Simultaneous Measurement of Bulk Density and Water Content by Nuclear Methods

Recent advances is nuclear technology which have evolved methods for the non destructive determination of the soil water content. Two methods have been used with success. One the neutran scattering method and the other is the y-radiation method.

Neutron method : In this method measurement is made of the number of hydrogen nuclear that are present per unit volume of soil and therefore, water content by volume θ, is measured. Neutrons are uncharged particles having almost the same mass as that of protons or that of hydrogen nuclei. A radium-beryllium mixture in form of pellets is used as source of fast neutrons. The measurement of soil water by the neutron method is essentially a measure of the density of the slowed neutron cloud developing when Ra-Ba source is inserted in the soil, the density of the slowed neutrons being a measure of the soil water content by volume θ.

Simultaneous determination of bulk density and water content :

With the increased interest in the behaviour of water in swelling soils and resulting theoretical studies (Smile and Rosenthal, 1968; Philip, 1969) on the subject, a method was required for measuring changes in both the water content and soil density in order to experimentally evaluate these theoretical analyses. Numerous authors have proposed measuring the attenuation of gamma rays of two different energies to non destructively determine both water content and soil density in the same sample. Soane (1967) illustrated the effectiveness of the dual gamma method for measuring bulk density and water content of three soils. The samples were placed between a cesium source and detector and then between americium to using a combined source but neither Soanenor other workers (Gardner and Calissendoff, 1967; Gardner, Campbell and Calissendaff, 1969) have attempted to combine the sources in a single collimator because the higher energy gamma photons produce Compton scatter through interaction with the sample and some of this Compton scatter will be counted as gamma rays from the lower energy source. The error can be eliminated by with equipment and method evolved by Corey, Peterson and Wakat, 1971). This method is feasible for measuring the water content and soil density of soil columns simultaneously when two sources are combined in a single collimator. Measurement of attenuation of ^{137}cesium and ^{241}americium is done in this method. Radiation intensity (counts/min) after passage through the soil, container and container alone is calculated by the equation (Corey *et al.*) given in method. To obtain values of intensity the equipment is required as shown in Fig. 1.

^{241}Am emits a large number of gamma rays of various energies but the major energy is 59.6 KeV ^{241}Am has a half life of 458 years eliminating the need for decay corrections ^{137}CS decays with a gamma ray of 662 KeV and has a half life of 30 years.

Detector and pulse height analyzers are used in this system.

Correction for comption scatter

Two methods were compared and dual source method evaluated. Known water content and soil densities were measured. The two soils were Houston black, a soil containing monitorillonitic clay and cecil a soil containing kaolinitic clay. The soil containing different amount of water was packed to varying densities into plastic boxes 7.5 x 4.46 x 4.95 cm. These boxes were placed between sources and detector with long axis parallel to beam. Comparisons between the known, soil density.

Water content and values determined by two methods for four cecil soil samples and five Housten Black soil samples are given in table 1.

Table 1: Water content and values determined by twi methods.

Water content g/cm^3			Soil density g/cm^3		
Known	Calculated		Known	Calculated	
Cecil soil					
0.001	0.000	0.00	1.831	1.830	1.830
0.044	0.081	0.073	1.712	1.711	1.720
0.120	0.124	0.143	1.999	2.001	1.980
0.175	0.118	0.151	2.050	2.067	2.081
Houston Black Soil					
0.010	0.000	0.000	1.504	1.504	1.504
0.141	0.147	0.139	1.297	1.281	1.290
0.270	0.284	0.281	1.343	1.367	1.371
0.438	0.472	0.446	1.369	1.349	1.378
0.495	0.532	0.514	1.297	1.234	1.255

There is little difference between the results obtained by the two correction techniques and method best suited will be determined by the instrumentation available.

Following satisfactory evaluation of the dual source method for determining the known water content and soil density of soils, the method was used to determine water content and soil densities of Houstan Black and Cecil soil columns following infiltration. More water was added to Houston soil because its greater water holding capacity. Twenty four hours following the addition of water the transmission measurements were repeated and the water content and density was calculated water remained ponded on Houston Black soil at the end of 24 hour period but had infiltrated completely with the Cecil soil. The two soils responded quite differently to the addition of water. Neither the soil density nor lengths of Cecil soil column changed. The column of Hoston black soil increased 2 cm in length and the soil density decreased in top 4 cm.

The combined source method is applicable to the study of swelling soils, the phenomena of freezing and thawing and measurement of water content and density of the soil inside core barrels.

References :

Cerey, J.C., Peterson, S.F. and Wakat, M.A. (1971). Measurement of attenuation of ^{137}CS and ^{241}Am gamma rays for soil density and water content determinations. Soil Sci. Soc. Am. Proc. Vol. 35 : 215-19.

Gardner, W.H. and Callissendortt, C. (1967). Gamma rays and neutron attenuation in measurement of soil bulk density and water content. Pp 101-113. In Isotope and radiation techniques in soil physics and irrigation studies. International atomic agency, Vienna.

Gaedner, W.H., Campbell, G.S. and Calissendorff, C. (1969). Water content and soil bulk density measured concurrent using two gamma photon energies US AEC Report, RLO 1543-6 Washington State University. Pullman Wash 42 p.

Philip, J.R. (1969). Moisture equilibrium in the vertical in swelling soil. I Basic theory. Anst. J. Soil Res. 7 : 99-120.

Smiles, D.E. and Rosenthal, M.J. (1968). The movement of water in swelling material. Aust. J. Soil Res. 6 : 237-248.

Soane, B.D. (1967). Dual energy gamma ray transmission for coincident measurement of water content and dry bulk density of soil. Nature 214 : 1273-1274.

Determination of Available (Mineralization) Nitrogen in Soil by Alkaline Permanganate Method

Preparation of soil sample :

The soil samples from definite depth are randomly collected from the field with the help of screw auger. All the possible technical precautions as prescribed for standard soil sampling are also taken. Samples are then brought to the laboratory, air-dried in the shade and grounded by wooden roller, thereafter sieved through 2 mm stainless steel sieve and stored in polythene bags and used for chemical assay.

Reactions involved:

I. Distillation :

$$2\ KMnO_4 + H_2O \xrightarrow[\text{medium}]{\text{Alkaline}} 2\ MnO_4 + 2KOH + 3O^-$$

(Nascent oxygen)

$$R.CHNH_2COOH + O^- \xrightarrow{\text{Oxidation}} R.CO.COOH + NH_3$$

Organic- N fraction (Ammonia)

$$NH_3 + H_2O \xrightarrow{\text{Distillation}} NH_4OH$$

$$3NH_4OH + H_3BO_3 \xrightarrow{\text{Absorption}} (NH_4)_3BO_3 + 3H_2O$$

(Green colour)

II. Titration

$$2(NH_4)_3BO_3 + 3H_2SO_4 \longrightarrow 2(NH_4)_3SO_4 + 2\ H_3BO_3$$

(Pink colour and original)

Principle :

A known weight of the soil is mixed with alkaline potassium permanganate ($KMnO_4$) solution and distilled. The organic matter present in soil is oxidized by the nascent oxygen, liberated by potassium permanganate, in the presence of sodium hydroxide and the released ammonia is condensed and absorbed in known volume of a boric acid with mix indicator to form ammonium borate, the excess of which is titrated with a standard sulphuric acid.

Equipment and apparatus :

1. **KEL PLUS Automatic Nitrogen Estimation System**

The said instrument is used for determination of available nitrogen in soil. It consists of the following:

- **Automatic Distillation System (Model Classic DX):** It is fully automatic distillation system with programmable auto run digital features, with automatic dilution and addition of boric acid, NaOH and $KMnO_4$. Both modes (auto and manual) are available for distillation reagents addition.

- **Refrigerated Water Cooling System for Condenser (Model Kel Freeze):** It is refrigerated water cooling system for distillation and condensing system with inbuilt compressor and recirculator pump.

2. Electronic balance
3. Burette
4. Conical flask
5. Distilled water

Reagents :

1. 0.32 % potassium permanganate ($KMnO_4$) solution.
2. 2.5 % sodium hydroxide (NaOH).
3. 2 % boric acid solution containing 20 - 25 ml of mixed indicator / liter.
4. Mixed indicator: 0.066g methyl red + 0.099g bromocresol green dissolve in 100 ml of 95 % alcohol.
5. 0.02 N sulphuric acid (H_2SO_4).

Procedure :

- Weigh 5 g of prepared soil sample and transfer it to the digestion tube.
- Load the tube in distillation unit and other sides of hose keep 20 ml of 2 % boric acid with mixed indicator in 250 ml conical flask.
- 25 ml each of potassium permanganate (0.32 %) and sodium hydroxide (2.5 %) solution is automatically added by distillation unit programme.
- The sample is heated by passing steam at a steady rate and the liberated ammonia absorbed in 20 ml of 2 % boric acid containing mixed indicator solution kept in a 250 ml conical flask.
- With the absorption of ammonia, the pinkish colour turns to green.
- Nearly 150 ml of distillate is collected in about 10 minutes.
- The green colour distillate is titrating with 0.02N sulphuric acid and the colour changes to original shade (pinkish color).
- Simultaneously, blank sample (without soil) is to be run.
- Note the blank & sample titer reading (ml) and calculate the available nitrogen in soil.

Calculations :

$$\text{Available N (kg ha}^{-1}\text{)} = \frac{\text{R (Titer reading - Blank reading)} \times \text{Normality of acid} \times \text{Atomic weight of nitrogen} \times \text{Weight of one hectare of soil}}{\text{Sample weight (g)} \times 1000}$$

$$= \frac{R \times 0.02 \times 14 \times 2.24 \times 10^6}{5 \times 1000}$$

Factor = R x 125.44

Interpretation of results :

Available N (kg ha^{-1})		**Soil rating**
< 280	:	Low
280-560	:	Medium
> 560	:	High

References :

Subbiah, B.V. and Asija, G. L. (1956). A rapid procedure for the estimation of nitrogen in soils. *Curr. Sci.*, **25**: 259-260.

Total nitrogen is estimated by the micro-Kjeldahl method as per procedure suggested by AOAC (1995).

Preparation of plant and soil samples :

The plant analysis has been considered as a superior diagnostic technique for mineral content. Whole plant is dried in open air for few days after that it was further dried in hot air oven at about 60 ± 2^{o} C for eight to ten hours per day to achieve complete drying. After drying, whole plant is powdered with the help of a grinder, passed through 2 mm stainless steel sieve and used for chemical assay. The soil sample from definite depth was randomly collected from the field with the help of screw auger. All the possible technical precautions as prescribed for standard soil sampling were also taken. Samples were brought to the laboratory, air-dried in the shade and grounded by wooden roller, thereafter sieved through 2 mm stainless steel sieve and stored in polythene bags and used for chemical assay.

Principle :

Nitrogen in samples like plant and soil exists in a very complicated bonding structure. During digestion, a known weight of the plant/soil samples in the presence of sulphuric acid with catalyst mixture under high temperature is digested where complicated structures are broken to simple structure, thereby releasing nitrogen in the form of ammonium radical (NH_4^+). During distillation in presence of sodium hydroxide, the released ammonia is condensed and absorbed in known volume of a boric acid with mix indicator to form ammonium borate, the excess of which is titrated with a standard sulphuric acid.

The micro-Kjeldahl method consists of the three steps;

1. Digestion
2. Distillation and
3. Titration.

Equipment and apparatus :

1. **KEL PLUS Automatic Nitrogen Estimation System :**

The said instrument is used for determination of nitrogen. It consists of the following;

- **Macro Block Digestion System (Model KES 12L):** This digestion system is suitable for soil, plant, water, pesticides, fertilizers, food and feed samples. It is microprocessor based automatic twelve place macro block digestion system with temperature controller fitted with sensor break protection (Microprocessor based) feature and temperature range from 50-450 ^{0}C.
- **Acid Neutralizer Scrubber (Model KEL VAC):** It is used to neutralize the acid fumes, which are absorbed in 15% sodium hydroxide and dissolved in water stored in the system tank. After every 2 cycles of digestion, the 15% sodium hydroxide solution is replaced and after 3 cycles of digestion, acid fumes dissolved in water tank is drained off and refilled with fresh water in the system tank.
- **Automatic Distillation System (Model Classic DX):** It is fully automatic distillation system with programmable auto run digital features, with automatic dilution and addition of boric acid and NaOH. Both modes (auto and manual) are available for distillation reagents addition.
- **Refrigerated Water Cooling System for Condenser (Model Kel Freeze):** It is refrigerated water cooling system for distillation and condensing system with inbuilt compressor and recirculator pump.

2. Electronic balance

3. Burette
4. Pipette
5. Conical flask
6. Measuring cylinder
7. Distilled water

Reagents :

1. Concentrated sulphuric acid (H_2SO_4).
2. Catalyst mixture: Mix with 250 g potassium sulphate (K_2SO_4), 50 g cupric sulphate ($CuSO_4$. 5 H_2O) and 5 g metallic selenium powder in the ratio of 50:10:1.
3. 40 % sodium hydroxide (NaOH).
4. 4 % boric acid containing 20 - 25 ml mixed indicator /liter.
5. Mixed indicator: 0.066 g methyl red + 0.099 g bromocresol green dissolve in 100 ml of 95 % alcohol.
6. 0.02N sulphuric acid (H_2SO_4).

Procedure :

I. Digestion :

- Weigh 0.5 g of prepared plant sample or 1 g of soil sample and transfer it to the digestion tube.
- Add 10 ml of concentrated sulphuric acid and 5 g of catalyst mixture to the sample.
- Load the digestion tubes in to the digester and heat the digestion block.
- Switch on the digestion unit and set the initial temperature 100 ^{0}C till frothing is over.
- Then block temperature is raised to 400 ^{0}C. The effective digestion starts only at 360 ^{0}C and beyond 410 ^{0}C.
- The sample turns light green colour or colorless at the end of the digestion process.

II. Distillation :

- After cooling the digestion tube, load the tube in distillation unit and other side of hose keep 20 ml of 4 % boric acid with mixed indicator in 250 ml conical flask.
- 40 ml NaOH (40 %) is automatically added by distillation unit programme.
- The digested sample is heated by passing steam at a steady rate and the liberated ammonia absorbed in 20 ml of 4 % boric acid containing mixed indicator solution kept in a 250 ml conical flask.
- With the absorption of ammonia, the pinkish colour turns to green.
- Nearly 150 ml of distillate is collected in about 8 minutes.
- Simultaneously, blank sample (without plant/soil) is to be run.

III. Titration :

- The green colour distillate is titrating with 0.02N sulphuric acid and the colour changes to original shade (pinkish color).
- Note the blank & sample titer reading (ml) and calculate the total nitrogen content present in plant/soil samples.

Calculations :

$$\textbf{Nitrogen content in plant (\%)} = \frac{\textbf{R}\text{ (sample titer-blank titer) x Normality of acid x Atomic weight of nitrogen x 100}}{\text{Sample weight (g) x 1000}}$$

$$= \frac{\text{R x 0.1 x 14 x 100}}{\text{0.5 x 1000}}$$

Factor = R x 0.28

$$\textbf{Nitrogen content in soil (\%)} = \frac{\textbf{R}\text{ (sample titer-blank titer) x Normality of acid x Atomic weight of nitrogen x 100}}{\text{Sample weight (g) x 1000}}$$

$$= \frac{\text{R x 0.1 x 14 x 100}}{\text{1 x 1000}}$$

Factor = R x 0.14

Crude protein content :

The total nitrogen is estimated by micro-Kjeldahl method as per procedure suggested by AOAC (1995) and the crude protein is calculated by the following formula:

Crude protein content (%) = micro-Kjeldahl nitrogen content (%) x 6.25 (based on the assumptions that nitrogen constitutes 16 % of protein).

References :

AOAC, (1995). *Official Methods of Analysis*. 16th edn. Association of Official Analytical Chemists, Washington, DC.

The phosphorus is an essential plant nutrient and it occurs in many different forms. Therefore, a reliable procedure for measuring the amount both in soil as well as in plant is needed. There are many methods available for the determination, however, colorimetric measurement is presented here:

Principle :

Phosphorus is extracted from the soil with 0.5 m $NaHCO_3$ at a nearly constant pH of 8.5. The phosphate ion in solution treated with ascorbic acid in an acidic medium provides a blue colour complex. Measurement of the quantitative determination of phosphorous in soil (Olsen's *et al*., 1954)

Reagents :

1. **0.5 M Sodium bicarbonate ($NaHCO_3$) solution:** Dissolve 42 g of $NaHCO_3$ in distilled water to get one litre solution and adjust the pH of the solution to 8.5 by small quantity of NaOH.
2. **Activated Charcoal:** Darco G-60 (P-Free)
3. **5 N Sulphuric acid (H_2SO_4) Solution:** Add 141 ml of con. H_2SO_4 to 800 ml of distilled water. Cool the solution and dilute to one litre with distilled water.
4. **Reagent A:**
   - Dissolve 12.00 g of ammonium paramolybdate in 250 ml of distilled water.
   - Dissolve 0.2908 g of potassium antimony tartrate ($KSbO.C_4H_4O_6$) in 100 ml distilled water.
   - Above both solution mix thoroughly and made one litre in volumetric flask with the help of distilled water.
   - Add these dissolved reagents to one litre of 5N H_2SO_4.
5. **Ascorbic acid working solution (Reagent B):** Dissolve 1.056 g of ascorbic acid in 200 ml of **reagent A** and mix. This ascorbic acid (**reagent B**) should be prepared as required because it does not keep more than 24 hours.
6. **Standard phosphate solution**: Weigh 0.4393 g of potassium dihydrogen phosphate (KH_2PO_4) into one litre volumetric flask. Add 500 ml of distilled water and shake the contents until the salt dissolves. Dilute the solution to one litre with distilled water to get 100 ppm P solution. Dilute 20 ml of 100 ppm P solution to one litre to get form-working solution of 2 ppm.

Preparation of standard curve :

- Take different concentration of P (0, 1, 2, 3, 4, 5, etc ml of 2 ppm standard P Solution) in 25 ml volumetric flasks.
- Add 5 ml of the 0.5M $NaHCO_3$ extracting solution to each flask, and acidify with 5N H_2SO_4 drop by drop.
- Add about 10 ml distilled water and 4 ml of reagent 'B', then shake the solution.
- Make the volume 25 ml by distilled water.
- The intensity of blue colour is read on spectrophotometer at 660 nm wavelengths after 10 minutes.

- Plot the curve by taking P concentration on X axis and colorimeter reading on Y axis. Repeat the process till you get straight line relationship.
- Calculate the factor i.e. 1 colorimeter reading is equal to how much ppm of phosphorus?

Procedure :

- Take 2.5 g of soil sample in 150 ml conical flask and 0.5 g Darco G-60 activated charcoal.
- Then add 50 ml of 0.5 M $NaHCO_3$ solution and shake the solution for 30 minute in a shaker. Similar processes run for a blank without soil.
- Filter the suspension through the Whatman no. 40 paper.
- Take 5 ml aliquot of the extract in a 25 ml volumetric flask, and acidify with 5N H_2SO_4.
- Add small quantity of distilled water, and then add 4 ml of reagent B.
- The intensity of blue colour is read on spectrophotometer at 660 nm wavelengths after 10 minutes.

Observations :

1. Weight of soil sample : 2.5 g
2. Volume of extractant used : 50 ml
3. Volume of filtrate used : 5 ml
4. Absorbency : R
5. Absorbency from standard curve : A
6. Concentration of P for absorbency A : B ppm

Calculation :

$$\text{Available P (kg ha}^{-1}) = \frac{R \times F \times 50 \times 2.24}{5 \times 2.5}$$

Where, F (factor) = B / A

Limits of available P in soil :

Very low	:	Less than 5 P kg ha^{-1}
Low	:	5-10 P kg ha^{-1}
Medium	:	10-20 P kg ha^{-1}
High	:	20-40 P kg ha^{-1}
Very high	:	More than 40 P kg ha^{-1}

Determination of total phosphorus in plant :

Principle : Vanadate molybdate and orthophosphates react to give a yellow colour complex in acidic medium. The intensity of colour provide the basis of quantitative measurement of total P in plant (Koenig and Johnson, 1942).

Apparatus and reagents :

- Colourimeter/spectrophotometer
- 50 ml volumetric flask
- ammonium molybdate ammonium vanadate (in NHO_3) solution : Dissolve 2.5 g $(NH_4)_6\ Mo_7O_2.4\ 4H_2O$ in 400 ml distilled water. Dissolve 1.25 g of ammonium vanadate in 300 ml boiling water. Add the ammonium vanadate solution to the ammonium molybdate solution and cool to room temperature. Add 250 ml conc. NHO_3 and dilute to 1 lit.
- Phosphate standard solution : Dissolve 0.2195 g KH_2PO_4 and dilute to 12%. This solution contains 50 µg phosphorus/ml.

Procedure :

Preparation of standard curve :

- Transfer 0, 1, 2, 3, 4 and 5 ml of 50 ppm P solution to 50 ml volumetric flasks in order to get 0, 50, 100, 150, 200 and 250 µg P.
- Add 10 ml vanadomolybdate reagent make up the volume and mix the content thoroughly.
- Read the transmittance/absorbance at 420 mµ (blue filter).
- Plot the reading against µg P and calculate the factor (F).

Digestion of plant material :

Take one gram of plant material in digestion flask. Add 10-15 ml of Diacid (3:1: Nitric acid : Perchloric acid) mixture and swirl the content in 150 ml volumetric flask. Place the content on hot plate till the digestion is over. Filter the solution in 100 ml conical flask, wash the residue on filter paper several times with the hot water. Make up the volume with distilled water, store the solution in air tight container.

Estimation :

- Transfer 10 ml dilute in 50 ml volumetric flask.
- Add 10 ml ammonium molybdate vanadate solution shake the content.
- Make up the volume and record the reading as per the procedure under preparation of standard curve.

Calculation :

50 µg = R
1 R = 50/R µg (Factor)

$$\text{Total (\%) P} = \frac{\dfrac{\text{Factor (F)} \times \text{Reading} \times 100 \times 100}{\text{sample}}}{10000 \times 1000 \times 10 \times 1}$$

Reference :

Koenig, R.A. and Johnson, C.R. (1942). Colorimetric determination of biological materials Ind. Eng. Chem. Analyt. Edn. 14 : 155-156.

Olsen, S.R., Cole, C.V., Watanabe, F.S. and Dean, L.A. (1954). Estimation of available phosphorus in soils by extraction with sodium bicarbonate. Circ. U.S. Dept. Agric. 939 : 1-19.

The available potassium i.e. exchangeable and water soluble potassium is determined by extracting soil with neutral normal ammonium acetate solution. The estimation of potassium is carried out by flame photometer.

1. Principle :

The principle underlying this is that a large number of elements when excited in a flame, emit radiation of characteristic wave length. The excitation cause one of the outer electron of neutral atoms to move to an outer orbit of higher energy level or the atoms may be excited sufficiently to loose an electron completely from the attractive force of the nucleus where excited atom return to lower energy level, light at characteristic wave length is emitted. Excited atoms or ions give line radiation at very definite wave length and thus K gives at 404.4 and 767 mμ. The flame photometer employees a relatively low temperature excitation and measures with a photocell the emission intensity which is proportional and to concentration in selected wave length (767 mμ) and for this red filter is used.

2. Apparatus and reagents :

a) Flame photometer with red filter,
b) Pipette, volumetric flasks and conical flask (100 ml).

3. Reagents :

(a) Neutral Normal Ammonium Acetate :

Add 58 ml of glacial acetic acid to about 600 ml H2O and then add 70 ml of concentrated ammonia (sp. gr 0.90) Dilute the solution to one litre. Then adjust pH of solution at 7.0 with the help of ammonia or acetic Acid or this can be prepared by dissolving ammo. Acetate (CH3COONH4) (77.08 eq.wt.) directly in H_2O and volume to be made one litre and then adjust the pH 7.0 .

(b) Standard Potassium Solution :

Dissolve 1.9066 g of dried KCl (AR) in distilled water and dilute to one litre. This is 1000 mg kg-1 K solution. 100 ml of this solution diluted to 1 lit. to make 100 ppm K solution.

4. Preparation of the standard curve :

Take 0, 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 ml of 100 mg kg-1 K solution in different 25 ml volumetric flasks. Make up the volume with 1N NH4OAc Soln. Adjust the flame photometer reading at zero with blank (zero K) solution and at 100 for 40 mg kg-1 K solution. Take the flame photometer readings for every dilution. Plot the standard curve on graph paper by taking K concentration on X axis and flame photometer reading an y axis. This will give a factor (F) of 1 flame photometer reading = 0.4 mg kg-1 K.

5. Procedure :

Take 5g soil in 100 ml conical flask and add 25 ml of 1N NH4OAc Soln. Shake the content for 5 minutes and then filter through Whatman No.1 filter paper. Potassium extract is measured by flame photometer after calibration.

6. Calculation :

$$\text{Available K (kg ha}^{-1}\text{)} = \frac{\text{RxFx25x100x20x1.121}}{5 \times 1000}$$

$$= \text{R x F x 11.217.}$$

Limits of available K in soil :

Very low	:	Less than 200 K kg ha^{-1}
Low	:	200 – 250 K kg ha^{-1}
Medium	:	250 – 400 K kg ha^{-1}
High	:	400 – 600 K kg ha^{-1}
Very high	:	More than 600 k kg ha^{-1}

8. Precaution :

a) These should not be any turbidity or suspended particles in extract, it will chock the capillary feeding tube .
b) The gas and air pressure should be constant.
c) If sample reading goes beyond 100 then dilute the extract.

9. Determination of k in plant sample :

(a) Wet digestion :

Place 1-2g of ground plant sample in 100ml digestion flask. Add 20-25 ml of acid mixture Acid mixture 750 ml conc. HNO_3 + 150 ml conc H_2SO_4 + 300 ml of $HClO_4$ and mix the contents of the flask by swirling well. Heat the flask at a low temp and then slowly increase the flame or temp. of hot plate. Completion of digestion is confirmed when liquid is colorless. After cooling, add 20-25 ml H_2O and filter through whatman No.40 into a 100 ml/250 ml volume flask and make up the volume.

(b) Determination of K :

Take the aliquot and get the reading of K through flame photometer using red filter and calculate the amount of K in the plant sample on the oven dry matter basis.

K (%) in plant sample = $X \times 4 \times 10^{-3}$

References :

Black, C.A. (1965) Methods of soil analysis Part I Am. Soc. Agron. Inc. Publi. Madison Wisconsin USA.

Principle :

Besides some amount in the soil solution, available sulphur in mineral soils occurs mainly as adsorbed $SO_4^=$ ions. Phosphate ions (as monocalcium phosphate) are generally preferred for replacement of the adsorbed $SO_4^=$ ions. The extraction is also carried out using $CaCl_2$ solution. However, the former is considered to be better for more efficient replacement of $SO_4^=$ ions. Use of Ca salts have a distinct advantage over and leads to easy filtration $SO_4^=$ in the extract can be estimated turbid metrically using a colorimeter/spectrophotometer.

A major problem arises when the amount of extracted sulphur is too low to be measured precisely.; To overcome this problem, addition of seed solution of known S concentration is added to the extract to raise concentration to easily detectable level. Sulphur in the extract can also be estimated by a colorimetric method using barium chromate (Nemeth 1964; Palaskar et al. 1981), but the turbidimetric method (Chesnin and Yien 1950) given below is mainly used in the soil testing laboratories.

Instruments :

(i) Colorimeter or spectrophotometer or autoanalyzer.
(ii) Mechanical shaker

Reagents

1. **Mono-calcium phosphate extracting solution (500 mg P L^{-1}):** Dissolve 2.035 g of $Ca(H_2PO_4)_2.H_2O$ per litre.
2. **Gum acacia-acetic acid solution:** Dissolve 5 g of chemically pure gum acacia powder in 500 mL of hot water and filtered in hot condition through Whatman No. 42 filter paper. Cool and dilute to one litre with dilute acetic acid.
3. **Barium chloride:** Pass AR grade $BaCl_2$ salt through 1 mm sieve and store for use.
4. **Standard stock solution (2000 mg S L^{-1}):** Dissolve 1.089 g of oven dried AR grade potassium sulphate per 100 mL.
5. **Working standard solution (10 mg S L^{-1})**: Measure exactly 2.5 mL of the stock solution and dilute to 500 mL.
6. **Barium sulphate seed suspension:** Dissolve18 g of AR grade $BaCl_2$ in 44 mL of hot water and add 0.5 mL of the standard stock solution (given above). Heat the contents to boiling and then cool quickly. Add 4 mL of gum acacia-acetic acid solution to it. Prepare a fresh seed suspension for each estimation everyday.
7. **Dilute nitric acid (approx 25%):** Dilute 250 mL of AR grade conc. HNO_3 to one litre.
8. **Acetic-phosphoric acid:** Mix 900 mL of AR grade glacial acetic acid with 300 mL of H_3PO_4 (AR grade).

Procedure :

1. Weight 20 g of soil sample in a 250 mL conical flask.
2. Add 100 mL of the monocalcium phosphate extracting solution (500 mg P L^{-1}) and shake for one hour. Filter through Whatman No. 42 filter Paper.
3. Measure 10 mL of the clear filtrate into a 25 mL volumetric flask.
4. Add 2.5 mL of 25% HNO_3 and 2 mL of acetic-phosphoric acid. Dilute to about 22 mL, stopper the flask and shake well.
5. Shake the $BaSO_4$ seed suspension and then add 0.5 mL of it and 0.2 g of $BaCl_2$ crystals. Stopper the flask and invert three times and keep.

6. After 10 minutes, invert 10 times and keep. After another 5 minutes, invert 5 times.
7. Allow to stand for 15 minutes and then add 1 mL of gum acacia-acetic acid solution.
8. Make up the volume, invert three times and keep aside for 90 minutes.
9. Invert 10 times and measure the colour intensity at 440 nm (blue filter).
10. Run a blank side by side.

Preparation of standard curve for S :

1. Place 2.5,5.0,7.5,10.0,12.5 and 15.0 mL portions of the working standard solution (10 mg S L^{-1}) into a series of 25 mL volumetric flasks to obtain 25,50,75,100,125 and 150 µg S.
2. Proceed to develop turbidity as described above for sample aliquots.
3. Read the colour intensity and prepare the curve by plotting readings against sulphur concentration (In µg in the final volume of 25 mL)

Calculation :

$$\text{Available S in soil (mg kg}^{-1}) = \frac{R \times 100}{10 \times 20}$$

Where, r stands for the quantity of S in mg as obtained on X-axis against a reading.

Determination of total sulphur in plant:

Sulphur is an essential plant nutrient and occurs in many different forms. The procedure for total sulphur estimation is as follows :

Digestion of plant material :

Take one gram of plant material in digestion flask. Add 10-15 ml of Diacid (3:1: Nitric acid : Perchloric acid) mixture and swirl the content in 150 ml volumetric flask. Place the content on hot plate till the digestion is over. Filter the solution in 100 ml conical flask, wash the residue on filter paper several times with the hot water. Make up the volume with distilled water, store the solution in air tight container.

Estimation :

Take 10 ml aligust from extract and proceed as per the method described under preparation of standard curve (Bardsley and Lancaster, 1960).

Calculation :

5 µg = R
1 R = R/5 µg (Factor)

$$\text{Total S (\%)} = \frac{\text{Factor x Sample R x1000 x 100 x 100}}{1000 \times 10 \times 1}$$

References :

Arora, C.L. and Bajwa, M.S. (1994). *Curr. Sci*. 66 : 314-316.
Bardsley, C.S. and Lancaster, J.P. (1960). Proc. Soil Sci. Soc. Am. 24 : 265.
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Determination of Zn, Cu, Fe and Mn in Soils and Plants by Atomic Absorption Spectrophotometer

All atoms can absorb light at certain discrete wavelengths corresponding to the energy requirement of the particular atom. When at ground state the atom absorbs light it is transformed into the excited state. It is the same atom containing more energy. This energy is measured in relation to the ground state and a particular excited state say for example in case of Na may be 2.2 eV (electron volts) above the ground state.

Each transition between different electronic energy states is characterized by a different energy and by a different wavelength. These wavelengths are sharply defined and when a range of wavelengths is surveyed, each wavelength shows as a sharp energy maximum (a spectronic line). These characteristic lines distinguish atomic spectra. The lines, which originate in the ground state of atom, are most often of interest in atomic absorption spectroscopy. These are called the resonance lines. The atomic spectrum, characteristic of each element, then comprises a number of discrete lines, some of which are resonance lines. Most of the other lines arise from excited states rather than the ground state. The lines of excited states are not useful generally in atomic absorption analysis as most of the atoms in a practical atomizer are found in the ground state.

The relationship of light absorbed by the atom in ground state and their concentration in the solution is defined in the fundamental laws of light absorptions.

Lambert's Law : The portion of light absorption by a transparent medium is independent of the intensity of the incidence light and each successive unit thickness of the medium absorbs an equal fraction of the light passing through it.

Beer's Law : Light absorption is proportional to the number of absorbing atoms in the sample.

The combined Beer - Lambert law may be given as :

$$I_t = I_o - (abc)$$

$$\text{thus } \log 10 \; \frac{I_o}{I_t} = abc = \text{absorbance}$$

Where, I_o = incident radiation power
I_t = transmitted radiation power
a = absorption coefficient
b = length of absorption path
c = concentration of absorbing atoms

i.e. the absorbance is proportional to the concentration of the elements for a given absorption path length at any given wave length.

In principle, it might be possible to calculate the concentration directly from the above equation. In practice, however, the a and b are constants hence the variation of results is directly related the concentration of atoms. For analysis, the absorbance of different concentration of standard solution is first measured with the help of atomic absorption spectrophotometer and then the results of unknown samples are compared with the standards and thus concentration of unknown sample is calculated.

Atomic absorption spectrophotometer :

Atomic absorption spectro-photometer is based on the principle that when atomic vapours of an element are irradiated by the radiation of a characteristic wavelength (i.e. the light from a source whose emission lines are those of the element in question), they absorb in direct proportion to the concentration of the element being determined.

Instrument features :

A wide range of atomic absorption spectrophotometer is available today, all of them have the basic features in common and consist of the following components:

(a) A Light source :

A Light source emits the spectrum of the element to be determined. The most widely used light source is hollow cathode lamp which is designed and operated in such a way that the lines to be measured are sharp, of stable intensity and free from background.

(b) Atomizer-Burner assembly :

A means of producing atomic vapours of the element to be analyzed. The solution to be analyzed is drawn by capillary and converted into stream of compressed air to a fine spray which after condensation of larger droplets is mixed with the fuel gas acetylene and burnt in a long flame (at 2100-2400^{o}C) in a stainless steel burner.

(c) A Monochromator :

It isolates the absorbing resonance lines from other non absorbing lines. When the light coming from the HCL, after traversing the flam, enters the monochromator which is already set at the wavelength of the resonance lines of the desired element, the monochromator performs its function.

(d) A Detector :

It measures the magnitude of absorption of the characteristic radiation.

(e) A Photomultiplier Tube :

It amplifies the absorption signal and converts the light radiation into electrical energy.

(f) A readout system :

It measures the absorbance in volts. It is normally a strip chart recorder, a digital display, a meter or printer. The presently available AAS have features like automatic calibration with one or more standards, automatic curve corrections, automatic and foolproof gas switching and calculation of average and standard deviations in repetitive runs.

Collection and preparation of soil and plant samples : To avoid contamination, soil samples are to be collected in plastic tub, using rust free instrument or wood and kept in polythene lined cloth bags. Samples are prepared with the help of wooden mortar and pastle and sieved through 2mm nylon screen/mosquito net cloth or stainless steel sieve.

Similarly plant samples (leaves, grains or straw) should be washed with 0.01N HCl, rinsed with glass distilled water dried in oven at 65°C and crushed with the help of stainless steel scissors.

Soil extraction : DTPA offers the most favourable combination of stability constants for the simultaneous complexing of Zn, Cu, Fe and Mn, Cd, Co, Ni and Pb (Lindsay and Norvell, 1978). Buffering of extractant in a slightly alkaline pH range (7.3) by including soluble Ca^{2+}, avoids the dissolution of $CaCO_3$ with the release of occluded micronutrients due to CO_2 partial pressure of approximately 10 times that in atmosphere, as the soil contains slightly higher CO_2 levels than found in the atmosphere.

(a) **Extracting solution** : (0.005 M DTPA) Dissolve 1.9679g of DTPA (Diethylene tri amine penta acetic acid) + 13.3 ml TEA (Triethanol amine) + 1.47g $CaCl_2 .2H_2O$ in 200 ml distilled water, dilute to 900 ml, adjust pH 7.3 with 6N HCl while stirring and then make upto 1 liter and mix thoroughly.

(b) **Apparatus required** : Shaker (Horizontal or Rotatory), iodine value flasks (100 ml capacity) or conical flasks with glass stoppers, funnels, filter paper whatman No.1, plastic storage bottles and Atomic absorption spectrophotometer.

(c) **Stock Standard Solutions** : The standard solutions of different micro-nutrients should preferably be prepared by using their wires. Dissolve 1g wire in a minimum volume of 1:1 nitric acid and dilute to 1000ml with distilled water to obtain 1000 μg/ml solution of micro-nutrient, or take salts of metals as follows:

Zn- 4.398g l^{-1} $ZnSO_4,7H_2O$
Cu- 3.929g l^{-1} $CuSO_4,5H_2O$
Fe- 4.977g l^{-1} $FeSO_4,7H_2O$
Mn- 3.598g l^{-1} $MnSO_4,H_2O$.

The prepared standards are also available in the market. Out of these standards, prepare working solution of 50 ppm. Then a series of standard solution of 0.5, 1.0, 1.5, 2.0 and 2.5 ppm may be prepared for each metal.

(d) Background correction : The reading of a spectral line always includes any contribution from the flame and sample matrix. Failure to correct properly for the background reading can be a source of serious error. Although the need for fast background correction is most obvious with graphite furnace work, it is also a consideration with flame atomic absorption.

The most common method of background correction in atomic absorption spectrometry involves the use of a continuum source such as a deuterium lamp to measure the background. The source used is a deuterium filled discharge lamp, which emits an intense continuum spectrum from 190 nm to about 400 nm. This is the region where most atomic absorption lines occur and where the effects of background absorption are most pronounced. The poly-atomic gas D_2, is used in the lamp because a continuum is produced rather than a line spectrum.

The deuterium lamp is different from a hollow cathode lamp in construction and operation. The lamp incorporates a heated, electron-emitting cathode, a metal anode and a restrictive aperture between the two. A discharge current of several hundred milli amperes excites the deuterium gas. The discharge is forced to pass through the small aperture, forming a defined area of high excitation and hence high light emission. A suitable window transmits the light to the spectrometer's optical system.

To obtain successful background correction the deuterium lamp must be correctly aligned, and its intensity must be matched to that of the hollow cathode lamp.

It is important that both the deuterium source and the hollow cathode source are aligned to follow the same optical path. If they are not, then the two measurements may not be made on the same atom population and significant errors may occur.

In order to balance the intensity of the deuterium lamp with the hollow cathode lamp, it may be necessary to change the hollow cathode lamp current to a higher or lower value depending on the relative intensities of the lamps.

Although most modern AA spectro-photometers incorporate so called "simultaneous" background correction, they rely on two measurements separated slight in time. One measurement is of the total absorbance (atomic plus background) and the other is of the background only. The background is electronically subtracted from the absorbance to give the background corrected atomic absorbance with the continuum source method of background correction, the total absorbance is measured during the hollow cathode lamp pulse and the background during the deuterium lamp pulse. With the Zeeman method using a modulated magnetic field, the total absorbance is measured with the magnetic field off and the background with the field on.

(e) Soil analysis : Weigh 12.5g soil sample in 100 ml iodine value flasks. Add 25 ml DTPA solution. Shake this mixture for 2 hours on shaker at 70 to 80 oscillation per minute, filter through acid washed distilled water rinsed, whatman No.1 filter paper and collect the filtrate in plastic bottles. Determine the content of micronutrients on atomic absorption spectrophotometer.

(i) Plant analysis : Weigh 0.5g plant sample in a conical flask (corning, 100 ml capacity). Add 10 to 12 ml of di acid mixture (1 perchloric + 4 nitric acid) and digest the mixture on hot plate till the residue is colour less. Now take off, cool dilute with distilled water and filter through whatman No.1 filter paper. Make up the volume of digestate to 50 ml. Read for micronutrient content on atomic absorption spectro-photometer.

Factors : For soil multiply the concentration read on AAS computer sheet by "2". Similarly for plants the multiplying factor will be 100 to get concentration in mg kg^{-1}.

Reference :

Lindsay, W.L. and Norvell, W.A. (1978). Proc. Soil Sci. Soc. Am. 42 : 421-428.

Boron occurs as anion in soils and is required by plants in very small quantity. Water soluble B makes the estimate of its availability to plants. Total boron in soils varies from 20 to 200 mg kg^{-1} and available (water soluble) boron in soils ranges from 0.03 to 12 mg kg^{-1} respectively. The threshhold value ranging from 0.1 to 0.5 mg kg^{-1} (water soluble B) depends upon the soil type, crops, and other factors, below which the response to applied boron may be expected. Some sensitive crops to boron deficiency are listed in table 1. Its availability is affected by soil pH as under:

- Deficiency of B is generally observed in old acid leached soils.
- Availability increased with the rise in soil pH having significant positive correlation with pH rising from 4.7 to 6.7.
- In neutral, saline and calcareous soils the B availability again decreases with the rise in soil pH having significant negative correlation with the rise in pH from 7.1 to 8.1. In calcareous soils B fixation occurs with the condensation of borate radical into long chains in the presence of Ca.

Table 1 : Sensitivity of crop to Boron deficiency

Sensitive	Medium	Low
Alfalfa	Apple	Barley
Cauliflower	Cabbage	Beans
Rape seed	Carrot	Corn
Conifers	Clover	Grasses
Peanuts	Cotton	Oat
Sugarbeet		Onion
Turnip		Pea
		Potato
		Soybean
		Wheat
		Rice

- In alkaline soils the availability of B is high and may be even toxic for plant growth.

Besides this the low moisture availability also causes B deficiency.

Irrigation water containing Boron between 0.3 to 0.6 mg kg^{-1} can be used safely, whereas, irrigating soils with water containing 1 to 3 mg kg^{-1} B causes toxicity of B in plants.

Boron determination (Azomethine H Method) :

Azomethine H forms coloured complex with H_3BO_3 in aqueous media. Over a concentration range of 0.5 to 10 µg B/ml the complex is stable at pH 5.1. Maximum absorbance occur at 420 nm with little or no interference from a wide variety of salts. This technique is rapid, reliable and more convenient to use than traditional procedures employing carmin, curcumin or quinalizarin (John *et al.*, 1975).

Apparatus :

(1) Spectrophotometer
(2) Poly-propylene tubes 10 ml capacity.

Reagents :

1. Distilled water
2. Buffer solution : Dissolve 250 g of ammonium acetate (NH_4OAc) and 15 g of ethylenediaminetetracetic acid (EDTA disodium salt) in 400 ml of distilled water. Slowly add 125 ml of glacial acetic acid and mix.
3. Azomethine H reagent : Dissolve 0.45 g of azomethine H in 100 ml of 1% L ascorbic acid solution. Fresh reagent should be prepared weekly and stored in a refrigerator.
4. Calcium hydroxide suspension : Add 0.4g $Ca(OH)_2$ to 100 ml distilled water.

5. 0.1 N HCl : Add 8.3 ml conc. HCl to 900 ml distilled water, mix, cool to room temperature and make up the volume to 1000 ml.
6. Calcium chloride 0.01 M Dissolve 1.11 g of anhydrous $CaCl_2$ in 900 ml distilled water and make up the volume to 1000 ml.
7. Boron standard solution : Dissolve 0.114g of Boric acid (H_3BO_3) in distilled water and adjust the volume to 1000 ml. Each ml contains 20 µg B. Dilute 10, 20, 30, 40 and 50ml of the stock solution to 100 ml with distilled water to have solution with B concentration of 2,4,6,8 and 10 µg of B/ml respectively. Include a distilled water sample for the 0.0 µg of B/ml standard solution.

Procedure :

Take 1 ml of aliquot of blank and diluted B standards into a 10 ml polypropylene tube, add 2 ml of buffer solution and mix. Add 2 ml of azomethine H reagent, mix and after 30 minutes read the absorbance at 420 nm on spetrophotometer. With the help of absorbance readings of standard solutions of different concentration of B the standard curve is drawn and a factor for concentration of B for 1 absorbance is calculated which is utilized to calculate B in the soils, plant or water sample.

Preparation of Extracts :

1. Soil extracts : The hot water soluble extraction procedure of Berger and Truog (1939) is being used widely with slight modification of adding dilute electrolyte (0.01 M $CaCl_2$) instead of water only. This provides clear, colourless extract which eliminates the need of charcoal for decolourzation. Beside this a negative error, associated with B adsorption by charcoal, is also removed.

Place 20 g air dry soil in 250 ml low B flat bottom flasks and add 40 ml of 0.01 M $CaCl_2$ solution. Attach water cooled reflux condenser to the flask. Heat the flasks for 5 minutes and then cool and filter the suspension in plastic bottles.

Transfer 20 ml aliquot to evaporating dish, add 2 ml $Ca(OH)_2$) suspension and evaporate the solution to dryness. Heat the evaporating dishes gently to destroy organic matter, cool to room temperature, add 5 ml 0.1N HCl. Triturate the residue with rubber policeman to ensure the complete dissolution of the residue (Bingham, 1982).

For analysis of B pipette 1 ml of the aliquot and proceed as for the standard curve.

2. Plant digest : Take 0.5 g plant sample in porcelain/platinum dishes Add 0.5 g $Ca(OH)_2$. Ignite the sample in the muffle furnace at 550°C for 4 hours to obtain white grey ash. Cool the dishes and moist the ash carefully with little distilled water and then add 5 ml 0.1N HCl. Transfer the content in to 25 ml volumetric flask mix and make up the volume to 25 ml with distilled water. For analysis of B take 1 ml of the aliquot and proceed as for the standard curve.

3. Water analysis : Take suitable quantity of water sample (containing 0.2 to 5.0 µg B) in porcelain dishes add 2 ml $Ca(OH)_2$ and proceed as described for soil extract. It is important to keep a definite volume of aliquot i.e. 1 ml of either soil, plant or water in final step of B determination.

References :

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Jackson, M.L. (Ed.) (1958). *Boron determination for soil and plant tissues*. In Soil Chemical Analysis page 370-387.

John, M.K., Chuah, H.H. and Neufld, J.H. (1975). Application of improved azomethine H method for the determination of boron in soil and plants. Anal. Lett. 8 : 559-568.

Deep black soils or Vertisoils occur globally under various parent materials and environmental conditions (Table 1).

Table 1. Distribution of Vertisols and associated soils

Juris-Diction	Location	Area (m ha)	% of Gross Black soils
Continent	Africa	105.0	38.7
	Asia & far East (Mainly India)	**70.3**	25.9
	Australia	48.8	17.7
	Latin America	27.0	9.9
	North America	10.0	3.7
	Near & Middle East	5.7	2.1
	Europe	5.4	2.0
	TOTAL	**271.4**	**100**
Country	India	**70.3**	25.9
	Australia	48.8	17.7
	Sudan	43.4	16.6
	USA	18.1	6.7
	CHAD	15.5	5.7
	China	11.6	4.3
	Others (in parts)	64.5	23.7
	TOTAL	**271.4**	**100**
India	MS	24.2	34.4
	MP	**21.2**	30.1
	GUJ.	4.9	7.0
	AP	9.4	13.4
	KTK.	5.8	8.2
	TN	2.6	3.7
	RAJ.	1.1	1.6
	UP	1.1	1.6
	TOTAL	**70.3**	**100**
MP	Vertisols	8.0	37.7
	Inceptisols	8.6	40.6
	Entisols	4.2	19.8
	Alfisols	0.4	1.9
	TOTAL	**21.2**	**100**

They clayey soils that shrink and swell extensively upon changing soil moisture conditions. Vertisols exhibit unique morphological properties such as the presence of slickensides, wedge-shaped aggregates, diapir (mukara), and gilgai. Shrink-swell phenomena are the dominant pedogenic processes in Vertisols and are attributed to changes in interparticle and intraparticle porosity with changes in moisture content.

Definition of Vertisols :

Taxonomically for defining Vertisols, there must be

1. A layer 25 cm or more thick with an upper boundary within 100 cm of the mineral soil surface, that has either SLICKENSIDES or WEDGE SHAPED PEDS that have their long axes tilted 10 to 60^o from the horizontal; and
2. A weighted average of 30 % or more clay in fine earth fraction either between the mineral soil surface and a depth of 18 cm or in Ap horizon, whichever is thicker and
3. 30 % or more clay in fine earth fraction of all horizons between a depth of 18 cm and either a depth of 50 cm or a densic, lithic or paralithic contact, a duripan, or a petrocalcic horizon if shallower and
4. Cracks that open and close periodically.

Vertisols are significant global resources that serve as the lifeline in subsistence agriculture due to their high productivity.

Efforts towards comprehension and successful utilization are imperative for continued productivity and long term sustainability of these resources for current and future civilizations.

Morphology of a soil is best evaluated from the *in situ* examination of the soil profile. A recently dug pit large enough for observation of a pedon is desirable. Old exposures such as road banks and ditches are acceptable only for preliminary studies because morphological features often become altered after prolong exposure.

Normally the size of profile pit is kept 1.8 m long, 1.2 m wide and 1.8 m deep but for the study of black soils, the width of pit varies from place to place depending on its cyclic wave length of puffs and shelves. It should be kept in mind that atleast half wave length covering both, puff and shelve is considered while exposing profile pit in order to study the pattern of cracks and slickensides perfectly.

The profile examination begins with a first approximation and marking of soil horizon boundaries on the profile. Each horizon is then carefully observed and described. Horizon boundaries are relocated as required by the detailed study (Buol *et al*. 1998). Th description sheet containing the columns of site and soil characteristics is filled up by the profile study group during pedon studies.

Vertisols are relatively homogneous in their morphology. Although horizonation is not distinct yet a few horizons above the parent material may be identified as self mulching surface (Ap), blocky subsurface (A12), slickensided horizon and wedge shaped subsoil (Bss).

The depth of these soils may vary from shallow to very deep. Previously the black soils were grouped as shallow (<30 cm) medium (30-100 cm) and deep (>100 cm) but lateron Sehgal (2008) modified the dept of shallow soil as less than 50 cm.

Requirement of Vertisols :

Main requirements of Vertisols are the presence of high content of clay (>30 %) and predominance of montmorillonite (2:1 expanding clay). Other important parameters for the development of Vertisols are:

(i) **Parent material** having basalt, argillaceous limestone, marine clays and shales

(ii) **Weathering period** must be extensive for the development of solum with 2:1 expanding clays

(iii) **Weathering environment** should be such that no further weathering of 2:1 expanding clays takes place. Even no inter-layering exists to the extent the properties are destroyed

(iv) **Sequence of events** should continue like churning/mixing, development of argilli-pedoturbation, development of slickensides and formation of wedge shaped structures

Pedogenesis of Vertisols :

1. Separation of blocks :

Deep wide cracks separate the soil into strong and massive prism like blocks in the upper part of the pedon that break into angular blocky peds of hard and firm consistence.

(a) ***Cracking of soil*** : During dry season, the soil cracks to the surface due to shrinkage of 2:1 expanding clays that may extend to a depth of 1 metre or more.

(b) ***Falling of surface soil material*** : While cracks are open, surface soil material falls into them by several mechanisms such as animal activity, wind or at the onset of rainy season by water.

2. Hydration of clays : The clay hydrate and due to their high coefficient of expansion and contraction, expand 3 dimensionally on wetting.

(a) ***Expansion of clays*** : Cracks close on the surface but because of the extra material now present in the lower part of the profile, a greater volume is attained and the expanding material presses and slides the aggregates against each other, developing a "lentil" angular blocky structure with sliekenside features on the ped surfaces.

(b) ***Shear stress development*** : The slipping occurs where shear strength is surpassed by shear stress acting upon a soil mass. The shear stress is a major force caused by swelling and develops when volume expansion results during the wet cycle.

(c) ***Formation of slickensides*** : The slickensides, intersecting or close enough to intersect, also result in wedge shaped structural aggregates, the most characteristics feature of Vertisols which develop with their longitudinal axes inclined at 30 to 60° from horizontal (Sehgal and Bhattacharjee, 1988).

(d) ***Buckeling of land space*** : This expansion buckles the land scape, forming the micro relief called gilgai. The micro basins contain more organic matter than the micro ridges and probably it results from admixtures of subsurface material into micro ridge area and slight erosion of organic rich fines from the ridges to the basins.

3. **Incomplete leaching** : In most shrink swell soils, the temperature being high, the potential evapotranspiration suggesting incomplete leaching and inducing the process of calcification in these soils.

Cyclic movement of soil material :

Amongst several processes acting in the formation of Vertisols, the predominant process seems to be haploidization i.e. mixing by argilli pedoturbation. The specific features of such soils are :

1. **Gilgai micro relief** : The term gilgai is an Australian aboriginal term meaning small water hole.

Pedogenic micro topographical features like puffs (microknolls) and shelves (microbasins) develop that remain intimately associated with one another (Bhattacharjee *et al*. 1977), Columbe *et al*. (1996) introduced a term "diapir" meaning a protusion of subjacent soil material which penetrates to the overlying horizons and approaches or reaches the surface. If diapir and gilgai occur, the mound in gilgai is always developed over the diapir.

Hallsworth and Beckman (1969) classified gilgai into 6 types i.e. normal or round, melon hole, Lattice, Linear or wavy, tank or stony but lateron Paton (1974) suggested only two types of gilgai i.e. linear and circular (Nuram or Pockmarked) each of which were grouped into 4 types.

α type - Mound and depression equally developed (No shelf present)

β type - Mound of much greater extent than depression (No shelf present)

γ type - Depression of much greater extent than mound (No shelf present)

δ type - Mound, shelf and depression all present

2. **Size of cyclic pedons** : Half cycle linear distance (HCLD) measures the lateral dimension of a cyclic pedon. It may be small, medium or large i.e. below 1, 1 to 2 or above 2 to 3.5 metre, respectively.

3. **Horizon sequence** : In Vertisols, the horizon sequence has been suggested to be A1-Bss-BC-C where "ss" indicates about the presence of slickensides.

4. **Thickness of horizon** : Thickness of A1 in Vertisols varies with the linear frequencies of puffs and shelves of gilgai micro relief.

5. **Horizon boundary (Amplitude)**: It is the difference between vertical distance from the surface of pedon to the lower boundary of crest of cycle and the lowest point of trough of cycle in same pedon. The amplitudes are grouped as low, medium of high according to the vertical distance as below 25, 25 to 75 or above 75 cm, respectively. Shape of apparent topography of the intermittent horizon is also graded as tongued (vertical extent > horizontal distance), wavy (vertical extent approximating the horizontal distance) and smooth (vertical extent < horizontal distances) as suggested by Bartelli (1971).

Age of Vertisols :

It is difficult to assign the Vertisols a place in the genetic scheme of soil classification as there are greater differences of opinion whether they are old, young or remain in equilibrium with the environment.

1. Views as Vertisols are old : The end product of a development sequence involves the soils whose B horizon has become so clayey that shrink-swell cycles developed and eventually "swallowed" the A horizon. It is possible because high content of fine clay and high fc/cc ratio may be produced by lessivage on a large scale.

2. Views as Vertisols are young : The fate of Vertisol may be to undergo alteration of 2:1 clays to non expanding type of clay. The profile would then cease to churn and eluviation process would dominate. This interpretation suggests that Vertisols are relatively young soils.

3. View as Vertisols are in equilibrium : Vertisols remain in equilibrium with their environment and that the 2:1 expanding lattice clays are stable and will persist, barring a climate change. Vertisols then can be considered diagnostic of environments in which the parent material is basic and gives rise to the formation of 2:1 expanding lattice silicates under the influence of wet dry climate.

Table 2 : Range in characteristics of Vertisols and Vertic Inceptisols

Horizon	Soil colour (10 YR)	Texture	Structure	Special features	Width of cracks (cm)
A. Typic Haplustert (10 YR - 2.5 YR)					
Ap/A11	4/2, 3/3, 3/2, 3/1	C	1f/1m sbk	1c/2c pr-3c pr	2-5
A12	3/3, 3/2, 3/1	C	2m/2c abk	2c pr - 3c pr	2-5
Bss	3/3, 3,2, 3/1, 2/1	C	2m/3c abk	Intersecting lickensides*	1-2
BC	4/4, 3/4	C	2m/2c abk	----do----	0.5-1
C	5/4, 4/4, 4/3	c-gc	2msbk/ massive	-	-
B. Vertic Haplustept (10 YR - 7.5 YR)					
Ap/A	5/2, 4/3, 4/2, 3/2	Cl	gr/1m sbk	1c pr-2c pr	2-2.5
AB	4/3, 3/3	cl-c	1m/2m sbk	----do----	2-2.5
B21	4/3, 3/3, 3/2	cl-c	2m sbk-3m/3c	2c pr - 3c pr or pressure faces/abk slickensides	1.5
B22	6/3, 5/3, 4/4, 4/3	gscl-cl	----do----	-	-
C	7/6, 6/3, 5/3, 4/4	gsl-gscl	1f sbk/ massive	-	-

*or parallelepipeds with long axes tilted from 35° to 55° from horizontal

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