.
- Standard reference gibbsite (purchased from the Wards Scientific Establishment Co.) is characterized by an absorption band at 3620 cm^{-1} , and by a doublet at 3540 and at 3480 cm^{-1} . In the low-frequency region, gibbsite shows only one dominant peak at 1030 cm^{-1} for O-Al-OH vibration.
- This peak is the reason for determining the kaolinite band at 1080 cm^{-1} as a separate independent band, rather than calling it a doublet together with the 1020 cm^{-1} band. On the other hand, soil gibbsite exhibits an infrared curve characterized by a triplet between 3600 and 3400 cm^{-1} , instead of the doublet observed for the reference gibbsite. The 3620 cm^{-1} band overlaps with that of the octahedral OH band of kaolinite in the Hayesville clay fraction.

Experiment No. 9

Objective: Identification of different rocks

Rock: In geology, naturally occurring and coherent aggregate of one or more minerals. Rocks are commonly divided into three major classes according to the processes that resulted in their formation. These classes are igneous rocks, which have solidified from molten material called magma. **Sedimentary rocks:** those consisting of fragments derived from preexisting rocks or of materials precipitated from solutions; and **metamorphic rocks,** which have been derived from either igneous or sedimentary rocks under conditions that caused changes in mineralogical composition, texture and internal structure.

1. Basalt

Rock type : Igneous (extrusive/volcanic)

Composition: It consists of feldspar, olivine, pyroxene and amphibole.

Environment: Basalt is solidified lava, like rhyolite which flows much quicker because it is less viscous.

Characteristics: Red-brown to black color, frothy with small visible holes where gas escaped while the lava cooled



2. Conglomerate

Rock type: Sedimentary

Composition: It is fragments of other rocks and minerals which are cemented by silica, calcite or iron oxide.

Environment: The rock fragments can be rounded from being rolled along a stream bed or a beach during transportation. While the matrix is angular instead of rounded, the rock is called a breccia.

Characteristics: Dark grey color with imbedded fragments



3. Dolostone

Rock type : Sedimentary

Composition: It is mainly composed with dolomite and fossils. Metamorphoses.

Environment: It formed under magnesium rich conditions, in limestone calcium is replaced by magnesium ions which form limestone to dolostone. The hard parts (skeleton, teeth and shell) and sometimes tissue (Leaves, flowers, muscle and cartilage) may be deposited/sediments to become rock.

Characteristics: Grey color with fossils that are visible



4. Gabbro

Rock type :Igneous (intrusive/plutonic)

Composition:feldspar, olivine, pyroxene and amphiboleetc

Environment:Gabbro is formed by magma that cools very slowly into hard rock below or within the Earth's crust

Characteristics:Dark grey-black and shiny surfaces of feldspar are visible.



5. Gneiss

Rock type :Metamorphic

Composition:quartz, feldspar and mica

Environment:Gneiss forms at high temperatures and pressures. The temperature needed is about 700°C and the pressure needs to be about 12-15 kilo bars at a depth of about 40 km.

Characteristics:It banded with alternating layers of dark and light minerals.



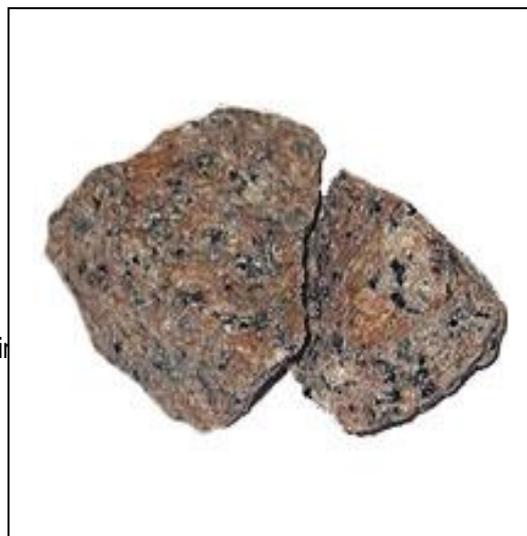
6. Granite

Rock type :Igneous (intrusive/plutonic)

Composition:It composed with having feldspar, quartz, mica and hornblend

Environment:Granite is formed by magma that cools very slowly into hard rock below or within the Earth's crust.

Characteristics:Pink color appear in crystal feldspar, white or grey quartz and black mica. There is no horizontal banding



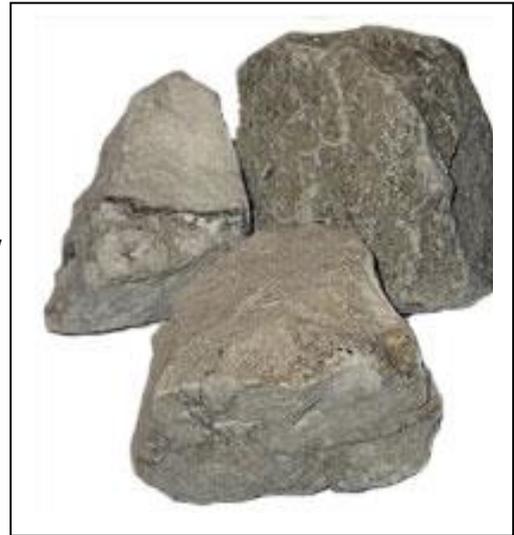
7. Limestone

Rock type: Sedimentary

Composition: Mostly calcite

Environment: The calcite comes out of solution and is deposited on the sea floor as a chemical precipitate. The precipitates can build up along with other sediments or on their own and eventually form limestone. Another way for limestone to form is by the buildup of the shells and skeletons of marine animals

Characteristics: Whitish-grey with a chalky texture. There are no visible fossils in these samples.



8. Marble

Rock type: Metamorphic

Composition: Very pure re-crystallized form of calcite.

Environment: Marble forms at many temperatures and pressures

Characteristics: Medium to coarser grained, light colored and calcite crystals may be visible in the rock.



9. Quartzite

Rock type: Metamorphic

Composition: recrystallized quartz grains Original Rock: sandstone

Environment: Quartzite forms at many temperatures and pressures

Characteristics: Light grey or white in color, medium grained and very hard in nature



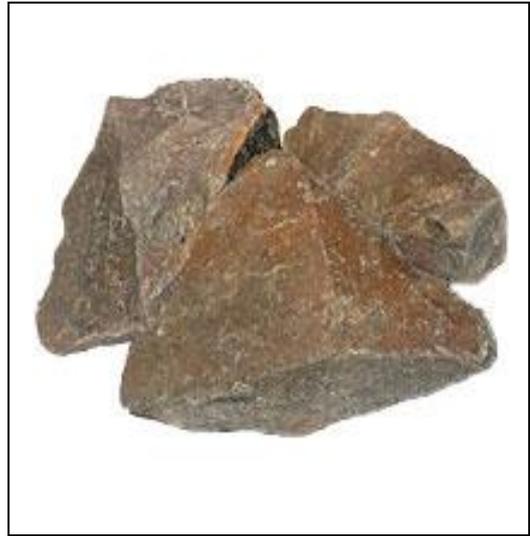
10. Rhyolite

Rock type: Igneous (extrusive/volcanic)

Composition: It consists of feldspar, quartz, mica and hornblende

Environment: It is formed by magma that has reached the Earth's surface (lava) and therefore cools very quickly.

Characteristics: Very fine grained, pinkish-grey, sometimes with dark streaks. If dipped in water and rubbed on a piece of paper, rhyolite will likely tear the paper rather than leave a muddy streak.



11. Sandstone

Rock type: Sedimentary

Composition: It contains feldspar, quartz and other minerals such as mica are also found but it depends on how much weathering has occurred.

Characteristics: Coarse to very fine grains, grey colour which feels like sandpaper.



12. Shale

Rock type: Sedimentary

Composition: It is mainly composed of grains of clay

Environment: Shale sediments are deposited in a lake or a deep, slow flowing river

Characteristics: It appears dull, reddish-brown, very fine grains (smooth to the touch), breaks easily. If an edge is dipped in water and drawn along a surface, shale will leave a muddy streak



13. Slate

Rock type:Sedimentary

Composition:clay minerals

Environment:Slate forms from the heat and pressure when shale is buried deep in the crust. The depth of burial is about 10 km and temperature at depth is about 200°C.

Characteristics:Dark grey to black color, very fine grains (smooth to the touch) ,harder than shale and distinct layers are visible.



Experiment No. 10

Objective: To study about the identification criteria of different minerals.

Some physical properties which is used for mineral identification are

1. Color

In nature, the color of minerals is variable; a mineral may have more than one color depending upon its chemical composition. The colors of a few important rock-forming minerals are:

SN	Mineral	Color
1.	Quartz	Color less
2.	Feldspars, calcite, dolomite, gypsum, kaolinite, muscovite	White to pale
3.	Iron pyrite (called "Fools Gold")	Yellow / golden
4.	Olivine , serpentine, hornblende	Greenish
5.	Garnet	Reddish - brown
6	Biotite, augite, haematite, magnetite, graphite	Black
7.	Orthoclase	Pink or flesh colored

2. Streak

Streak is fine powder of the mineral which representing its true color and greater reliability. Streak is produced and determined by rubbing the specimen on a piece of unglazed porcelain plate i.e. called streak plate.

3. Striation

The parallel thread-like lines or narrow bands running across the surfaces of mineral are called striations. These are reflections of the internal arrangement of atoms in to the crystals. These are clearly observed on crystals of quartz, feldspars and pyrite

4. Hardness

The resistance of a mineral to scratching is known as hardness. The hardness is expressed in Mho's scale and indicated by numerals (1 to 10).

Hardness (Mho's scale)	Mineral Substance	Test
1	Talc	Scratches by a finger nail
2	Gypsum	Just scratches by a finger nail
3	Calcite	Scratches not easily by a copper coin piece
4	Fluorite	Scratches not easily by a steel knife
5	Apatite	Just scratches by a knife
6	Feldspars (orthoclase)	Scratches soft glass
7	Quartz	Scratches glass easily

8	Topaz	Scratches glass but not hard enough to be used as grinding material
9	Corundum	Very hard and used as grinding material for all minerals
10	Diamond	The hardest mineral known

5. Lustre

Lustre is the general appearance of a mineral in reflected light. For example iron minerals have metallic lustre, clay minerals have dull lustre, micas have a shining lustre and quartz have a vitreous lustre.

6. Transparency

It is the degree of penetration of light through a mineral.

Transparent - mica,

Translucent - quartz

Opaque- pyrite and magnetite.

7. Specific gravity

It is the ratio between the weight of a mineral or a substance to the weight of an equal volume of water.

- Heavy minerals: Mineral have specific gravity > 2.85, eg- pyroxene , amphiboles, garnet, zircon etc
- Light minerals have specific gravity < 2.85, eg quartz, micas, muscovite, feldspars etc.

8. Tenacity

The mineral resistance to breaking, crushing or bending is called tenacity. Minerals may be brittle, malleable and flexible or elastic.

9. Cleavage and fracture

The tendency of a mineral to split in certain preferred directions along smooth plane surfaces is called cleavage. The cleavage planes are governed by the internal arrangement of atoms and the direction in which the atomic bonds are relatively weak such as mica and feldspars.

Fracture, on the other hand is the property of a mineral to break along an irregular surface which is not connected with the crystalline form such as in glass and quartz

10. State of aggregation and crystal form

Almost all the rock-forming minerals have got crystalline structure. The specific atomic arrangement of the minerals (as in a crystal) is called its crystal form. A crystal may be defined as a substance with regular geometric faces and definite structure. The angle between the faces is always constant for a particular crystal.

Objective: To study about the description of soil profile

The vertical section of the soil from the surface extending in to the parent material is called soil profile. The various layers, composing the soil, are called horizons.

Materials required

Digging tools - Spade, pickaxe, khurpi, Cutting knife with a strong wooden handle, Abney's level or inclinometer, Altimeter, Measuring scale / tape, Munsell's colour chart, Dilute hydrochloric acid (10%), Wash bottle (with distilled water), Magnifying lens, Indicator papers, Tray with shoulder, Profile description papers, Soil sample bags with polythene lining, Copying pencil/ ball pen

Write the identification procedure of soil profile.

1.1 Selection of area for profile study

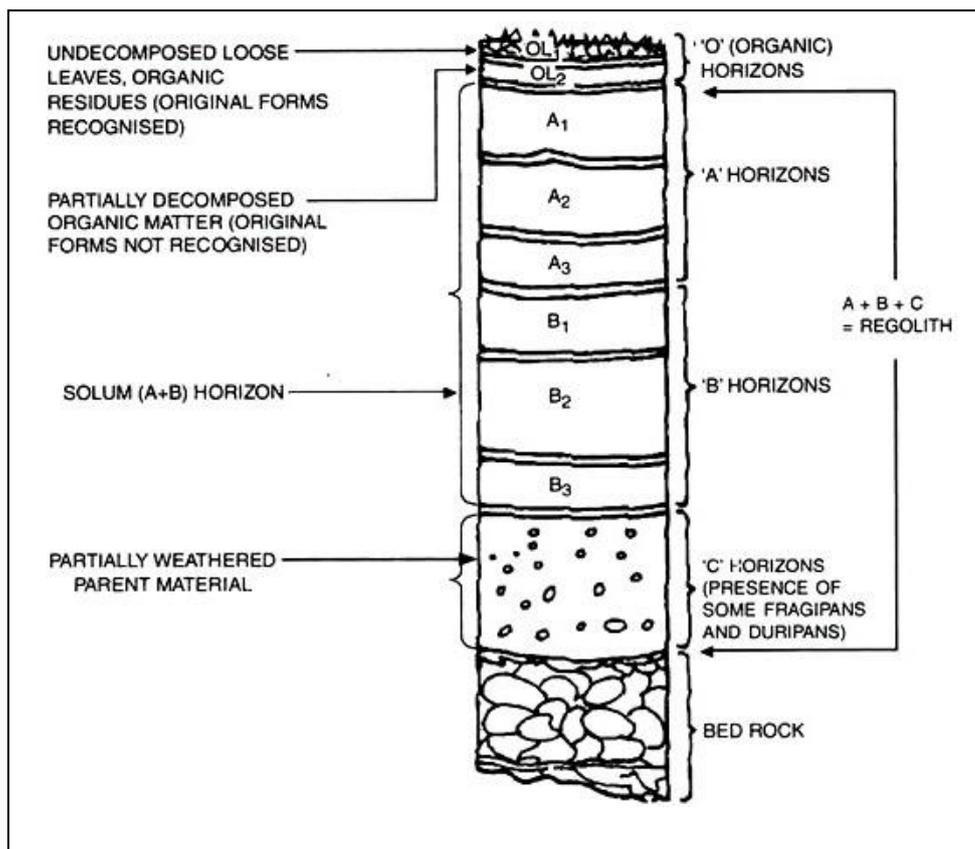
- It is representative of the normal conditions prevailing in the area.
- It is away from tree an irrigation channel/ditch/rivers, roads, human inhabitation etc.
- It shows minimum of human interference, if, possible virgin area should be selected

1.2 Digging of the profile

Dig out a pit of size 2m x 2m to a depth of 2 m or upto the parent material. Provide steps, if necessary, on one side for getting into the pit.

1.3 Examine and description of soil profile

- Identify the horizons of the profile on that side of the pit which receives maximum daylight.
- Demarcate the horizons on the basis of characteristics that can be seen or measured in the field such as color, texture, structure, consistence, presence of carbonates etc.
- If the horizons are not properly differentiated as in case of alluvial and black soils, examination of soil should be done at a depth intervals of 15 cm down to 1 m depth and thereafter, at intervals of 15 cm up to 2m or up to the parent material, whichever occurs earlier.
- Location of the profile: Give longitude and latitude with distance and direction from a nearby bench-mark reference site.
- Parent material e.g. alluvium, mica schist, granite etc.
- Physiographic position of the site e.g. terrace, flood plain, depression, plateau, valley bottom etc.
- Land form of surrounding area and elevation (in meters above the mean sea level)
- Hydrology, drainage condition, depth of ground water, moisture condition in the profile and artificial drainage.
- Biological activity, root development etc
- Evidence of erosion and its intensity, presence of salts or alkali
- Human influence like tillage, levelling, use of amendments, heavy manuring etc.
- Presence of surface stones or rock out crop



Source: Introductory Soil Science, Dilip Kumar Das (Scanned)

2. Observations to be recorded during profile study

Horizon symbol, Depth of from the top to bottom of a particular horizon in centimeters, Nature of boundary with the horizon below, Color : Moist and dry –Munsell colour chart, Colour of mottling (if any), Texture: Feel method which will be explained in the field, Structure: Three feature of structure viz. grade, class and type, Consistence : wet, moist and dry, Cutans(ped coatings), pressure faces, slickensides , Roots traces : The quantity, size and location of roots in each horizon, Nodule concretions and cementation (if any), Pores Lithorelics: The content of rock and mineral fragments in each horizon, note down their percentage, Hard pans, Content of carbonates, soluble salts etc, Artefacts : The activity of man's activity, Soil reaction

3. What should be precaution taken during profile study

- Expose the profile at such a place that it is representative of the whole area.
- Do not expose it at elevated or depressed locations in field or in a direction directly facing the sun.
- Dig the profile at such a place that maximum visibility is obtained.
- Remove the plant cover if any before digging the pit in a manner so that the soil surface is not disturbed.
- Mark the soil sample bags properly indicating location, depth, horizon details, etc.
- Dig the profile pit in such a way that one side of it has steps for movement of the personnel.

Experiment No. 12

Objective: Determination of soil color by using munsellcolor chart.

The color of soil is due to organic matter, mineral matter and mixture of these two. Munsell color chart is used for identifying color of the soil. This chart is a note book having cards of different color. Each card has chips of different color alongwith a hole below each chip. The three variable of a color i.e. Hue, value and chroma are indicated on each card. Hue indicates main color of spectrum and main wavelength of light. Value indicates relative blackness or whiteness of color. Chroma indicates relative purity of color.

Materials required:

Munsell soil color chart , Water bottle, Tube or post hole auger, Small clods of soil

Write the procedure for determination of soil color

- Take a small clod of soil and note its moisture condition, weather it is dry or moist.
- Match the color of the clods with the cards showing 'Hue' values.
- Note the 'Hue'.
- Place the clod below the hole of chip and match the nearest 'Value' and 'Chroma'.
- Note the Munsell notation.
- Write the color of the soil according to munsell notation.
- Find out the color of soil at both dry and moist condition. If soil is dry first note the color of dry soil. Then moist the clod with few drops of water and note the color

1. Observations

Sample No.	Dry soil					Moist soil				
	Munsell notation					Munsell notation				
	Hue	Value	Chroma	Comb-ination	Color name	Hue	Value	Chroma	Comb-ination	Color name
1.										
2.										

2. Result

1. Sample number =
2. Color of dry soil =
3. Name of the dry soil color =
4. Color of moist soil =
5. Name of the moist soil color =

.....

Objective: To study about the different chemical weathering indices for soil development

Chemical weathering indices, sometimes referred to as indices of alteration, are commonly used for characterizing weathering profiles. Chemical weathering indices incorporate bulk major element oxide chemistry into a single value for each sample. Weathering indices are typically applied by plotting some specific index versus depth in the weathering profile, providing a visual representation of changes in bulk chemistry with presumed increasing (or decreasing) weathering of the parent rock.

A useful chemical weathering index should provide values that do more than simply vary relative to one another on a given weathering profile. Ideally, a chemical weathering index should permit comparison between studies performed at different localities on different parent materials and on weathering profiles of different ages. The following criteria in evaluating the utility of weathering indices.

- A chemical weathering index should be easy to use, involving chemical elements common in soil analyses. This is desirable for wide applicability and maximum comparability with other studies.
- A weathering index should incorporate elements with a range of mobility in the weathering environment. Weathering indices based exclusively on the most mobile elements may not be applicable to assessing profiles or intervals representing advanced stages of weathering, because these are likely to have been depleted in highly mobile elements.
- A useful weathering index should exhibit chemically appropriate trends with increased weathering (e.g., varying monotonically with depth) and should change greatly with increasing weathering. This is especially important in weathering profiles developed on metasedimentary parent rocks, which are commonly compositionally layered at scales much smaller than the overall dimensions of the weathering profile.
- A useful weathering index should be applicable to a wide range of rocks and rock types, and should yield values for the unweathered parent material that are distinct from values for weathered material, regardless of rock type. Metamorphosed sedimentary rocks might be expected to exhibit “weathered” chemical signatures by virtue of their pre-metamorphic history of weathering, differential erosion, and “chemical maturation” during transport in the previous pass of their protoliths through the sedimentary cycle. A useful weathering index should allow users to distinguish between a “chemically mature” metasedimentary parent material and material post-metamorphically weathered from that parent material.
- Ideally, a chemical weathering index should not assume that any element is immobile. This is difficult to apply in practice. Any element may be mobile in a weathering environment. At best, those elements that are least mobile should be used.

The following weathering indices used for the development of soil profile

1. **Ruxton Ratio:** Ruxton (1968) proposed a simple weathering index, which has been termed the Ruxton Ratio by Chittleborough (1991). Ruxton (1968) stated that his simple weathering index is best suited for weathering profiles developed on uniform acid to intermediate bedrock, with constant sesquioxide content during weathering, and which produces kaolin and/or allophane weathering products (products which have mole ratios of $\text{SiO}_2/\text{Al}_2\text{O}_3$ of 2:1). The Ruxton Ratio relates silica loss to total-element loss and considers alumina (and other sesquioxides) to be

immobile during weathering. Ruxton (1968) tested R on weathering profiles developed on igneous and metamorphic rocks from humid regions around the world, and found that R correlated well with total-element loss.

2. **Weathering Index:** Parker (1970) introduced the weathering index, termed the Parker Index, or the Weathering Index of Parker (WIP) for silicate rocks. Parker's Weathering Index is based on the proportions of alkali and alkaline earth metals (sodium, potassium, magnesium and calcium) present. These elements are the most mobile of the major elements, and there is no need to assume that sesquioxide concentration remains approximately constant during weathering. The WIP also takes into account the individual mobilities of sodium, potassium, magnesium and calcium, based on their bond strengths with oxygen. Parker (1970) applied the WIP to acid, intermediate and basic igneous rocks. However, because the WIP relies only on the mobile alkali and alkaline earth, its application to highly weathered materials (i.e., sedimentary ferricretes and bauxites) is uncertain.
3. **Vogt's Residual Index:** Vogt (1927) proposed a geochemical method for assessing the maturity of residual sediments; this index has been termed Vogt's Residual Index. Roaldset (1972) used this index to determine the extent to which clays have been weathered in Quaternary deposits of the Numedal Area, Norway. He used V to compare the bulk chemistry of moraine and marine clay deposits and concluded that the moraine clays were more weathered than the marine clays, suggesting that weathering ceased following deposition in a marine basin.
4. **Chemical Index of Alteration:** Nesbitt and Young (1982) developed the Chemical Index of Alteration (CIA) in reconstructing paleoclimate from Early Proterozoic sediments of the Huronian Supergroup, north of Lake Huron. Nesbitt and Young (1982) used major element analyses of lutites to calculate their CIA. Their results suggested that the depositional basin of the lutites migrated through various climate zones, from approximately 32 °latitude to approximately 60° latitude. These conclusions were confirmed with paleomagnetic studies. Because the CIA is interpreted as a measure of the extent of conversion of feldspars (which dominate the upper crust) to clays such as kaolinite.
5. **Chemical Index of Weathering:** Harnois (1988) developed the Chemical Index of Weathering (CIW). This index is identical to the CIA, except that it eliminates K₂O from the equation. Because the CIW does not account for the aluminum associated with K- feldspar, it may yield very high values for K-feldspar- rich rocks, whether they are chemically weathered or not. Like the CIA, the CIW is also essentially a measure of the extent of conversion of feldspars to clays.
6. **Plagioclase Index of Alteration:** Fedo et al. (1995) proposed the Plagioclase Index of Alteration (PIA) as an alternative to the CIW. Because plagioclase is abundant in silicate rocks and dissolves relatively rapidly, the PIA may be used when plagioclase weathering needs to be monitored.
7. **Silica– Titania Index:** The Silica– Titania Index (STI) was developed to correlate the degree of chemical weathering of metamorphic silicate rocks in tropical regions with their engineering properties (e.g., point load strength and uniaxial compressive strength). For fresh rock and weathering profiles developed on high grade gneisses and quartzite of Sri Lanka.

Summary of weathering indices (if calculated using molecular proportions of elements oxides) evaluated in this study

Index	Formula	Optimumfreshvalue	Optimumweatheredvalue	Idealtrendofindexup-profile (increaseinweathering)	Allows Almobility
R	$\text{SiO}_2/\text{Al}_2\text{O}_3$	>10	0	Negative	No
WIP	$(100)[(2\text{Na}_2\text{O}/0.35) + (\text{MgO}/0.9) + (2\text{K}_2\text{O}/0.25) + (\text{CaO}/0.7)]$	>100	0	Negative	Yes
V	$(\text{Al}_2\text{O}_3 + \text{K}_2\text{O}) / (\text{MgO} + \text{CaO} + \text{Na}_2\text{O})$	<1	Infinite	Positive	No
CIA	$(100)[\text{Al}_2\text{O}_3 / (\text{Al}_2\text{O}_3 + \text{CaO} + \text{Na}_2\text{O} + \text{K}_2\text{O})]$	V50	100	Positive	No
CIW	$(100)[\text{Al}_2\text{O}_3 / (\text{Al}_2\text{O}_3 + \text{CaO} + \text{Na}_2\text{O})]$	V50	100	Positive	No
PIA	$(100)[(\text{Al}_2\text{O}_3 - \text{K}_2\text{O}) / (\text{Al}_2\text{O}_3 + \text{CaO} + \text{Na}_2\text{O} - \text{K}_2\text{O})]$	V50	100	Positive	No
STI	$(100)[(\text{SiO}_2/\text{TiO}_2) / ((\text{SiO}_2/\text{TiO}_2) + (\text{SiO}_2/\text{Al}_2\text{O}_3) + (\text{Al}_2\text{O}_3/\text{TiO}_2))]$	>90	0	Negative	No