Methods Manual
Soil Testing In India

Department of Agriculture & Cooperation

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FOREWORD

I am glad to note that INM Division has brought out a manual on soil testing. The manual provides exhaustive guidance for setting up soil testing laboratories, carrying out soil tests, preparing soil fertility maps and issuing soil health cards to the farmers. Use of soil test kits and Mobile Soil Testing Laboratories have also been explained in the manual.

I trust that all State Government agencies engaged with soil testing and the Fertilizer Industry will find the manual useful in implementing the soil testing programme. I am confident that the manual would contribute towards soil test based fertilizer usage which will help in increasing the fertilizer use efficiency and farmers' incomes.

19th January, 2011

(P.K. Basu)
19/1/11

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PREFACE

Soil Testing is well recognized as a sound scientific tool to assess inherent power of soil to supply plant nutrients. The benefits of soil testing have been established through scientific research, extensive field demonstrations, and on the basis of actual fertilizer use by the farmers on soil test based fertilizer use recommendations. Soil testing was initiated in the country in the beginning of planning era by setting up of 16 soil testing laboratories during 1955. Government of India has been supporting this programme during different plan periods to increase the soil analysing capacity in the country. The numerical strength does not, however, decisively indicate the quality and success of the programme. Planners and agriculturalists have recognized the utility of the service fully but it suffers due to inadequate scientific support in its execution.

To provide the soil testing laboratories with suitable technical literature on various aspects of soil testing, including testing methods and formulations of fertilizer recommendations etc., the Union Ministry of Agriculture have decided to bring out this manual. The manual provides elaborate information on major soil types of India, their composition, plant nutrient and their functions, typical deficiency symptoms of nutrients in plants, apart from procedure of sample collection and methods of analysis. Detailed information has been provided about the establishment of soil testing laboratories, basic cares required in the laboratories, calibration procedures for testing methods and the need and procedures for collaborating with Soils Research Institutes in ICAR system and concerned State Agricultural Universities. Information about the usefulness of soil testing kit and mobile soil testing vans along with their limitations and usefulness has been provided. Since the soil testing laboratories are invariably required to analyse irrigation water samples, hence a chapter on irrigation water analysis has been provided.

It may be pointed out that the methods to extract available nutrients from the soil are rather old in terms of their enunciation, being mostly given out in the years as early as 1940s and 50s but are still popular and being followed world over. What has fundamentally changed is to categorize the available nutrients extracted by these methods into the limits of sufficiency, deficiency or somewhere in between in relation to present day crop varieties and soil nutrient status. This is achieved through extensive research trials by ICAR and SAUs system to establish a correlation between soil test values so obtained and crop response to applied fertilizers. Thus, the critical input in improving the soil test based fertilizer use recommendation would be 'ratings' given to these values. Another aspect of latest scientific input in the soil testing programme would be to analyse these extracted amounts of nutrients by modern, heavy duty and fast analyzing equipment so that the capacity and accuracy of the soil testing laboratories could be enhanced. Description of such equipments, like, auto analysers, atomic absorption

spectrophotometer and inductively coupled plasma-atomic emission spectrometer has been given in the manual.

It is suggested that each state may set up a modal / central soil testing lab where modern equipments may be provided. Also the system of making online fertilizer use recommendation may be introduced only in the central labs at the initial stages. It has been emphatically described in the manual that the soil testing laboratories may maintain a live relationship with the soils department in the SAUs / ICAR system where regular training programmes may be organized for the heads of Central Lab while the technical staff of other laboratories may be trained at nodal and central laboratory with the help and support of soil scientists from State Agriculture Universities.

It is expected that the manual will be useful for the technical staff of the soil testing laboratories in doing their day-to-day analytical work and framing fertilizer use recommendations. Implementation of various suggestions given in the manual would also help in improving the quality of the work being done by the soil testing laboratories.

Integrated Nutrient Management Division, Department of Agriculture & Cooperation, Government of India acknowledges the contribution of Dr. M.R. Motsara in compiling this manual as consultant.

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Integrated Nutrient Management Division,
Department of Agriculture & Cooperation,
Government of India

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CHAPTER 1

SOILS

Soil may be defined as a thin layer of earth's crust which serves as a natural medium for the growth of plants. It is the unconsolidated mineral matter that has been subjected to, and influenced by genetic and environmental factors – parent material, climate, organisms and topography all acting over a period of time. Soil differs from the parent material in the morphological, physical, chemical and biological properties. Also, soils differ among themselves in some or all the properties, depending on the differences in the genetic and environmental factors. Thus some soils are red, some are black; some are deep and some are shallow; some are coarse-textured and some are fine-textured. They serve in varying degree as a reservoir of nutrients and water for crops, provide mechanical anchorage and favourable tilth. The components of soils are mineral material, organic matter, water and air, the proportions of which vary and which together form a system for plant growth; hence the need to study the soils in perspective.

Rocks are the chief sources for the parent materials over which soils are developed. There are three main kinds of rocks: (i) igneous rocks, (ii) sedimentary rocks and (iii) metamorphic rocks.

The rocks vary greatly in chemical composition and accordingly the soils differ in their properties because they are formed from the weathering of rocks. Weathering can be physical or chemical in nature. The agents of physical weathering are temperature, water, wind, plant and animals while chemical processes of weathering are hydration, hydrolysis, carbonation, oxidation and reduction.

A developed soil will have a well defined profile which is a vertical section of the soil through all its horizons and it extends up to the parent materials. The horizons (layers) in the soil profile which may vary in thickness may be distinguished from morphological characteristics which include colour, texture, structure etc. Generally, the profile consists of three mineral horizons – A, B and C.

The A horizon may consist of sub-horizons richer in organic matter intricately mixed with mineral matter. Horizon B is below A and shows dominance of clay, iron, aluminium and humus alone or in combination. The C horizon excludes the bedrock from which A and B horizon are presumed to have been formed.

A study of the soil profile is important from crop husbandry point of view, since it reveals the surface and the sub-surface characteristic and

qualities namely, depth, texture, structure, drainage conditions and soil moisture relationship which directly affect the plant growth. **A study of soil profile supplemented by physical, chemical and biological properties of the soil will give full picture of soil fertility and productivity.**

Physical properties of the soil include water holding capacity, aeration, plasticity, texture, structure, density and colour etc. Chemical properties refer to the mineralogical composition and the content of the type of mineral such as Kaolinite, illite and montmorillonite, base saturation, humus and organic matter content. The biological property refer to a content of extent and types of microbes in the soil which include bacteria, fungi, worms and insects.

1.1. Major Soil Types of India:

Some dominant groups of Indian soil, classified according to soil taxonomy and chemical property are mentioned below:

1) Red Soil: They are quite wide in their spread. The red colour is due to diffusion of iron in the profile.
2) Lateritic soil: are composed of a mixture of hydrated oxides of aluminium and iron with small amounts of manganese oxide.
3) Black soil: contains a high proportion of Calcium and Magnesium Carbonates and have a high degree of fertility.
4) Alluvial soils: This is the largest and agriculturally most important group of soils.
5) Desert soils: occur mostly in dry areas and important content is quartz.
6) Forest and Hill Soils high in organic matter.

Taxonomically these soils are classified as follows:

Red soil	: Alfisol, Inceptisol and Ultisol
Lateritic soil	: Alfisol, Ultisol and Oxisol
Black soil	: Vertisol, Inceptisol and Entisol
Alluvial soil	: Entesol, Inceptisol and Alfisol
Desert soil	: Entisol and Aridisol
Forest and Hill soils	: Alfisol

Problem soils: According to salt content:

(i) Saline and Alkali soil
(ii) Acid soil
(iii) Peaty and Marshy soil

The soils are studied and classified according to their use which is termed as land capability classification. In this classification, inherent soil characteristic, external land features and environmental factors are given prominence. For this purpose, soil survey is carried out to record the crop limiting factors such as soil depth, topography, texture-structure, water holding capacity, drainage features, followed by evaluation of soil

fertility status, based on soil testing / analysis. The soils are thus classified in to 8 classes, four of which are considered suitable for agricultural purpose (Class I & IV) and Class V to VIII are non-arable lands and can be used for silviculture and forest and need strong conservation measures. An effective linkage between soil testing and soil survey is useful to ensure formulation of a sound soil fertility evaluation programme.

The above soil groups which have been extensively studied because of their extent and agricultural importance are described below:

I. Red Soils: The red soils of India, including red loams and yellow earths, occupy about 200,000 sq.miles and extend over a large part of Tamil Nadu, Mysore, south-east Maharashtra and a tract along the eastern part of Madhya Pradesh to Chota Nagpur and Orissa. In the north and north-east these extend into and include great part of the Santhal Parganas of Bihar; Birbhum, Bankura and Midnapur districts of West Bengal; Khasi, Jaintia, Garo and Naga Hills areas of Assam; Mirzapur, Jhansi, Banda and Hamirpur districts of Uttar Pradesh; Baghelkhand division of Madhya Pradesh and Aravallis and the eastern half of Rajasthan.

The main features of these soils, besides their lighter texture and porous and friable nature, are: (a) the absence of lime (*kankar*) and free carbonates, and (b) the usual presence of soluble salts in a small quantity, not exceeding 0.05 percent. These soils are generally neutral to acid in reaction, and deficient in nitrogen, phosphoric acid, humus and perhaps lime. They differ greatly in depth and fertility, and produce a large variety of crops under rainfed or irrigated conditions. They are divided into two broad classes: (1) the red loams, characterized by a cloddy structure and the presence of only a few concretionary materials; and (2) the red earth with loose top-soil, friable but rich secondary concretions of a sesquioxidic clayey character.

The soil contains a high percentage of decomposable hornblende, suggesting a comparatively immature nature. The silica-alumina ratio of the clay fractions is 2.7-2.46 and their base exchange capacities are below 20 m.e. per 100 gm., suggesting their predominantly kaolinitic nature. In the typical red earth the silica-alumina ratio of the clay fractions is higher than 2 and they are fairly rich in iron oxide.

The soils have undergone excessive weathering and very low amount of decomposable mineral hornblende.

In Tamil Nadu the red soils occupy a large part of cultivated area. They are rather shallow, open in texture with the pH ranging between 6.6 and 8.0. They have a low base status and low exchange capacity, and are deficient in organic matter, poor in plant nutrients, and with the clay fraction ratio of 2.5 – 3.0.

The predominant soil in the eastern tract of Mysore is the red soil, overlying the granite from which it is derived. The loamy red soils are

predominant in the plantation districts of Shimoga, Hassan and Kadur. **They are rich in total and available K_2O, and contain fair amounts of total P_2O_5 (0.5 – 0.3 percent); the lime content is 0.1 – 0.8 per cent, nitrogen below 0.1 per cent, and iron and alumina 30 – 40 per cent**. A broad strip of area lying between the eastern and western parts of Coorg is red loam, easily drained and with a fairly dense growth of trees.

The acid soils in the south of Bihar (Ranchi, Hazaribagh, Santhal Parganas, Manbhum and Singhbhum) are red soils. Their pH is 5.0 – 6.8 and they have high percentage of acid-soluble Fe_2O_3 as compared with Al_2O_3 ; sufficient available potash but P_2O_5 is low. The soils from Manbhum, Palamau and Singhbhum are preponderant in zircon, hornblende and rutile respectively; those of Ranchi contain a mixture of epidote and hornblende, neither of which is preponderating.

In West Bengal the red soils, sometimes misrepresented as laterites, are the transported soils from the hills of the Chhota Nagpur Plateau.

The existing tracts of soils in north-west Orissa are quite heterogeneous. There seems to be a prominent influence of the rolling and undulating topography on soil characteristics.

The soils are slightly acidic to neutral in reaction and the total soluble salts are fairly low. Ferruginous concretions are invariably met with, whereas calcareous concretions are present only in a few cases at lower depths of the profiles.

In a typical red soil profile the total exchangeable bases is about 20 m.e., the SiO_2- R_2O_3 ratio of the clay fractions varies between 2 and 3, and the C – N ratio is near about 10.

The soils of Raipur district (Chattisgarh area) are grouped into the following classes :

Matasi : Upland or level land soils, yellow, loam to clay loam and loamy clay yielding good paddy.

Dorsa : Soils along the slopes, somewhat darker; same texture as above; good paddy lands.

Kanhar : Lowland soils, dark, slightly heavier than the above; paddy is the main crop; wheat is also grown.

Bhate : Barren waste lands with gravel and sandy reddish-yellow; usually in uplands.

A part of Jhansi district (Uttar Pradesh) comprises red soils. These are of two types : *Parwa*, a brownish-grey soil, varying from good loam and sand or clay loam, and *rakar*, the true red soil generally not useful for cultivation.

In the Telangana division of Andhra Pradesh both red and black soils predominate. The red soils or *chalkas* are sandy loam located at higher levels and are utilized for cultivation of *kharif* crops.

Another type of soil occurring in Andhra Pradesh is locally known as *dubba*. It is loamy sand or very coarse sandy loam, and mostly pale-brown to brown with reddish-brown patches here and there ; clay content is quite low (less than 10 percent) and very low fertility ; invariably neutral in reaction and low in soluble salt content. The content of organic matter is little to negligible. The soils are severely eroded with surface soil depth below five inches and very often covered with multi-sized gravels and cobbles. Being sub-marginal lands they are well suited for pasture and forage crops rather than for rice growing.

II. Laterite and lateritic soils: These soils occupy an area of about 49,000 sq.miles in India. The laterite is specially well-developed on the summits of the Deccan Hills, Central India, Madhya Pradesh, the Rajmahal Hills, the Eastern Ghats, in certain plains of Orissa, Maharashtra, Malabar and Assam. These are found to develop under fair amount of rainfall, and alternating wet and dry periods. The laterite and lateritic soils are characterized by a compact to vesicular mass in the subsoils horizons composed essentially of mixture of the hydrated oxides of aluminium and iron. These soils are deficient in potash, phosphoric acid and lime. On higher levels these soils are exceedingly thin and gravelly, but on lower levels and in the valleys they range from heavy loam to clays and produce good crops, particularly rice.

Both the high-level and low-level laterites occur in Tamil Nadu. They are both *in situ* and sedimentary formations, and are found all along the West Coast and also in some parts of the East Coast, where the rainfall is heavy and humid climate prevails. In the laterites on lower elevations paddy is grown, while tea, cinchona, rubber and coffee are grown on those situated on high elevations. **The soils are rich in nutrients including organic matter. The pH is generally low, particularly of the soils under tea (pH 3.5 – 4.0) and at higher elevation.**

In Maharashtra laterites are found only in Ratnagiri and Kanara; those in the latter are coarse, **poor in lime and P_2O_5, but fairly good in nitrogen and potash**. In the former, coarse material abounds in large quantities. These are rich in plant food constituents, except lime.

In Kerala, between the broad sea belt consisting of sandy soil and sandy loams and the eastern regions comprising the forest and plantation soils, the mainland contains residual laterite. **These are poor in total and available P_2O_5, K_2O and CaO**. Laterite rock in cochin is found to the east of the alluvial areas in Trichur, Talapalli and Mukundapuram taluks. Soil is mostly laterite in Trichur taluk. The nitrogen content varies from 0.03 – 0.33 per cent; the lime is very poor and the magnesium is 0.11 – 0.45 per cent. The laterite soils in Mysore occur in the western parts of Shimoga,

Hassan, Kadur and Mysore districts. All the soils are comparable to the laterites and to the similar formations found in Malabar, Nilgiris, etc. These soils are very low in bases, like lime, due to severe leaching and erosion. These are poor in P_2O_5. The pH is not as low as that in the plantation soils.

In West Bengal the area between the Damodar and the Bhagirathi is interspersed with some basaltic and granitic hills with laterite capping. Bankura district is known to be located in the laterite soils zone. The SiO_2 – Al_2O_3 ratio of the clay fraction is quite high. **The percentage of K_2O, P_2O_5 and N are low, showing considerable leaching and washing out of these substances due to chemical weathering.** The soils of Burdwan are in all respects similar to the Birbhum and Bankura soils with one or two exceptions. The high value of the SiO_2 – Al_2O_3 ratio is significant.

In Bihar laterite occurs principally as a cap on the higher plateau but is also found in some valleys in fair thickness.

The laterites of Orissa are found largely capping the hills and plateau occasionally in considerable thickness. Large areas in Khurda are occupied by laterites. At Balasore, it is gravely. Two types of laterites are found in Orissa, the laterite *murrum* and the laterite rock. They may occur together.

III. Black soils: These soils cover a large area throughout the southern half of the peninsula, the Deccan Plateau, greater part of Maharashtra State, western parts of Madhya Pradesh and Andhra Pradesh, and some parts of Tamil Nadu State, including the districts of Ramnad and Tinnevelly. The black soils or *regur* include a large number of physiographic regions, each within a zone having its own combination of soils. These soils may be divided into three groups : (1) deep and heavy; (2) medium and light; and (3) those in the valleys of rivers flowing through *regur* area.

The main features of the black soils are: (1) depth one to two or several feet deep; (2) loamy to clayey in texture; (3) cracking heavily in summer, the cracks reaching up to more than three or four feet in depth, especially in the case of heavy clays; and (4) containing lime *kankar* and free carbonates (mostly $CaCO_3$) mixed with the soil at some depths. These soils are often rich in montmorillonitic and beidlite group of minerals, and are usually suitable for the cotton cultivation. They are generally deficient in nitrogen, phosphoric acid and organic matter; potash and lime are usually sufficient. The content of water-soluble salts is high, but the investigations carried out in connection with Tungabhadra and Nizamsagar projects have shown that these soils may be irrigated without any danger, if irrigation is carried out on sound lines.

Though the black soils do not have distinct demarcation of horizons between the unweathered parent material and the weathered soil, the soil profile may be said to possess approximately three principal horizons A, B and C, the alluvial or A horizon being predominant and of two types,

namely, darker with high organic matter content and lighter. The zone of accumulation of carbonates ($CaCO_3$) and sulphates (chiefly $CaSO_4$) may be taken as the B or illuvial horizon. In regions of fairly high and evenly distributed rainfall the zone of carbonate accumulation is found deeper in the profile and sometimes incorporated with horizon C.

The occurrence of black and red soils in close proximity is quite common in India. In Maharashtra soils derived from the Deccan trap occupy quite a large area. On the uplands and on the slopes, the black soils are light coloured, thin and poor; and on the lowlands and the valleys they are deep and relatively clayey. Along the Ghats the soils are very coarse and gravelly. In the valleys of the Tapti, the Narmada, the Godavari and the Krishna heavy black soil is often 20 feet deep. The subsoil contains good deal of lime. Outside the Deccan trap the black cotton soils predominate in Surat and Broach districts. Degraded solonized black soils, locally known as *chopan*, occur along the canal zones in the Bombay Deccan.

A large number of typical black soil profiles have been examined in Tamil Nadu. They are either deep or shallow and may or may not contain gypsum in their profile, and accordingly four types of profiles are distinguished : (1) shallow with gypsum, (2) shallow without gypsum, (3) deep with gypsum, and (4) deep without gypsum. The shallow profiles are three to four feet deep, often with partially weathered rock material even at a depth of 1.5 – 2.0 feet; the deep ones extend even up to nine feet or more.

The black soils are very heavy, contain 65-80 per cent of finer fractions, have high pH (8.5 – 9.0) and are rich in lime (5-7 per cent); they have low permeability, high values of hygroscopic coefficient, pore-space, maximum water-holding capacity and true specific gravity. **They are low in nitrogen but contain sufficient potash and P_2O_5.** They have generally a high base status and high base exchange capacity (4 60 meg.) ; about 10-13 per cent iron content, and the CaO and MgO contents are formed from a variety of rocks, including traps, granites and gneisses.

In Madhya Pradesh the black soils are either deep and heavy (covering the Narmada Valley) or shallow (in the districts Nimar, Wardha, west of Nagpur, Saugor and Jabalpur). The cotton-growing areas are mainly covered by the deep heavy black soils which, however, gradually change in colour from deep-black to light. The $CaCO_3$ content increases with the depth. Clay content is 35-50 per cent, the organic matter is low and SiO_2-R_2O_3 ratio is 3 – 3.5.

The black soils of Mysore are fairly heavy with high salt concentration, and rich in lime and magnesia. The SiO_2-R_2O_3 ratio of clay fraction is 3.6.

IV. Alluvial soils: The so-called alluvial soils of India form an ill-defined group. Various types of alluvium are classed as alluvial, e.g., calcareous soils, saline and alkali soils, and coastal soils.

The alluvial soils occur mainly in the southern, north-western and north-eastern parts of India : the Punjab, Uttar Pradesh, Bihar, West Bengal, parts of Assam, Orissa, and coastal regions of southern India including the local deltaic alluvia. **These soils are the most fertile amongst the Indian soils**. The whole of the Indo-Gangetic plain is, in this alluvial area, of 300,000 square miles. These soils are very deep, deeper than 300 ft. at some places, and deficient in nitrogen and humus, occasionally in phosphoric acid but not generally in potash and lime. They support a variety of crops, including rice, wheat and sugarcane. They may be sub-divided into two broad groups, the old and the new; both are geological groupings. The former, locally called *bangar*, represents reddish-brown, sandy loams with increasing content of clay in the lower horizons ; the latter, known as *khaddar*, represents the fairly coarse sand on the *chars* and banks of the river to the soils of very fine texture in the low-lying marshy tracts. The old alluvium reddish in colour, is deficient in nitrogen and humus, and occasionally in phosphoric acid.

The large expanse of these soils is yellowish to brownish and their common feature is the presence of *kankar* or lime nodules intermixed with soil at varying depths. They vary from sandy loam to clayey loam. The subsoil occasionally has calcareous reaction. There is no marked differentiation into various horizons, and the profile is often characterized by the absence of stratification. The surface soil is generally grey, varying from yellow to light brown, the intensity of colour increasing with the depth.

The immature soil near the rivers is calcareous and light brown in colour with salt impregnation. On higher situations it becomes brown to deep brown in colour and is non-calcareous. *Kankar* beds are found in the soil. Most of the alluvial soils in Uttar Pradesh and Bihar are of the above pattern.

V. Desert Soil: A large part of the arid region in Rajasthan and part of Haryana, lying between the Indus and the Aravallies, is affected by desert conditions of recent geological origin. This part is covered under a mantle of blown sand which inhibits the growth of soils.

The Rajasthan desert proper (area about 40,000 sq. miles), owing to its physiographic conditions receives no rain though lying in the tract of the south-west monsoon. Some of the desert soils contain high percentage of soluble salts, high pH, varying percentage of calcium carbonate and poor organic matter, the limiting factor being mainly water. The soils could be reclaimed if proper facilities for irrigation are available.

VI. Forest and hill soils: Nearly 22-23 per cent of the total area of India is under forests. The formation of forest soils is mainly governed by the characteristic deposition of organic matter derived from the forest growth. Broadly two types of soil-formation may be recognized(1) soils formed under

acid conditions with presence of acid humus and low base status; and (2) soils formed under slightly acid or neutral condition with high base status which is favourable for the formation of brown earths.

The soils of the hilly districts of Assam are of fine texture and reveal high content of organic matter and nitrogen, perhaps due to the virgin nature. Their chemical and mechanical composition show great variations.

In Uttar Pradesh the sub-Himalayan tract comprises three regions, viz., *bhabar* area immediately below the hills, *terai* and the plains. Four major groups, i.e., red loams, brown forest soils, podsols and transitional podsols have been observed in the Himalayan tract ; of these, brown forest soils and podsols are predominant. The *terai* area is characterized by excessive soil moisture and luxuriant vegetation. The soils are clay loam, loam and sandy loam. The loam may have high content or minor quantities of lime or lack of it altogether.

In the Himachal Pradesh, the typical soil profiles under deodar, spruce, blue pine and *chir* are related to the podsols but have significant differences, probably due to the relatively high calcium content of the first layer. These differences are brought out in the high degree of saturation of various horizons.

The weathering of metamorphic rocks in Coorg has produced very fertile deep surface soil which annually receives the decomposition products of the virgin forest. The areas towards the west are for the greater part reserved forests and mountain areas. The land surface has a laterite bed, is easily drained and is full of pebbles.

In Nilambur the soils which do not grow teak are more clayey, contain more MnO and possess a lower Sio_2-R_2O_3 ratio; this makes them suitable for teak plantation.

The cinchona-growing soils of West Bengal resemble brown earths. Their surface layer consists of well-decomposed humus and mineral soil which shades off gradually, at varying depths, into the colour of the parent rock. These soils are strongly acidic in reaction. Their high base exchange capacity is due to the high organic matter content. Water-soluble manganese is present in appreciable amount.

Soils classified as per the problem of salt content:

I. Saline and alkaline soils: The distribution of saline and alkaline soils is extensive throughout India in all climatic zones. Many parts of Bihar, Uttar Pradesh, Punjab, Haryana and Rajasthan, give rise to saline and alkaline efflorescence in the same way as the soils capping the Upper Tertiary rocks. There are many yet undecomposed mineral fragments in these alluvial clays and silts, which, on weathering, liberate sodium, magnesium and calcium salts. Large areas, once fertile and populous, have

become impregnated with these salts locally known as *reh* or *kalkar* with highly deleterious effect on their fitness for cultivation. The injurious salts are confined to the top layers of the soils, which are charged by capillary transference of saline solutions from the lower layers. This transference is facilitated in lands irrigated by canal waters.

Reh is a mixture of carbonate, sulphate and chloride of sodium, and some other salts of calcium and magnesium. It is also introduced by rivers and canals. The water of the great alluvial plains which are without any underground drainage become concentrated with salts. Capillary action during the summer months brings them to the surface where they form a white efflorescent crust.

It has been estimated that two lakh acres of land in the Punjab and about 1 lac are in Haryana have been affected by *usar* and 25 thousand acres are being added every year. Methods of reclamation based on irrigation, application gypsum, where necessary, and growth of salt-resistant crops like rice, berseem and sugarcane are suggested. In cases of badly damaged alkali patches, treatment with sulphur or gypsum accompanied by adequate watering has led to steady improvement in the soil, and successful crops have been raised.

The soils of Hardoi, Lucknow and Kanpur districts are characterized by very high pH values and by almost complete absence of gypsum due to restricted internal drainage. Soils appear to be the carbonate-chloride type of saline-alkali in contrast to soils of more arid localities of temperate climates.

Alkali soils are met with all over the states of Gujarat and Maharashtra, but badly affected lands are found in Gujarat, Karnatak and the Deccan.

Such soils show high content of exchangeable monovalent bases and magnesium with a predominance of chlorides, amounting to more than 50 per cent. Reclamation of these lands by bunding and leaching of soluble salts is possible.

Portions of Dharwar district and Bijapur *taluk* are affected by what is locally known as *kari* soils, which are saline-alkaline, fairly deep and clayey. The salt lands of the Nira Valley have developed as a result of excessive irrigation given to the deep black soils of the locality. In these soils two groups of profiles, one resembling steppe alkali soils and the other the solonetz, might be distinguished.

The soils of Delhi State have one of these pedogenic groups: (a) saline (mostly in the *khaddar* area); and (b) saline-alkali, with *kankar* formation (mostly in *dabar* and *bangar* areas, and in depression of *khadar* areas).

II. Acid Soils: In India about 6.5 million hectares of land area is covered by strongly acid soils (pH < 5.5). The acid soil areas occur in and around Bhawali (UP), Palampur, Kangra (HP), Baster and Jagdalpur (MP), Jorhat and Titabar (Assam), Ratnagiri (Maharashtra) and large areas in Ooty (Tamil Nadu) and Kutanad (Kerala). The acid soils suffer due to lack of calcium and magnesium and in some cases due to aluminium and iron toxicity. However, these soils are being cultivated and their productivity can be improved when limed.

III. Peaty and marshy soils: Peaty soils originate in humid regions as a result of accumulation of large amounts of organic matter in the soil. They may also contain considerable amounts of soluble salts. They are found in Kerala. They are generally submerged under water during the monsoon. After the rains, these are put under paddy cultivation. Soils are black, heavy and highly acidic, with pH as low as 3.9, and contain 10-40 per cent of organic matter. Their acidity is due to the decomposition of organic matter under anaerobic conditions, and no nitrification is possible. Sometimes the soils contain ferrous and aluminium sulphates.

The depression formed by dried river-basins and lakes in alluvial and coastal areas sometimes give rise to peculiar waterlogged and anaerobic conditions of the soils. The soils of these places are generally blue due to the presence of ferrous iron and contain varying amounts or organic matter. Marshy soils of this type are found in the coastal tracts of Orissa, the Sunderbans and some other places in West Bengal, central portion of north Bihar, Almora district of Uttar Pradesh, and south-east coast of Chennai.

Source: Soils of India and their management , FAI(1985)

1.2 . Soil Composition

(a) Solid phase

- Mineral constituents including weathered rock fragments.
- Organic matter, both dead and living micro and macro organisms.

The mineral constituents are

Sand Large particles which are coarse, and individual particles are easily visible (0.02 – 2 mm in diameter) most common mineral component is silica (SiO_2)

Silt Medium-sized particles which are 0.002-0.02 mm in diameter.

Clay Small particles which are less than 0.002 mm in diameter and are referred to as soil colloids.

By definition, particles larger than sand are not soil.

(b) Liquid phase

- Soil water which occupies the pore spaces (between mineral particles) carries the plant nutrients to the roots.

(c) Soil air

- It occupies pore spaces similar to soil water. It fills these voids when soil water is absent.
- It carries respiratory products of roots and soil-organisms.
- It has a higher concentration of carbon dioxide than atmospheric air.

(d) Soil texture

- Refers to the relative proportion of sand, silt and clay present in a soil.
- Based on these proportions soils are classified into various textural classes.
- Clayey soils have a larger percent of clay. They are considered more fertile than sandy soils but are difficult to work.
- Sandy soils are easy to work but are less fertile. They have low water retention capacity.
- Loamy soils are in between sandy and clayey soils. They are best for arable cropping.

(e) Soil structure

- Refers to the arrangement of the different particles into soil aggregates.
- Roots move between these aggregates.
- A compact soil will resist root movement.
- The organic matter content helps the soil aggregation process.

Soil fertility

It is the capacity of a soil to supply plant nutrients in adequate amounts to facilitate optimum growth and obtaining the yield potential of a crop. Many soil properties which determine the availability of plant nutrients and thus soil fertility are as follows:

(i) Soil colour

Reddish to brownish colour shows well-drained conditions. Degree of yellowness and mottling show poor drainage. Gray to dark colour indicates the presence of organic material.

(ii) Soil depth

Refers to the depth to which plant roots can penetrate the soil and is the distance between the lowest and the upper most horizons of the soil.

(iii) Bulk density

It is the weight of the soil in the given volume. A compact soil has a higher value while an organic soil has a lower value. It also affects water holding capacity of the soil.

(iv) Field capacity

- Refers to the moisture content of a soil after the loss of gravitational water.
- At this point, water is held in soil micropores, which is available to plant roots, until the water content down to a lower value.
- This lower value is referred to as the permanent wilting point.
- The amount of water available between field capacity and permanent wilting point is referred to as available water.
- This value is important in determining irrigation intervals.

(v) Soil pH

- Measures the negative logarithm of the hydrogen ion activity of the soil solution.
- Is a measure of the soil acidity or alkalinity of a soil.

(vi) Soil acidity

Is caused by many factors such as

- Excessive rain which leaches basic cations (Ca, Mg, K)
- Use of nitrogenous fertilizer like urea, ammonium sulphate etc.
- Oxidation of iron pyrite containing minerals.

Correction of soil acidity

- Soil acidity is corrected by liming; soils which have a pH value of less than 5.5 should be limed.

(vii) Salinity and alkalinity

- Occurs in arid and semi arid regions, where precipitation is insufficient to meet evapo-transpiration needs of plants, when salts move up to the surface.
- Salt affected soils occur within irrigated lands.
- Salts are added through irrigation water; through over-irrigation and salts accumulate in poorly drained areas.
- Salt content in soils is measured in terms of electrical conductivity (EC).
- A saline soil has a pH of less than 8.5, but soils are well flocculated. The EC is more than 4 deci Seimens per meter (4dS m^{-1}).
- Soils which are saline cause problems to crops during dry weather. Loss of crop yield from poor growth is common.
- Alkaline soils have high pH (more than 8.5) and a high concentration of sodium in them.
- Alkaline soils are deflocculated, drainage is poor and growing plants is difficult due to high pH and higher content of sodium in the soil.

- Draining, flushing after ploughing and addition of organic matter and gypsum could correct these problems.

(viii) Cation Exchange Capacity (CEC)

- The power to retain cations at the surface of soil colloids is referred to as the cation exchange capacity.
- Soil colloids of clay and organic matter have this property due to the presence of negative charges at the surface.
- CEC is defined as the sum of cations held by a kilogram of soil. It is expressed in Cmo/Kg soil.
- Clays like kaolinite have low CEC and the CEC is pH dependent. Organic matter has a large CEC but it too is pH dependent.
- Montmorillinitic clay has high CEC due to the negative charges developed through loss of cations during formation of these clays.
- Practices like fertilization, liming, irrigation and addition of organic manures can increase the exchangeable cations.

(ix) Soil organic matter (SOM)

- Consists of living organisms, dead plant and animal residues.
- Is the most chemically active portion of the soil.
- Is a reservoir for various essential elements.
- Contributes to CEC.
- Promotes good soil structure.
- Buffers soil pH.
- Promotes good air and water relations in plants.

Functions of organic matter in soil

- Supplies N,P, S and other secondary and micro-nutrients for plant growth.
- Increases CEC in a soil.
- Have large surface area and has high CEC.
- Holds up to twenty times their weight of water.
- Holds cations and anions and releases them slowly.
- The ratio of C:N:P:S is 10:1:.5:0.1
- Effects the breakdown of pesticides and weedicides.
- Chelates micronutrients such as Zn, Mn and Cu making them more available to plant roots.
- Buffers extreme acidity, salinity and alkalinity.

Fate of organic material added to soils

- Undergoes decomposition (only bio-degradable materials) through soil macro and micro fauna and flora.
- Final degradation is made by soil microorganisms.
- The final product is a humus type material which has a C:N ratio around 9:1 – 12:1.
- All complex carbon products are converted into simple compounds

(x) Nitrogen cycle

This is one of the most important naturally occurring events. It is discussed under:

- Nitrogen fixation.
- Conversion of N in soil.
- Mineralization

(a) Nitrogen fixation

- Nitrogen is a very inert gas which constitutes 78% of the atmospheric air; it has low chemical reactivity.
- For use by plants it has to be converted to ammonium (NH_4^+) or nitrate (NO_3^-)
- Only a few microorganisms can utilize N gas which is referred to as N fixation.
- Fixation is effected by microbial action, industrial synthesis and high thermal combustion and lightning.

(b) Conversion of N in soil

N occurs in soils in the following form :

- Soluble mineral forms, ammonium, nitrate, nitrous oxide (gas)
- Soluble organic compounds, urea, aminoacids.
- Living organisms, plant roots, fungi, bacteria, soil animals.
- Insoluble forms, organic nitrogen, ammonia bonded to clay.

Transformation between the different forms is mediated by soil microorganisms.

(c) Mineralization

- Conversion of N in organic residues and soil organic N into soluble forms through mineralization.
- Carbon sources are degraded sources of energy.
- N in excess of microbial need is liberated.

The following sequence of reactions takes place.

i) **Ammonification:** Complex protein compounds are broken down to ammonium compounds by micro-organisms.

ii) **Nitrification:** Ammonium compounds are oxidized to nitrite and to nitrate by two specific types of soil bacteria, *Nitrosomonas* and *Nitrobactor.*

iii) **Denitrification:** The nitrates are reduced to nitrogen gas under poorly aerated conditions through specific micro-organisms.

CHAPTER 2

PLANT NUTRIENTS AND THEIR FUNCTIONS

Several elements take part in the growth and development of plants, and those absorbed from the soil are generally known as plant nutrients. Besides these, the plant takes up carbon, oxygen and hydrogen, either from the air or from the water absorbed by roots. **In all, 16 elements have been identified and are established to be essential for plant growth.** There are carbon (C), hydrogen (H), Oxygen (O), nitrogen (N), phosphorus(P), potassium (K), calcium(Ca), magnesium (Mg), iron (Fe), sulphur(S), zinc (Zn), manganese (Mn), copper (Cu), boron (B), molybdenum (Mo), and chlorine(Cl). These elements serve as raw materials for growth and development of plants, and formation of fruits and seeds.

Most of the essential elements are found in liberal quantities in the mineral soils. In spite of the fact that these are available in plenty, these may not be available to the plants, as they are tied up in mineral and chemical compounds. The roots cannot absorb and deliver them to the growing plants for synthesis, and hence, the need for assessing the plant available amounts of nutrients in the soil and meeting deficiency by application of manures and fertilizers to such soils for optimum crop production.

2.1. Plant Nutrients

Although plants absorb a large number of elements, all of them are not essential for the growth of crops. The elements are absorbed became they happen to be in the soil solution and those taking active part in the growth and developmental processes are called the essential ones. Some of these are required in large amounts and some in traces. These are classified as major and micro nutrients, and are further classified as follow:

Major nutrients

Group I :	Carbon, hydrogen and oxygen.
Group II:	Nitrogen, phosphorus, potassium

Secondary Nutrients: Calcium, magnesium, sulphur

Micro nutrients:

Iron, manganese, boron, zinc, copper, molybdenum and chlorine.

It has been found that the presence of some elements which are not considered essential for plant growth and are not directly concerned in the

nutrition of the crop, but are present in the plants used as food and feed, are of vital importance to the health of man and animals. The elements within this group are iodine, cobalt and sodium. In addition, there is another group of elements which are toxic to the animals feeding on the plants containing them. These are selenium, lead, thallium, arsenic and fluorine. Elements, such as sodium, fluorine, nickel, lead, arsenic, selenium, aluminium and chromium, when occurring in soils in high available amounts, may also prove toxic to the plants and restrict their growth.

Some elements, occurring freely in the soil, are absorbed by the plants as impurities. They may occasionally stimulate growth although they are not essential for plant growth. They include lithium, strontium, tin, radium, beryllium, vanadium, barium, mercury, silver and bromine.

Silica is reported to be 'beneficial', particularly in rice but is not classified as essential as per the criteria fixed for this purpose.

Essential plant nutrient

i) The completion of the life cycle of the plant cannot be achieved in the absence of such an element.

ii) Plays a specific role in the plant.

iii) Causes set back to growth of the plant showing visual symptoms when the plant is deficient in it.

Sixteen elements identified as essential are listed in **Table-A.** Carbon, hydrogen and oxygen are obtained from air & water. The other thirteen elements are referred to as fertilizer elements and have to be obtained from the soil and or added through chemical fertilizers or organic manures. Their addition in quantities necessary for plant growth will increase the growth rate, dry matter content and yield of the crop.

- Plant nutrients are usually absorbed through roots. Roots have the ability to absorb nutrients selectively.

- Root absorption takes place both as active and passive absorption.
    - Active absorption takes place as an exchange phenomenon and requires energy. Most plant nutrients are absorbed in this manner.

    - Passive absorption is part of the transpiration cycle (mass flow). Water and some dissolved solutes are absorbed by this process.

- Gas exchange takes place through the stomata found in leaves. Carbon dioxide required for photosynthesis and oxygen required for plant and respiration are exchanged through the leaves.

The supply of an adequate quantity of a particular nutrient for crop growth depends on both the behaviour of that nutrient in the soil and the ability of the crop root system to utilize it.

When these elements are not available to the plant in quantities optimum for growth, the quantity and quality of yield is affected. This requires regular fertilization of crops after determining soil nutrient deficiency.

Table A: Nutrients Essential for plant growth and forms in which taken up by plants

Nutrient	Chemical symbol	Form taken up by plant
Primary Nutrients		
1. Carbon	C	CO_2, HCO_3
2. Hydrogen	H	H_2O
3. Oxygen	O	H_2O, O_2
4. Nitrogen	N	NH_4^+ , NO_3^-
5. Phosphorus	P	$H_2PO_4^-$, HPO_4^{-2}
6. Potassium	K	K^+
Secondary Nutrients		
7. Calcium	Ca	Ca^{2+}
8. Magnesium	Mg	Mg^{2+}
9. Sulphur	S	SO_4^{2-}
Micro Nutrients		
10. Iron	Fe	Fe^{2+} , Fe^{3+}, chelate
11. Zinc	Zn	Zn^{2+}, $Zn(OH)_2$,
12. Manganese	Mn	chelate
13. Copper	Cu	Mn^{2+}, chelate
14. Boron	B	Cu^{2+} , chelate
15. Molybdenum	Mo	$B(OH)_3$
16. Chlorine	Cl	MoO_4^-
		Cl^-

Deficiency of an element

When an essential element is at a low concentration in the plant tissues, it will result in the decrease in normal growth of the plant, affect the crop yield and produce more or less distinct deficiency symptoms.

- Typical deficiency symptoms are not often clearly defined. Masking effects due to other nutrients, secondary causes like disease, herbicide toxicity or insect infestation can confuse field diagnosis.

- Waterlogged conditions or dry soils and mechanical damage can often create symptoms that mimic deficiencies.

- Deficiency symptoms always indicate severe starvation.

Insufficient levels

When the level of an essential plant nutrient is below the required amount for optimum yields or when there is an imbalance with other nutrients it is considered insufficient. The symptoms of this condition are seldom clearly visible, resulting in poor yield.

Toxicity

Certain essential plant nutrients, if taken up in excess will often cause nutrient imbalances and will result in poor plant growth, delayed maturity, stunted and spindly growth and also show visible symptoms of chlorosis or necrosis.

2.2. Fate of Nutrient Elements in Soil

Nutrients are lost through Crop removal, Erosion, Leaching, Volatilization, Denitrification and Fixation.

Crop removal

- Plant species have specific requirements of plant nutrients.
- Nutrient removal depends on Growth condition, Crop sanitation, Cultivation and Yield obtained
- Grain crops require more nitrogen than other nutrients. Pulses require more phosphorous while crops such as tomato, banana and pineapple require more potassium compared to other nutrients.

Erosion

- Entire top soil is lost through erosion by water or wind; this results in loss of soil phosphorus.

Leaching

- Water percolating through a soil profile carry dissolved nutrient elements. Nutrients are easily lost in humid regions and sandy soils.
- Bare soil loses more nutrients than cultivated soils.

Volatilization
Nitrogen is easily lost through volatilization as ammonia, particularly in paddy soils and upland soils in poorly drained areas. This is referred to as ammonia volatilization. This loss is enhanced by high temperature and wind.

De-nitrification
Nitrate form of N is lost through denitrification where nitrogen gas or nitrous oxide is released. This loss occurs mainly in paddy soils and in upland soils which are saturated with water periodically or part of the time

Fixation
- Takes place by conversion of a nutrient to an unavailable form.
- Phosphorus is converted to unavailable forms both in acidic and alkaline soils, as Al/Fe phosphate or $Ca_3 (PO_4)_2$ respectively.
- Potassium and ammonium N can be fixed by certain clay minerals.

2.3. Function of Nutrients in Crop Productions

In the nutrition of a crop various nutrients perform distinct functions. Their relative essential role depends upon whether they enter into chemical composition or regulate various physiological processes in the plant.

Carbon. The plant absorbs carbon dioxide directly from the atmosphere. This combines with water in the presence of light and forms the primary sugars, such as glucose and fructose (fruit sugar). Chlorophyll is the pigment which absorbs the radiant energy of the sun and brings about complex chemical syntheses of carbon dioxide and water resulting in simple sugars. This process is called photosynthesis or synthesis in light. Thus, by the combination of carbon with water, sun's energy is stored in the plant body, and the first carbohydrates are formed in the plant. From the carbohydrates complex sugars, starches, hemicelluloses and celluloses are formed.

These simple sugars also polymerise (chemically combine) into oils and fats. For instance, the soluble carbohydrates (sugars) decrease from 37.5 to 4.5 per cent during the ripening of sunflower seed. Same has been observed about oil in niger seeds.

Oxygen. Oxygen is part of water as well as carbon dioxide. When water combines with carbon dioxide oxygen is evolved:

$$6CO_2 + 6H_2O \text{ -- } C_6H_{12}O_6 + 6O_2$$

Thus, the oxygen evolved in the process equals the volume of carbon dioxide absorbed by the plants. This evolution of oxygen takes place in the process of photosynthesis.

The process is reverse in respiration when a simple or a complex sugar, fat or oils breaks up. It requires oxygen and gives out CO_2.

$$C_6H_{12}O_6 + 6O_2 - 6CO_2 + 6H_2O$$
(Glucose)

Six molecules of carbon in the glucose combine with six molecules of oxygen to form six molecules of carbon dioxide. In the process six molecules of water are formed. Thus, oxygen plays a dominant role in the processes of photosynthesis and respiration in plants.

Hydrogen. It is one of the most important elements in the nature. It readily combines with oxygen to form water and with carbon to form complex chemical organic compounds. The growth of plants would only take place if adequate quantity of water is supplied to meet the needs of hydrogen for synthesis of organic substances. When organic compounds either break up in the plant or decompose in the soil or atmosphere, the released hydrogen always combines with oxygen and forms water. Thus, the exchange of hydrogen takes place in either of the synthesis or decomposition (including respiration) processes.

Nitrogen. It is not only an essential part of carbohydrates, fats and oils but also an essential ingredient of proteins. Other constituents of proteins are oxygen, hydrogen and nitrogen, usually sulphur and sometimes phosphorus. The majority of the proteins have the following composition (in per cent):

Carbon	50.0-55.0
Hydrogen	6.5-7.3
Nitrogen	15.0-17.6
Oxygen	19.0-24.0
Sulphur	3.0-5.0

When proteins decompose through hydrolysis they give out amino acids; reversely, when proteins are formed or synthesized the basic substances are amino acids.

Nitrogen is the basic nutrient and makes up 1-4% of day weight of plants and it forms chlorophyll, amino acids, proteins, alkaloids and protoplasm. In the plant sap ammonia, nitrates and nitrites are found only in traces or very small quantities. When the plant takes up large quantities of nitrogen from the soil the colour of the plant changes to dark-green, indicating the increase of chlorophyll in the plant. Since the amount of chlorophyll in the plant determines the carbohydrate synthesis, nitrogen, in a way, may be said to control this activity. When there is less uptake of nitrogen, the leaves remain small and pale-yellow in colour. As the level of nitrogen supply increases, compared with other nutrients, the extra protein produced enlarges the leaves which provides larger leaf surface for

photosynthesis and makes the leaves more succulent and less coarse, increases the length of the growing season and delays maturity. In relation to shoot growth the root growth is depressed. It helps in seed formation and increases the food and feed value of crops. **But when the crop plants become more succulent due to larger availability of nitrogen they become susceptible to pests and diseases.**

Source of Nitrogen to the plants are following:

i) Free living micro-organism fix 16-50kg N/ha/year.
ii) Organic matter in the soil by decomposition produces 1-2 percent N per ha and contribute 20-45 kg N/ha.
iii) Rain water adds about 5-6 kg N/ha/year.
iv) Nitrogenous chemical fertilizers and organic manures / compost / vermi compost are an important source of N supply to crops.

Phosphorus. It is a constituent of the cell nucleus, essential for cell division and the development of meristematic tissues at the growing points. It makes 0.1 to 0.5% of dry weight of the plant. Therefore, plants which cannot absorb adequate quantities of phosphorus from the soil have small root system and leaves, and their growth is stunted. In cereals tillering is reduced and maturity is delayed. **Phosphorus is particularly helpful in the production of legumes, as it increases the activity of nodular bacteria which fix nitrogen in the soil.** It aids the formation of seeds and fruits, particularly in the legumes. It stimulates early root growth and development. Optimum quantity of phosphorus available to the crop in combination with nitrogen balances their shoot and root growth.

Potassium. Unlike nitrogen and phosphorus, potassium is not a constituent of the carbohydrates, oils, fats and proteins, the substances which form the fabric of the plants. But it plays a vital role in the formation or synthesis of amino acids and proteins from ammonium ions which are absorbed from the soil. It is also considered essential in the photosynthetic activity of the leaves. When potassium is in short supply the carbon dioxide is synthesized into sugars more slowly than when it is available in optimum quantity. The relative concentration of sodium and calcium also influences the activity of potassium in the plant. It helps in moving manufactured food, viz., carbohydrates (sugars) and proteins (amino acids), from leaves to roots. It favours the growth of legumes in competition with other plants. **The stalks and stems (of plants) are more stiff when an adequate supply is available than otherwise. In consequence the lodging in cereals is reduced. It increases the plumpness of the grains. In general it imparts vigour and resistance to diseases.** Some crops, such as potato, tomato, clovers, Lucerne and beans, are more responsive to potassium than other crops. As larger quantities of carbohydrates and proteins are stored, it increases winter hardiness of some plants, such as Lucerne. It constitutes 0.8to3.0% of dry matter in cereals.

Sulphur. Sulphur is a constituent of many proteins, and aids in the formation of chlorophyll and root growth. Plants having an abundant supply of sulphur develop dark-green leaves and extensive root system. In legumes the nodular activity is appreciably increased by adequate supply of sulphur. Due to larger availability of proteins the plant growth is vigorous. It improves the starch control of tubers.

Calcium. Calcium is essential for the formation of cell-walls, as calcium pectate forms part of the middle layer of the cell-wall. The middle lamella regulates the entry of only those nutrients which are not toxic to the plant. In root-tips calcium is very essential for the meristematic activity or formation of new tissues. It also helps to keep up sustained activity of the nodule bacteria in legumes.

Besides its direct nutrient value, calcium when applied to acid soils increases the availability of other nutrients, like phosphorus, nitrogen and molybdenum. Excess of calcium in the calcareous soils depresses the uptake of potassium and magnesium. These are secondary effects of calcium on plant growth.

Magnesium. The chlorophyll development is much reduced when magnesium uptake is restricted because it is an integral part of the pigment. It maintains the dark-green colour of leaves and **regulates the uptake of other materials, particularly nitrogen and phosphorus**. It appears to play an important role in the transport of phosphorus, particularly into the seeds. It is also said to promote formation of oils and fats, possibly by increasing photosynthetic activity in the leaves.

Iron. Although iron does not enter into the composition of chlorophyll, **its deficiency manifests itself in chlorosis, yellowing or whitening of leaves**. The concentration of iron ions plays and important part in the oxidation process in leaf cells. When iron is not taken up in adequate quantity, the growth of plants is less vigorous, and seed and fruit development suffer as a consequence of decreased photosynthetic activity in the leaves. Too much liming results in iron deficiency. Sever deficiency results in chlorosis and leaves turn white and eventual leaf loss.

Manganese. Manganese is an essential element and appears to have a role in the formation or synthesis of chlorophyll. Due to deficiency of manganese the carbohydrate synthesis is disturbed, resulting in retarded growth, decrease in the content of ash and failure to reproduce. The leaves and roots of plants deficient in manganese have much less of sugars than those which can absorb sufficient quantity of manganese. **Manganese, probably in association with iron, is a constituent of some respiratory enzymes and some enzymes responsible for protein synthesis from the amino acids formed in the leaves.**

Boron. One of the most marked effect of boron deficiency observed is the restricted development of nodules on the roots of legumes. Very little

nitrogen is fixed in these nodules. Besides, its deficiency influences the growing points of stems, buds and roots. The tissues carrying the minerals and water from the soil to the leaves are also disorganized in the absence of boron. The leaves become brittle. **Many deficiency diseases, such as internal cork of apples, heart rot of sugar-beets, top rot of tobacco and cracked stem of celery, are caused by its deficiency.**

Zinc. In a general way zinc is associated with the development of chlorophyll in leaves and a high content of zinc is correlated with a high amount of chlorophyll. In its absence growth is less, buds fall off and seed development is limited. In peach and apricots zinc-deficiency symptoms are manifested in leaf. In small trees bronzing of leaves is mitigated by spraying zinc sulphate on leaves. The citrus mottling of leaves may be frequently due to the deficiency of zinc in the plant.

Copper. In the chloroplasts of leaves there is an enzyme which is concerned with the oxidation-reduction processes. The presence of copper is essential for this enzyme to function. **Thus, copper plays an important role in the process of photosynthesis**. Deciduous fruit trees affected with chlorosis, resetting and dieback recover quickly on application of copper sulphate to the soil in amounts ranging from 0.5-2 lb. per tree.

Molybdenum. The presence of molybdenum is very essential for the fixation of atmospheric nitrogen the roots of legumes by nodule bacteria. In plants it is essential for the nitrate-reducing enzyme, as plants well supplied with ammonium do not need it as an essential element. The deformity of 'whiptail' produced in cauliflower is due to the deficiency of molybdenum.

Considering the role played by various essential elements they may be grouped as follows:

Group I	Energy exchangers	Hydrogen and oxygen
Group II	Energy storers	Carbon, nitrogen, phosphorus and sulphur
Group III	Translocation regulators	Potassium, sodium, calcium and magnesium
Group IV	Oxidation-reduction regulators	Iron, manganese, molybdenum, copper, boron and zinc

This grouping is based on the physiological functions of the elements in the synthesis of carbohydrates, proteins, fats, oils, enzymes and other substances which are part of the active mass of protoplasm. In plants all functions are well coordinated for growth and development. Growth is disturbed in proportion to the deficiency of any of these nutrients.

CHAPTER 3

DEFICIENCY SYMPTOMS OF NUTRIENTS IN PLANTS

The plants exhibit hunger signs when they cannot adequately absorb plant nutrients. These symptoms of hunger for nutrients are readily recognizable under field conditions. The hunger can be readily satisfied by the application of fertilizers to the soil.

Hidden hunger

There are no visual symptoms of deficiency but the plant is not producing at its capacity. When the plant reaches the level where symptoms appear, the yield may already have been greatly reduced.

Identification of nutrient hunger signs is basic to profitable crop production as it helps in deciding about its application to the soil/crop. Deficiency symptoms can be categorized into five types.

i) Chlorosis, which is yellowing, either uniform or interveinal of plant leaf tissue due to reduction in the chlorophyll formation.
ii) Necrosis, or death of plant tissue.
iii) Lack of new growth or terminal growth resulting in rosetting.
iv) An accumulation of anthocyanin and / or appearance of a reddish colour.
v) Stunting or reduced growth with either normal or dark green colour or yellowing.

The symptoms of nutrient-wise deficiency are described below. Typical deficiency symptoms in one of the major serial crop i.e. rice are depicted in plates 1-11*. The effect of soil salinity on crop condition is shown in plate 12*. [***Source:- A practical Guide to Nutrient Management – International Rice Research Institute, DAPO Box 7777, Metro Manila, Philippines. Edited by Thomas Fairhurst & Christian Witt.]***

Nitrogen. The nitrogen-deficient plants are light green in colour. **The lower leaves turn yellow and in some crops they quickly start drying up as if suffering from shortage of water.** The growth is stunted and stems or shoots are dwarfed. In cereals tillering is restricted. In corn if nitrogen deficiency persists the yellowing will follow up the leaf midrib in the typical V-shaped pattern with the leaf margins remaining green. The drying up of lower leaves is generally referred to as firing. In small grains, namely, wheat, barley and oats, the nitrogen- starved plants are erect and spindly and the leaves have yellowish-green to yellow colour. The stems are

purplish-green. In potato, in the later stages of growth, the margins of lower leaflets lose their green colour and become pale-yellow. In cotton the blades and petioles are reduced in size, turn yellow or brown and die. Plants produce fewer lateral branches, reduced number of fruiting branches, and very much reduced number of flowers and bolls. In legumes the growth is stunted and the lower leaves are pale-yellow or brownish in colour. In citrus the leaf shedding is heavy. Their leaves are small in size, thin and fragile and have light green colour. In deciduous fruit trees the leaves have yellowish green appearance. The old, mature leaves are discoloured from base to tip. Under prolonged deficiency twigs become hard and slender. In vegetables there is retarded growth with leaf chlorosis. The stems are slender, fibrous and hard. (Plate-1)

Phosphorus. Generally the plant is dark-green but the lower leaves may turn yellow and dry up. Growth is stunted and leaves become smaller in size. In corn, leaves and stems have a tendency to become purplish; young plants are stunted and dark-green in colour. Small grains have dark-green colour and often have purplish tinge. They have retarded growth. In potato, in early stages, the plants have stunted spindly growth. The tubers have rusty-brown lesions in the flesh in the form of isolated flecks which sometimes join together to produce larger discoloured areas. The cotton plants have dark-green colour, leaves and stems are small, and the bolls mature late. Besides the dark-green colour of legume plants their petioles and leaflets are tilted upwards. The plants are spindly and stunted. Their stems often turn red. In citrus the plants show reduced growth. The older leaves at first lose their deep-green colour and luster, and develop faded green to bronze colour. Necrotic areas develop on such leaves. In deciduous fruit trees the young leaves have dark-green colour while mature ones have bronze or ochre dark-green colour. The new twigs are slender. In vegetables although the growth is retarded the leaves do not show symptoms of chlorosis. In many crops the under surface of leaves develops reddish-purple colour. The stems are slender and woody. They bear small, dark-green leaves. (Plate-2)

Potassium. The margins of leaves turn brownish and dry up. The stem remains slender. In tobacco there appear small spots of dead tissue between the veins, at leaf tips and margins which are tucked or cupped up. In maize, in the young stage, the edges and tips become dry and appear scorched or fired. At a later stage in well-grown plants the leaves are streaked with yellow and yellowish-green colour, and the margins dry up and get scorched. Similar symptoms are shown by oats, wheat and barley. In potato the deficiency of potassium is acutely manifested. The plant growth is retarded, the internodes are somewhat shortened, the leaf size is reduced and they form a sharper angle with the leaf petiole. The leaflets become crinkled and curve downward. The older leaves become yellowish, develop a brown or bronze colour, starting from the tip and edge and gradually affecting the entire leaf, and finally die. Malnutrition symptom in cotton is observed in 'cotton rot', which first appears as yellowish-white mottling and then changes to yellowish-green; subsequently yellowish spots

appear between the veins. The centres of these spots die and numerous brown specks occur at the top, around the margin and between the veins. The breakdown first occurs at the tip and margin of the leaf. The leaf curls downwards before it becomes reddish-brown and dries up. In legumes the first symptoms consist of yellow mottling around the edges of the leaf. This area soon dries up and dies. The plants have stunted growth. In citrus there occurs there occurs excessive shedding of leaves at blossom time. There is a tendency for the young shoots to shed before they become hardened. The leaves are small. In deciduous trees the necrosis (death of tissues) in foliage occurs, the necrotic areas varying in size from very small dots to patches or extensive marginal areas. Foliage, especially of peach, becomes usually crinkled. Twigs are usually slender. In vegetable crops in the older leaves bronze and yellowish-brown colours are manifested near the margins. Specks develop along the veins of the leaf. Ultimately the tissue deteriorates and dies. (Plate-3)

Magnesium. The symptoms of magnesium deficiency at first manifest themselves in old leaves. In tobacco the lower leaves are chlorotic but do not show dead spots. The tips and margins of the leaf are turned or cupped upwards. the stalks are slender. In maize leaves a slight yellow streak develops between the parallel veins in the leaves. In acute deficiency these streaked tissues may dry up and die. In small grains the plants are dwarfed and turn yellow. Sometimes leaves exhibit yellowish-green patches. In potato the affected leaves are brittle. The chlorosis in legumes begins at the tip and margins of the lowermost leaf, and progresses between the veins towards the centre of the leaflet. Eventually the tissue between the veins is filled with brown, dead areas. In cotton the lower leaves have purplish-red colour with green veins. In legumes the areas between main veins of the leaves become pale-green, which later turn deep yellow. At a later stage of growth the leaf margins curl downwards accompanied by a gradual yellowing and bronzing from the margin inward. In vegetable crops the symptoms are similar. The chlorosis appears first between leaf veins of new leaves and then spreads to older leaves. The chlorotic areas become brown or transparent and ultimately marked necrosis of affected tissue occurs. In citrus trees the green colour fades in the leaf, parallel to the midrib, and spreads from there. However, the base of leaf usually remains green even in very advanced stages of deficiency of magnesium in the plants. In deciduous fruit trees necrosis occurs as fawn-coloured patches on most mature, large leaves. The affected leaves drop, leaving a tuft or rosette of thin, dark-green leaves at the terminal part of the twigs. (Plate-4)

Calcium. Generally the deficiency symptoms due to calcium starvation are localized in new leaves and in bud leaves of plants. In severe cases the terminal bud dies. In tobacco the young leaves making up the terminal bud first become typically hooked and dieback at tips and margins. The stalk finally dies back. In maize the tips of the unfolding leaves gelatinize and when they dry they stick together. In potato a light green band appears along the margins of the young leaves of the bud. The leaves often have a wrinkled appearance. In cotton calcium deficiency makes the petioles bend

and later collapse. In vegetables the stems grow thick and woody, and the new leaves are chlorotic. The new growth lacks turgidity. In legumes the nodules developed are small and fewer in number. In citrus the green colour fades along the edges of the leaf and this spreads to areas between veins. The symptoms appear first in immature leaves of deciduous fruit trees, especially those at the top which dieback from tips and margins or along the midribs. Later on the twigs also dieback. (Plate-5)

Zinc. Various plant species show different symptoms of zinc deficiency. In tobacco lower leaves are at first involved. They are mottled or chlorotic with spots which rapidly enlarge involving secondary and primary veins in succession. The leaves are thick. They have short internodes. On maize seedlings 'white bud' disease is noticed. It is a type of chlorosis or fading of dark-green colour. These are small white spots of inactive or dead tissue. The leaves of opening buds have white or light yellow colour. Hence he zinc deficiency disease is called 'white bud' disease. Potato plants without zinc form grayish-brown to bronze-coloured irregular spots, usually appearing in the middle of the leaves. The affected tissue sinks and finally dies. Extreme deficiency of zinc manifests in chlorotic conditions and in darker-coloured veins of leaves. It is difficult to distinguish these symptoms under field conditions. In vegetable crops the new leaves have mottled appearance with yellow colour. In acute cases the necrotic or dead areas are found on new leaves. (Plate-6)

Boron. The deficiency symptoms of this nutrient are usually localized on nerve or bud leaves of the plant. In tobacco the young leaves have light green colour at their bases. This is followed by breakdown for this tissue. In old leaves with acute deficiency they show twisted growth. The stalks finally dieback at the terminal bud. In corn the younger leaves are dwarfed. Their tissues are white and the growing tips dead. Under field conditions the plants have weaker ear-shanks and stalks. Their leaves are yellowish in colour. In the potato fields boron-deficiency symptoms occur in the tubers rather than on the veins. the tubers on boiling show much sloughing, are fairly saggy and have a flat flavour. In sand culture devoid of boron, the plants are short and bushy. The growing points are soon killed and the growth of lateral buds is stimulated. The leaves thicken and margins roll upwards. The leaf points and margin of older leaves die prematurely. The tubers, besides being small in size, have a ruptured surface. In cotton the effect is localized to terminal buds which dieback, resulting in multi-branched plant. The young leaves are yellowish-green and flower buds are chlorotic. In vegetables the growing tissues of stems and roots are involved. The new bud leaves and petioles have light colour, are brittle and are often deformed in shape. Rosetting due to short internodes is pronounced at the shoot terminals. The legumes also have resetting at the terminal buds. The buds appear as white or light brown dead tissue. The plants have little flowering. In citrus the deficiency symptoms are localized to new growth. New leaves have water-soaked flecks, which become translucent. The fruits have hard, fumy lumps in the rind. In deciduous trees symptoms appear on terminal tissues of twigs. The young leaves have chlorotic appearance and

are wrinkled. Due to severe deficiency the twigs and spurs show symptoms of dieback. (Plate-7)

Manganese. In this case also the symptoms are localized to terminal buds which remain alive, but the bud leaves are chlorotic with veins light or dark-green. In tobacco the young chlorotic leaves develop dead tissues scattered over the leaf. The smallest veins tend to remain green, which gives chequered effect on the leaves. In oats the 'grey speck' disease has been found associated with manganese deficiency. In potato the terminal buds remain alive, chlorosis of newer tissue occurs and numerous small brown patches develop which in time become more extensive. In cotton the terminal buds remain alive but upper or bud leaves become yellowish-grey or reddish-grey while veins remain green. In vegetables the new leaves become chlorotic while veins remain green. In cereals the leaves turn brown or transparent; this is followed by necrosis of the affected tissues. In legumes the terminal buds remain alive but leaves become light green or yellow with green veins. Later on dead tissues appear on the leaf. Although in citrus the leaves have normal shape and size their veins remain green while the tissue in between becomes light green to grey in colour. (Plate-8)

Iron. The iron-starved plants have short and slender stalk. Their terminal buds remain alive but their new leaves show chlorosis of tissues in between the veins, which themselves remain green. In tobacco the young leaves from the terminal buds show chlorotic appearance. The veins of these leaves remain typically green. In young leaves a slight uniform chlorosis is at first noticed. The margins and veins retain green colour. The leaves become pale-yellow and subsequently white. In vegetable crops the new leaves develop light yellow colour in between the veins. Later on the entire leaf becomes yellow. In legumes the leaves turn yellow with the veins remaining green, and on leaves spots of dead tissues appear, particularly at the margins. These dead tissues, in due course, drop away. In citrus the dying of twigs is common, accompanied by chlorosis of leaf tissues in between the veins. The growth of plants is very much restricted. (Plate-9)

Copper. The terminal buds remain alive but wilting or chlorosis of bud leaves takes place with or without spots of dead tissues. The veins of these leaves remain light or dark-green. In tobacco the young leaves remain permanently wilted. They do not have spotting or marked chlorosis. Deficiency in potato is recognized by the wilting of young leaves and loss of turgor of terminal buds which drop when flower buds are developing. There is no pronounced chlorosis but drying of leaf tips occurs in advanced stages. In vegetables the growth is retarded and leaves lack turgidity. They exhibit chlorosis as if they are bleached. In legumes the young leaves wilt with or without chlorosis. In extreme deficiency there may occur excessive leaf shedding. In citrus the large leaves are frequently malformed, and have a fine network of green veins on a light green background. The fruits have gummy excrescences. (Plate-10)

Sulphur. Generally the terminal bud remains alive. The chlorosis of younger leaves takes place. In tobacco the whole leaf has light green colour; only younger leaves show these symptoms without injury to terminal buds. The symptoms of chlorosis in young leaves of potato develop slowly but growth of plants is materially checked. Similar dwarfing of plants occurs in cotton but the green colour of new leaves does not show any change. In vegetables, the leaves develop yellowish-green colour, and become thick and firm. The stems harden and sometimes become abnormally elongated and spindly. In legumes the younger leaves turn pale-green to yellow, while terminal buds remain alive. The growth of citrus slows down. The new leaves develop very light yellow-green to yellow colour. (Plate-11)

Molybdenum. The deficiency is markedly evident in legumes, particularly in the subterranean clover. Molybdenum-starved plants have yellowish to pale-green colour.

Table – B Prominent nutrient deficiency symptoms in plants

Nutrient	**Colour change in lower leaves**
N	Plant light green, older leaves yellow
P	Plants dark green with purple cast, leaves and plants small
K	Yellowing and scorching along the margin of older leaves
Mg	Older leaves have yellow discolouration between veins-finally reddish purple from edge inward
Zn	Pronounced interveinal chlorosis and bronzing of leaves
Nutrient	**Colour change in upper leaves** (Terminal bud dies)
Ca	Delay in emergence of primary leaves, terminal buds deteriorate
B	Leaves near growing point turn yellow, growth buds appear as white or light brown, with dead tissue.
Nutrient	**Colour change in upper leaves** (Terminal bud remains alive)
S	Leaves including veins turn pale green to yellow, first appearance in young leaves.
Fe	Leaves yellow to almost white, interveinal chlorosis at leaf tip
Mn	Leaves yellowish-gray or reddish, gray with green veins
Cu	Young leaves uniformly pale yellow. May wilt or wither without chlorosis
Mo	Wilting of upper leaves, then chlorosis
Cl	Young leaves wilt and die along margin

CHAPTER 4

SOIL TESTING

Soil testing refers to the chemical analysis of soils and is well recognized as a scientific means for quick characterization of the fertility status of soils and predicting the nutrient requirement of crops. It also includes testing of soils for other properties like texture, structure, pH, Cation Exchange Capacity, water holding capacity, electrical conductivity and parameters for amelioration of chemically deteriorated soils for recommending soil amendments, such as, gypsum for alkali soils and lime for acid soils. **One of the objectives of soil tests is to sort out the nutrient deficient areas from non-deficient ones.** This information is important for determining whether the soils could supply adequate nutrients for optimum crop production or not. As farmers attempt to increase their crop production, one of the questions they ought to ask is whether the addition of fertilizer will increase the yield and whether it will be profitable? Fertilizer use could be aimed at economic optimum yield per hectare. The National interest would be to obtain the maximum yield from the area under cultivation while the farmer's interest would be to obtain profitable yields and not necessarily the maximum yields. Indiscriminate use of fertilizer is not an answer to any one of the problems as this not only increases the cost of crop production but also results in deleterious effects on soil fertility. The concept of balanced nutrition of crops also guides the use of plant nutrients in a definite proportion as required by the crops which is possible only if one knows the available nutrient status of his soils. Soil testing helps in understanding the inherent fertility status of the soils. Further, various factors other than poor soil fertility may also be responsible for poor crop production but soil fertility status assumes a greater importance. Each fertilizer recommendation based on a soil analysis should take into account the soil test value obtained by the accurate soil analysis, the research work conducted on a crop response to fertilizer application in a particular area and the practices and level of management of the concerned farmer. The soil test aimed at soil fertility evaluation with resulting fertilizer recommendation is, therefore, the actual connecting link between the agronomic research and its practical application to the farmers' field.

4.1 Historical background of Soil Testing Service and Fertility status of Indian Soils

The soil testing programme was started in India during the year 1955-56 with the setting-up of 16 soil testing laboratories under the Indo-US Operational Agreement for "Determination of Soil Fertility and Fertilizer Use". In 1965, five of the existing laboratories were strengthened and nine new laboratories were established with a view to serve the Intensive

Agricultural District Programme (IADP) in selected districts. To meet the increasing requirement of soil testing facilities, 25 new soil-testing laboratories were added in 1970. In addition to this, 34 mobile soil testing vans were established under the joint auspices of the Technical Cooperation Mission of USA (TCM), IARI and Government of India to serve the farmers in remote areas and also provide education to the farmers on the benefits of balanced fertilization through group discussions, demonstrations, film shows etc. The idea to create the mobile soil testing facility was to serve the farmers almost at their doorsteps. The capacity of the soil testing laboratories in the intensive agricultural districts was initially created to analyse 30,000 soil samples annually by each laboratory.

The installed capacity of the laboratories varied from 1000 samples/yr/lab (some cases in UP) to 30,000 to 70,000 samples/year in Tamil Nadu. A laboratory with 30,000 samples/year capacity used to be called as a standard laboratory in the initial years of the programme. Out of 354 testing laboratories functioning with an analysing capacity at approx. 4 million soil samples per year during 1981, 90 laboratories each had less than 5000 sample analysing capacity per annum, 142 labs had 6-10,000 samples capacity, 65 labs had a capacity between 11-20,000 samples/year. A total of 47 labs had a capacity of 21-30,000 samples per year per lab and 10 labs had more than 30,000 samples/year/lab. Presently, the thinking is to set-up smaller laboratories with the analysing capacity of 10-12,000 samples/year. Till the year about 1980, the laboratories generally used to analyse soil samples for pH, texture, electrical conductivity, and available N P K. Gypsum and lime requirement were also estimated. After 1980 onwards, micronutrient analysis has also been taken up by the soil testing labs. The process of setting up of soil testing laboratories has continued, year after year, with the financial support from Government of India. The State Government and the Fertilizer industry is also setting-up the soil testing laboratories.

Presently, there are 661 soil testing laboratories including 120 mobile vans operating in 608 districts of the country with an annual sample analysing capacity of 7.2 million. State-wise position of the capacity is at great variance from one State to another. Among major States, in MP, Chattishgarh, Orissa, Jharkhand and Assam, the number of soil testing laboratories is less than the number of districts in each State. In other States, such as Rajasthan, Himachal Pradesh, Uttarakhand, Bihar and West Bengal, the number of labs are just about equal to the number of districts while the remaining States have larger number of labs than the number of districts.

Under the National Project on Management of Soil Health and Fertility, 500 stationery and 250 mobile soil testing labs are proposed to be set up during XI Plan. This will increase the analysing capacity by 7.5 million soil samples per year by the end of XI Plan when these labs become fully functional. Total sample analysing capacity will become 14.7 million per year. However, still there will be a big gap between the projected

capacity and the number of farm holding being over 110 million in the country which ideally require sampling, analysis and fertilizer use recommendation annually and which if not possible, at least after a gap of three years period as a practical measure.

A list of equipment approved for setting up of stationery and mobile soil testing labs along with estimated approved cost and funding pattern under the National Project is given at **Annexure-1** and the requirement of staff is at **Annexure-2.**

In view of the limited analysing capacity, a strategy which is scientifically justified and practical has been suggested which is as under:-

i) Continue to recommend fertilizer use on individual sample basis, in case samples are received directly from the farmers.
ii) Recommend fertilizer use based on 'Composite Soil Sample Analysis'.
iii) Recommend fertilizer use on the basis of village / block level fertility map.

The practice being followed in some states to make fertilizer use recommendation for the State as a whole, twice in a year, i.e. during kharif and rabi conferences is not expected to give beneficial results.

Soil nutrient as an index of soil fertility

Generally, the soil testing laboratories use organic carbon as an index of available N, Olsen's and Bray's method for available P and neutral normal ammonium acetate for K. In semiarid tropics, nitrate nitrogen is also used as an index of available N in soil.

Available nutrient status in the soils is generally classified as low, medium and high which are generally followed at the National level and are as follows **(Table-1)**.

Table 1

S N.	Soil Nutrients	Soil Fertility Ratings		
		Low	Medium	High
1	Organic carbon as a measure of available Nitrogen (%)	< 0.5	0.5-0.75	>0.75
2	Available N as per alkaline permanganate method (kg/ha)	<280	280-560	>560
3	Available P by Olsen's method (kg/ha) in Alkaline soil	<10	10-24.6	>24.6
4	Available K by Neutral N, ammonia acetate method (kg/ha)	<108	108-280	>280

The Soil Test Crop Correlation (STCR) Projects of ICAR including one for micro nutrients which were initiated in 1967 and many State Agricultural Universities (SAUs) are engaged in refining the limits and categories of soil fertility classification. It is important to note that over the decades, only 3 levels of available N, P, K as determined with the testing method as indicated above, continue to be most operative. In many situations, the testing method and the limit fixed for available K is not found to be satisfactory while nitrogen content continues to be represented by organic carbon which at times has no direct relation with soil available nitrogen. The broad classifications for soil nutrient Status is too general and may be only indicative for national level appreciation of soil fertility status and not for the benefit of an individual farmer. This classification needs refinement.

Some states like West Bengal, Maharashtra and some others are following 6 classes of the nutrient status as given in **Table 2 & 3** below:

West Bengal :

Table 2

Soil Fertility Level	Organic Carbon (%)	Available P_2O_5 kg/ha	Available K_2O kg/ha
Very high	> 1.0	> 115	> 360
High	0.81-1.0	93-115	301-360
Medium	0.61-0.80	71-92	241-300
Medium Low	0.41-0.60	46-70	181-240
Low	0.21-0.40	23-45	121-180
Very Low	<0.21	<23	<121

Maharashtra :

Table 3

Soil Fertility Level	Organic Carbon (%)	Available N kg/ha	Available P_2O_5 kg/ha	Available K_2O kg/ha
Very high	> 1.00	> 700	> 80.0	> 360
High	0.81- 1.00	561 – 700	64 – 80	301 – 360
Medium	0.61-0.80	421 – 560	48 – 64	241 – 300
Medium Low	0.41-0.60	281 – 420	32 – 48	181 – 240
Low	0.21-0.40	141 – 280	16 – 32	121 – 180
Very Low	< 0.20	< 140	< 16.0	< 120

Source : Tandon H.L.S.(2005)

In some states 5 levels of soil nutrient ratings are followed such as very low, low, medium, high and very high.

Micronutrient deficiencies also started becoming critical, beginning with the intensification of agriculture and using mostly high analysis chemical fertilizers. Thus micro nutrient testing facilities were also required to be created in the soil testing labs. Under Indo-UK bilateral programme on strengthening the soil testing facilities in India during 1980, 20 atomic absorption spectrophotometers were supplied to the soil testing labs in 20

states. Apart from this, some state governments have also provided this equipment to their soil testing labs under their state plans. Government of India is strengthening the soil testing labs by providing funds for equipment, including atomic absorption spectrophotometers. Thus, the testing of micro-nutrient also began by the soil testing labs. State Agricultural Universities and coordinated ICAR scheme on micro nutrient in soils and Plants are delineating micro nutrient deficient areas and setting standards for micronutrient sufficiency and deficiency in soils and plants.

4.2. Nutrient indexing and preparation of soil fertility maps

Capacity of the soil testing labs to analyse soil samples has always been inadequate and it continues to be so being at 7.2 million samples / annum as against more than 110 million farm holdings in the country out of which about 35 million are irrigated and partially irrigated holdings where fertilizer use is being practiced at various levels and these farmers need advise on fertilizer use. Thus the need for expanding the soil testing service is well recognized. Apart from expanding the capacity, simultaneously, the available soil test data are interpreted to work out nutrient index and prepare fertility maps and make generalized fertilizer use recommendations, in the absence of a specific soil test for all and individual farms.

Ramamoorthy and Bajaj (1969) prepared soil fertility maps showing available nitrogen, phosphorus and potassium status of Indian soil. These maps were based on 1.3 million soil samples analysed by the end of 1967 by 31 soil testing laboratories. Parker's (1951) method of calculating Nutrient Index (NI) values was used to indicate fertility status of soils for the purpose of mapping. Even a minimum of 500 soil samples were taken for working out nutrient index for a given area since the total number of soil analyses data were rather small at the country level.

The following equation is used to calculate Nutrient Index Value:-

$$\textbf{Nutrient Index} = \frac{(Nl \times 1) + (Nm \times 2) + (Nh \times 3)}{Nt}$$

Nt = Total number of samples analysed for a nutrient in any given area.

Nl = Number of samples falling in low category of nutrient status.

Nm = Number of samples falling in medium category of nutrient status.

Nh = Number of samples falling in high category of nutrient status.

Separate indices are calculated for different nutrients like nitrogen, phosphorus and potassium.

Parker had classified the nutrient index values less than 1.5 as the indicative of low nutrient status and between 1.5 to 2.5 as medium while higher than 2.5 as high nutrient status. Ramamoorthy and Bajaj had categorised these values as less than 1.7 being indicative of low fertility

status, between 1.71 to 2.33 as medium and more than 2.33 to classify as high.

Limits of soil test values used were as given in the **Table-1 above**.

Based on the information available from 224 districts for N analysis, 226 districts for P analysis and 184 districts for K, the districts falling into different levels of N, P, K status were reported to be as follows:

Nutrient	Districts falling into		
	Low	Medium	High Status
Nitrogen	117	97	10
Phosphorus	106	110	10
Potash	36	98	50

The soil test information is thus used to depict nutrient requirement of crops on larger areas as per the soil fertility maps. This information is also useful in assessing the fertilizer use efficiency in a given soil plant situation.

Ghosh and Hasan (1980) had prepared soil fertility maps for N, P and K on the basis of 9.2 million soil samples analysed in 250 soil testing laboratories covering 365 districts in the country. The reported status of soil fertility maps was as under:

Nutrient	% samples in the districts falling into		
	Low	Medium	High Category
Nitrogen (% Org. Carbon)	62.5	32.6	4.9
Phosphorus	46.3	51.5	2.2
Potash	20.0	42.0	38.0

Motsara et. al.(1982) had worked out nutrient index for 307 districts on the basis of 5.0 million soil analysis data collected from 300 soil testing laboratories. The soil nutrient status classified as low, medium and high was correlated with the fertilizers being consumed in these districts. It was observed that there was no relationship with actual fertilizer use in these districts and the level of soil fertility as depicted from nutrient index values. This reflects the inadequacy of the quality of soil analysis and or a possibility of other factors affecting the fertilizer use.

However, the utility of preparing soil fertility maps continues to be recognized in view of the fact that such maps can be used for a general planning of fertilizer supply / use on area basis, in the absence of a specific soil analysis for each and every farm holding.

Motsara (2002) had computed nutrient index values and prepared a soil fertility map for N, P and K using 3.65 million soil analysis data collected from 533 soil testing labs representing 450 districts in the country. The level of soil fertility emerged was as follows:

Nutrient	Percent of Districts as		
	Low	Medium	High Fertility Soils
Nitrogen	63.0	26.0	11.0
Phosphorus	42.0	38.0	20.0
Potash	13.0	37.0	50.0

If the maps are prepared for the block / village level, their utility for making fertilizer use recommendation widens.

The soil testing capacity has continuously been increasing in the country. By 1998-99 there were 396 stationary soil testing labs and 118 mobile vans out of which fertilizer industry had set up 36 static and 20 mobile vans. The analysing capacity in the country was 6.4 million soil samples of which approx. 75% capacity was utilized by analysing app. 4.8 million samples.

By 2004-05 the number of soil testing labs had increased to 551 with a total analysing capacity of 6.7 million soil samples. Zone wise and agency wise status of the facility was as under:

Zone	No. of labs set up by States		By Fertilizer Industry		Total	
	Static	Mobile	Static	Mobile*	Total Labs	Total Analysing capacity Per year (000 Nos.)
East zone	80	23	--	--	103	740.0
North zone	163	36	5	3	207	2775.0
South zone	78	33	8	2	121	2157.0
West zone	78	21	14	7	120	1075.0
Total	**399**	**113**	**27**	**12**	**551**	**6747.0**

Latest (2008-09) number of soil testing labs (661) and the analysing capacity (7.2 million soil sample / year) is given in the **Table 4** below:-

Table 4

Zone	No. of labs set up by States		By Fertilizer Industry		Total	
	Static	Mobile*	Static	Mobile	Total Labs	Total Analysing capacity Per year (000 Nos.)
East zone	67	8	0	0	75	485.0
North East zone	20	11	0	0	31	223.0
North zone	194	29	4	5	232	3288.0
West Zone	105	24	10	8	147	1286.0
South Zone	136	33	5	2	176	1919.0
Total	**522**	**105**	**19**	**15**	**661**	**7201.0**

* Some of MSTLs have gone into dis-use.

State wise information is given in **Annexure-3** indicating number of districts, labs in each State along with analysing capacity.

4.3. Soil testing for micro and secondary nutrients:

To ensure that deficient micro and secondary nutrients are supplied to the crops, a systematic delineation study has been initiated through All India Coordinated Research Project of Micronutrients in Soils & Plants since renamed as Micro, Secondary and Pollutant Elements in Soils & Plants. Testing methods for assessing available micronutrients have been standardized and are being used in soil testing laboratories. The amounts of micronutrients that can be removed yearly with the normal crop yields vary from element to element and crop to crop.

In a typical example, it can be noted that rice crop removes 40 gm Zinc, 153 gm Fe, 675 gm Mn, 189 gm Cu, 15 gm B and 2 gm Mo while removal by wheat crop is 56 g zinc, 624 g Fe, 70 g Mn, 24g cu, 489 B and 2 g Mo/ha (Tandon, 1989). The total amounts of nutrient elements present in the soils generally far exceed the requirements of crops and it is the availability of micronutrients in the soil that matters and is primarily dependent on their solubility as determined by various soil factors. The form of nutrients and their mobility in the plant are given in **Table 5** below:

Table 5 The form of nutrients absorbed and the relative mobility of elements in plants

Element	Form Absorbed	Mobility in Plant
Sulphur	SO4 =, SO2 *	Relatively immobile
Calcium	Ca++	Relatively immobile
Magnesium	Mg++	Mobile
Boron	H3BO3	Relatively immobile
Copper	CU+, Cu++	Relatively immobile but mobile under sufficiency conditions
Iron	Fe++	Relatively immobile
Manganese	Mn++	Relatively immobile
Molybdenum	MoO_4	Moderately mobile
Zinc	Zn++	Low mobility
Chlorine	Cl-	mobile

- Plants can absorb SO2 gas directly from the atmosphere.

Suitable soil extractants have, therefore, been developed for various micronutrients to predict the available forms of elements in soils. In India, zinc is the most widely reported deficient nutrient element. Other micronutrients like copper, manganese, iron, boron, molybdenum and secondary nutrients like sulphur are also becoming deficient. Suitable testing methods are being standardized under the All India Coordinated Research Project on Micronutrients. Generally accepted critical limits in soils and plants and the soil test methods are given in **Table 6 & 7** below:

Table 6 : Usually advocated soil tests and critical levels of nutrients in soils and plants

Element	Soil Test Method	Critical level in soil	Critical level in plant
Sulphur	Hot water, $CaCl_2$ or phosphate	Usual 10 ppm range 8 - 30	< 0.15 – 0.2%
Calcium	Ammonium acetate	<25% of CEC or < 1.5 me Ca/100 g	<0.2%
Magnesium	Ammonium Acetate	<4% of CEC or <1 me Mg/100g	<0.1 – 0.2%
Zinc	DTPA	0.6 (0.4 – 1.2) ppm	<15 – 20 ppm
Manganese	DTPA	2 ppm	<20ppm (10-30)
Copper	DTPA or Ammonium acetate	0.2 pm	<4 ppm(3-10)
Iron	DTPA Ammonium acetate	2.5 – 4.5 ppm 2 ppm	<50 ppm(25– 80)
Boron	Hot water	0.5 ppm	<20 ppm
Molybdenum	Ammonium oxalate	0.2 ppm	<0.1 ppm

Crop/variety differ in their nutrient requirement and thus have different levels of critical limits below the content of which, the plant starts suffering due to the shortage of the nutrient and start showing deficiency symptoms. In the table given below, the critical levels of micro and secondary nutrient content in major soil types and in important cereal crops are shown.

Table 7: Critical levels of secondary and micronutrients in different soils and crops

Nutrient	Soil	Crop	Critical Level	Method
S	Alluvial	Rice Wheat	<10 mg kg -1 <10 mg kg -1	0.15 CaCl2 0.15 CaCl2
Ca	Alluvial	Rice Wheat	<1.0 c mol kg -1 <1.0 c mol kg -1	NH4OAC extractable NH4OAC extractable
Mg	Alluvial	Rice Wheat	<1.0 c mol kg -1 <1.0 c mol kg -1	NH4OAC extractable NH4OAC extractable
Zinc	Red and black	Rice Wheat Maize Sorghum	0.45 – 2.00 ppm 0.46 – 0.60 ppm 1.00 – 1.20 ppm 1.00 – 1.20 ppm	DTPA extraction DTPA extraction DTPA extraction DTPA extraction
	Red	Rice Maize	0.60 – 1.00 ppm 0.65 – 0.80 ppm	DTPA extraction DTPA extraction
	Black	Rice Wheat	0.84 – 1.30 ppm 0.54 ppm	DTPA extraction DTPA extraction
	Alluvial	Rice Wheat Maize	0.38 – 0.90 ppm 0.40 – 0.80 ppm 0.54 – 1.00 ppm	DTPA extraction DTPA extraction DTPA extraction
	Tarai & river belt	Rice	0.78 – 0.95 ppm	DTPA extraction
Iron	Alluvial	Wheat Sorghum	3.20 ppm 4.40 ppm	DTPA extraction DTPA extraction
	Black	Sorghum Maize	6.00 ppm 4.00 ppm	DTPA extraction DTPA extraction
Manganes	Alluvial	Wheat	3.50 ppm	DTPA extraction
Boron		Rice Wheat	0.50 ppm 0.50 ppm	DTPA extraction Hot water
Molybdenum		Rice and wheat	0.20 ppm	Ammonium oxalate

Source: Tandon, HSL, Secondary and micronutrient recommendation for soil and crops – a guide book, FDCO, New Delhi- pg 1-59 (1989)

Delineation micro-nutrient deficient areas

Under coordinated research project on "Micro-Nutrients"(1988), 2.51 lakh surface soil samples were analysed from 20 sites. As per the critical limits applicable to different micronutrient, it was observed that 48% of the samples were deficient in zinc, 33% in boron, 12% in iron, 13% in Mo, 5% in Mn, 3% in Cu. State-wise information is given in **Table 8 :**

Table 8 : Extent of micronutrient deficiency in Indian Soil

Name of State/ Union Territory	No. of samples	Percent Samples deficient (PSD)					
		Zn	Cu	Fe	Mn	B	Mo
Andhra Pradesh	8158	49	<1	3	1	-	-
Assam	12166	34	<1	2	20	-	-
Bihar	19214	54	3	6	2	38	-
Delhi	201	20	-	-	-	-	-
Gujarat	30152	24	4	8	4	2	10
Haryana	21648	60	2	20	4	0	28
Himachal Pradesh	155	42	0	27	5	-	-
J&K	93	12	-	-	-	-	-
Karnataka	27860	73	5	35	17	32	-
Kerala	650	34	3	<1	0	-	-
Madhya Pradesh	32867	44	<1	7	1	22	18
Maharashtra	515	86	0	24	0	-	-
Meghalaya	95	57	2	0	23	-	-
Orissa	16040	54	-	0	0	-	-
Pondicherry	4108	8	4	2	3	-	-
Punjab	16483	48	1	14	2	13	-
Rajasthan	183	21	-	-	-	-	-
Tamil Nadu	28087	58	6	17	6	21	-
Uttar Pradesh	26126	46	1	6	3	24	-
West Bengal	6547	36	0	0	3	68	-
All India	**251547**	**48**	**3**	**12**	**5**	**33**	**13**

The deficient areas were also identified by crop response in field trials. Crop response to the **application of zinc** (**Table 9**) and **Fe, Mn and B** (**Table 10**) and **Sulphur(Table 11**) show the need for the application of these micronutrients.

Table 9: Response of crops to zinc application

Crop	No. of Expt.	Range of Response t ha^{-1}		Average response t ha^{-1}
		Individual Expt.	Mean of Expt.	
Wheat	2447	0.00-4.70	0.01-1.47	0.42
Rice	1652	0.00-5.47	0.14-1.27	0.54
Maize	280	0.01-3.09	0.11-1.37	0.47
Barley	17	0.11-1.18	0.49-0.73	0.55
Sorghum	83	0.07-1.35	0.21-0.65	0.36
Groundnut	83	0.04-0.60	0.21-0.47	0.32
Soybean	12	0.08-0.69	0.16-0.39	0.36
Mustard	11	0.02-0.34	0.14-0.26	0.27
Linseed	5	0.12-0.21	0.15-0.20	0.16
Sunflower	8	0.01-0.67	0.15-0.20	0.24
Seasamum	6	0.08-0.15	0.08-0.15	0.11
Potato	45	1.10-7.60	2.40-3.90	2.96
Sugarcane	6	8.00-4.30	1.72-2.40	37.70

Source: Micronutrient Project

Table10: Response of crops to Fe, Mn and Boron application

Crop	No. of Expt.	Range of Response t ha^{-1}	
		Range	Mean
Iron			
Wheat	81	0.0-2.50	0.82
Rice	31	0.20-4.40	1.39
Maize	2	-	1.04
Sorghum	23	0.03-2.9	0.60
Chickpea	7	0.05-0.82	0.33
Groundnut	10	0.05-0.70	0.89
Sunflower	3	0.46-0.80	0.55
Soybean	3	0.21-1.00	0.34
Potato	37	1.1-6.90	3.40
Manganese			
Wheat	69	0.0-3.78	0.64
Rice	110	0.40-1.78	0.49
Sorghum	5	0.29-0.51	0.83
Groundnut	1	-	0.11
Sunflower	1	0.40-0.70	0.55
Soybean	2	0.03-1.03	0.31
Potato	35	1.00-3.90	1.90
Boron			
Wheat	35	0.03-1.19	0.39
Rice	107	0.00-1.67	0.32
Maize	5	0.17-1.05	0.57
Chickpea	7	0.09-0.90	0.35
Black gram	2	0.04-0.35	0.17
Lentil	4	0.04-0.49	0.24

Pigeonpea	2	0.03-0.32	0.19
Groundnut	11	0.05-0.42	0.21
Linseed	2	0.11-0.14	0.12
Mustard	2	0.21-0.31	0.26
Onion	4	3.87-7.30	4.47
Sweet Potato	2	0.67-7.0	4.42
Cotton	2	0.06-0.35	0.21

Table 11 : Crop response to sulphur application :

Crop	No. of Expts.	Yield without S q/ha	Mean Increase in yield due to sulphur	%age average response
Wheat	6	26.1	0.7	27
Rice	3	36.0	13.1	36
Maize	1	14.3	4.7	36
Groundnut	8	13.7	2.2	15
Potato	3	119.0	35	30
Source : Tandon(1989) – Secondary and Micronutrient Recommendations for Soils and Crops				
Mustard	57	13.66	4.67	42.00
Groundnut	23	17.85	5.66	31.7
Soyabean	8	14.26	3.61	25.3
Sunflower	6	12.33	2.49	20.2
Source: Hegde DM and Suddakar Basu S.N.(2009) Indian Journal Fertilizer. Vol 5(4), pp.29-39				

Soil test data for contents of available N, P, K and micronutrients in soils show that these nutrients vary from low to medium in large areas. The data from field trials on crop response clearly bring out the benefits of the application of these plant nutrients in varying quantities and proportions.

Some typical data from all India projects presented in the foregoing pages generally brings out the existence of wide spread deficiencies of essential plant nutrients in Indian soils. This clearly shows that there is well established need to test the soils to determine specific nutrient requirement of crops or to evaluate soil fertility status.

4.4. Soil Fertility Evaluation Techniques:

Basic aim of the soil testing is to assess the fertility status of the soils so as the recommendations for the fertilizer use and soil amendments, if necessary, can be made. There are, however, other techniques which can be employed for soil fertility evaluation. Different techniques can be used alone or in combination with each other but the soil testing is still most commonly employed technique (Dev, 1997). Different techniques are listed and briefly described below:-

I. Nutrient deficiency symptoms
II. Biological tests
III. Plant analysis

I. Nutrient Deficiency Symptoms:

The appearance of a plant has long been a clue to its nourishment or lack of it. When a plant badly needs a certain plant food, it shows starvation signs called deficiency symptoms or hunger signs. These symptoms are nutrient specific and show different patterns in crops for different essential nutrients. It is a good tool to detect deficiencies of nutrients in the field. However, it requires learning to identify nutrient deficiencies. In applying this technique, one must develop diagnostic proficiency through practice and close observation. It may be pointed out that deficiency symptoms in many cases are not always clearly defined and in cases, the symptoms can be common to other causes or may be masked by other nutrients. Some typical examples are given below:

- N deficiency can be confused with S deficiency.
- Calcium deficiency can be confused with B deficiency
- Fe deficiency can be confused with Mn deficiency
- Leaf stripe disease of oat can be confused with Mn deficiency
- Effect of virus, little leaf etc. can be confused with zinc deficiency, B deficiency.
- Brown streak disease of rice can be confused with Zn deficiency.

Deficiency symptoms always indicate severe starvation and therefore the crop may have suffered before the deficiency symptoms appear. Many crops start losing yields well before deficiency signs start showing. This yield limiting condition is called Hidden Hunger which refers to a situation in which a crop needs more of a given nutrient and yet has not shown any deficiency symptom. The nutrient content is above the deficiency symptom zone but still considerably below that needed for optimum crop production. In this case, significant responses can be obtained with application of nutrients even though no recognizable symptoms have appeared. To over-come such conditions, it is always recommended to confirm deficiency problem with other diagnostic techniques and most common is soil testing. Nutrient wise deficiency symptoms are described in detail in **Chapter 3.**

II. Biological Tests

(a) Field Tests: The field plot technique essentially measures the crop response to nutrients. In this, specific treatments are selected, randomly assigned to an area of land, which is representative of the conditions. Several replications are used to obtain more reliable results and to account for variations in soil. Field experiments are essential in establishing the equation used to provide fertilizers recommendations

that will optimise crop yield, maximise profitability and minimise the environmental impact of nutrient use.

When large numbers of tests are conducted on soils that are well characterized, recommendations can be extrapolated to other soils with similar characteristics. Field tests are expensive and time-consuming; however, they are valuable tools and are widely used by scientists. They are used in conjunction with laboratory and greenhouse studies in calibration of soil and plant tests.

(b) Greenhouse tests: The greenhouse techniques utilize small quantities of soil to quantify the nutrient-supplying power of a soil. Generally, soils are collected to represent a wide range of soil chemical and physical properties that contribute to the variation in availability for a specific nutrient. Selected treatments are applied to the soils and a crop is planted that is sensitive to the specific nutrient being evaluated. Crop response to the treatments can then be determined by measuring total plant yield and nutrient content.

(c) Laboratory tests: Neubauer seedling method – The Neubauer technique is based on the uptake of nutrients by a large number of plants grown on a small amount of soil. The roots thoroughly penetrate the soil, exhausting the available nutrient supply within a short time. The total nutrients removed are quantified and tables are established to give the minimum values of macro and micronutrients available for satisfactory yields of various crops.

(d) Microbiological methods: In the absence of nutrients, certain micro-organisms exhibit behaviour similar to that of higher plants. For example, growth of Azotobacter or Aspergillus niger reflects nutrient deficiency in the soil. The soil is rated from very deficient to not deficient in the respective elements, depending on the amount of colony growth. In comparison with methods that utilize higher plants, microbiological methods are rapid, simple and require little space. These quick laboratory tests are not in common use in India.

III. Plant Analysis

This involve two approaches. One is analysis of plant in the laboratory and the other is tissue test on fresh tissue in the field. The basis on plant analysis is that amount of a given nutrient in a plant is directly related to availability of the nutrient in the soil. Plant analysis is used to:

- Confirm a diagnosis made from visible symptoms;
- Identify hidden hunger where no symptoms appear;
- Locate soil areas where deficiencies of one or more nutrients occur;
- Determine whether applied nutrients have entered the plant;
- Learn about interactions among various nutrients;

- Study the internal functioning of nutrients in plants;
- Suggest additional tests or studies in identifying a crop production problem.

Plant Analysis is generally not done in the soil testing laboratories. However, relevant details of plant tissue sampling guidelines for major crops are given in Annexure-4.

Plant analysis is used more for fruit and vegetable crops. Because of the perennial nature of the fruit crops and their extensive root systems, plant analysis is especially suitable for determining their nutrient status. As more is known about plant nutrition and nutrient requirements throughout the season, and as application of nutrients through irrigation systems is possible, plant analysis assumes greater importance. Also, to produce high yields, it is helpful in monitoring the plant through its growing season. It is becoming more useful for row crops and forage crops.

The critical nutrient concentration (CNC) is used in interpreting plant analysis results and diagnosing nutritional problems. CNC is the level of a nutrient below which crop yield or quality is unsatisfactory. In India, a great deal of information has been generated on critical nutrient level in crops especially for micronutrients and sulphur. **The demand for this service should increase in India in future as research emphasizes opportunities to manage nutrient availability during the growing season.**

An important phase of plant analysis is sample collection. Plant composition varies with age, the portion of the plant sampled, the condition of the plant, the variety, the weather and other factors. Therefore, it is necessary to follow proper sampling instructions.

For plant analysis, a typical plant part is selected which indicates the nutrient status with crop to determine whether the crop needs fertilization. The optimum values are pre-standardized so as to make recommendation after sample analysis.

4.5. Soil Testing & Balanced Fertilization

Soil testing and its objectives

Soil testing has long been accepted as a unique tool for rational fertilizer use. In fact, however, soil testing can act as a watchdog to safeguard soil quality as a whole. Thus, the major objectives of soil testing are:

i) To assess the soil fertility status and recommend suitable and economic nutrient doses through chemical fertilizers and organic manure for different corps and cropping systems.

ii) To identify the type and degree of degradation problems/ abnormalities like soil acidity, salinity and sodicity etc. and to suggest effective remedial measures.
iii) To generate data for compilation of soil fertility maps.
iv) To study soil pollution-related aspects and to advise preventive as well as remedial measures for a safe food-chain.
v) To make continued efforts for improving the science of wholesome analysis and interpretation of the test data for more meaningful use of this tool for soil care and crop production.

Balanced fertilization

Balanced fertilization does not mean a certain definite proportion of Nitrogen, Phosphorus and Potash (or other nutrients) to be added in the form of fertilizers and organic manures but it has to take into account the availability of nutrients already present in the soil, crop requirement and other factors.

Generally referred 4:2:1 NPK use as a desired ratio does not substitute the need and importance of actually working out the nutrient deficiency in the soil and addition of required nutrient through fertilizers and manures to meet the crop need. This ratio actually relates to the general fertilizer use recommendation started for the two major cereal crops, i.e. rice and wheat as 120 : 80 : 40 kg NPK per hectare (4 : 2 : 1). Till the beginning of green revolution era, farmers have been traditionally using only nitrogen as the first nutrient being almost universally deficient in Indian soils which also showed conspicuous response to its application. Thus, an emphasis on a particular ratio (4 : 2 : 1) has helped in increasing the use of P&K which was necessary to be used over the application of N when high yielding varieties were introduced.

Balanced fertilization should also take into account the crop removal of nutrient, crop species to be sown, farmers' investment ability, soil moisture regimes, weed control, soil salinity, alkalinity, physical environment, microbiological conditions of the soils which determine the status of available nutrients in soil and cropping sequence etc. Von.Uexkule & Mutert (1992) observed that soil testing which is deployed to assess the soil fertility is not static but a dynamic concept. It should not mean that every time a crop is grown, all the nutrients should be applied in a particular proportion, rather fertilizer application should be tailored to the crop needs, keeping in view the capacity of the soils. The balanced fertilizer should aim at:

I. Increasing crop yield and quality.
II. Increasing farm income
III. Correction of inherent soil nutrient deficiency.
IV. Maintaining or improving soil fertility
V. Avoiding damage to the environment

VI. Restoring – fertility and productivity of the land that may have been degraded by exploitive activities in the past.

The above observation was elaborated by Tandon and Kimmo (1993) suggesting that 'Balanced fertilizer use' or rather balanced crop nutrition ensures an optimum supply of all essential nutrients, it promotes synergetic interactions and keeps antagonist interactions out of crop production system. It discourages lopsided application of any nutrient or over-fertilization. The exact schedules to be followed are invariably based on soil testing, field trials and nutrient balances for specific soil-crop situation.

Balanced fertilization implies (i) the minimum supply of nutrients from any source, (ii) which is at the same time sufficient to meet the requirement of crop and maintain soil fertility, (iii) it implies the efficient use of plant nutrients, which is achieved by following research based site-specific recommendations and adopting best agriculture practices, (iv) it recognizes the existence of natural soil processes, which makes certain losses unavoidable.

In principle, the key to balanced fertilization appears to lie in a system of soil-crop management that would ensure efficient use of fertilizer nutrients and maintain crop yield and soil productivity. The balanced fertilizer would thus essentially mean rational use of fertilizers and organic manures for supply of nutrients for agricultural production in such a manner that would ensure:

I. Efficiency of fertilizer use which results in saving of cost.
II. Harnessing of best possible positive and synergistic interactions among various other factors of production i.e., seed, water, agro-chemicals etc.
III. Least adverse effect on environment by minimizing nutrient losses.
IV. Maintain soil productivity and profitability of crop production.
V. Sustaining high yield commensurate with biological potential of crop variety under the unique soil, climate and agro-ecological set up.
VI. The soil-crop management system that would ensure efficient use of fertilizer nutrient and maintain crop yield and soil productivity will be nothing but balanced fertilization. It must be eventually based on the concept of Integrated Nutrient Management for a Cropping System which is the only viable strategy for accelerated and enhanced use of fertilizers with matching adoption of organic manures and bio-fertilizers so that productivity is maintained for a sustainable agriculture.
VII. The outcome of AICRP on 'long-term fertilizer experiments' can be viewed as an example to see that the over the last few decades continuous use of nitrogenous fertilizer alone produced the highest decline in yields at almost all the centres barring a few and had deleterious effect on long term fertility and sustainability in particular, showing deficiency of other major and micro nutrients.

Even in NPK fertilized system, the deficiencies of micro-nutrients and secondary nutrients have become yield limiting factors after a number of years and their application becomes necessary to sustain high yield level. In almost all the long-term fertilizer experiments, balanced and integrated use of optimal dose of NPK and FYM gave enhanced and more sustainable yields. As a policy, govt. and ICAR are recommending soil test based balanced and integrated fertilizer use involving organics namely FYM, compost, vermi-compost, green manures, bio-fertilizers etc. to reduce the excessive use of fertilizers and environment protection.

It would thus be seen that a scientifically sound soil test methodology forms the key for ensuring a successful, efficient, economic and balanced use of fertilizers in agriculture.

A balance use of fertilizers is possible only when the soil available nutrient content is assessed followed by the crop requirement of the nutrients. Thus, formulating a recommendation about the quantities and types of nutrients that are in short supply in the soil and need to be added in proper balance and as per their crop need, through external source like chemical fertilizers and organic manures. Soil nutrient availability can be assessed through soil testing only. Hence, the importance of soil testing can be appreciated in ensuring balanced fertilization for a profitable and sustainable crop production.

In view of the importance of balanced fertilization in increasing crop production, the following recommendations were made in the FAI's Annual (2009) International Seminar on Fertilizer Policy for Sustainable Agriculture which is widely attended by the State Governments, fertilizer industries and experts.

Recommendation No.9 : It relates to soil health and it is stated that "Soil specific nutrient management, which is considered as Best Fertilizer Management Practice (BFMPs) needs to be promoted to improve soil health and crop productivity."

Recommendation No.11: "There is an imminent threat to India's food security arising out of imbalanced use of fertilizers, absence of component-wise locations / crop specific fertilizer recommendations and limited use of organic fertilizers, wide spread deficiencies of secondary and micro-nutrients, diminishing effect of crop response to fertilizers, limited irrigation resources, stagnation of area under cultivation and stagnating food grain productivity."

Recommendation No.12 : It is stated that "Inadequate and unreliable soil testing facilities, poor awareness of farmers about balanced plant nutrition and lack of appropriate policy are the major constraints in adoption of Fertilizer Best Management Practices."

The wording of recommendation (No.12) may be considered too critical but the fact is realized that the soil testing service has not made the desired impact and farmers have not yet been able to adopt it in large numbers. While at the same time, the importance of the soil test based balanced fertilizer use is being advocated by the Government, Fertilizer Industry and others concerned.

The quality of the soil testing programme, therefore, needs to be improved in all its aspects.

4.6 Soil Testing procedures

Procedurally, the soil testing programme can be divided into the following Components:

4.6.1 Setting up of a soil testing laboratory - Basics of analytical Laboratory

In chemical laboratories, the use of acids, alkalis and some hazardous and explosive chemicals is inescapable. Apart from this, some chemical reactions during the process of analysis may release toxic gases and if not handled well, may cause explosion. Inflammable gases are also used as a fuel/heating source. Thus, safe working in a chemical laboratory needs special care, both in terms of design and construction of the laboratory building, and handling and use of chemicals. For chemical operations special chambers also need to be provided.

Air temperature of the laboratory and working rooms should be maintained at a constant level between 20-25^0 C. Humidity should be kept at about 50% soil samples are often affected by the temperature and humidity. Even some chemical operations get influenced by the temperature. Hence, maintenance of temperature and humidity as specified is critical.

Proper air circulation is also important so as hazardous and toxic fumes and gases do not stay in the laboratory for long. The release of gases and fumes in some specific analytical operation are controlled through fume hood or trapped in acidic/alkaline solutions and washed through flowing water. Maintenance of clean and hygienic environment in the laboratory is essential for the good health of the workers.

Care is needed to be taken to store acids and hazardous chemicals in separate and safe racks. An inventory of all the equipment, chemicals, glassware and miscellaneous items in a laboratory should be maintained. A format has been suggested for this purpose (**Annexure- 5**). A list of commonly required equipment, chemicals and glass wares is at **Annexure-6.** Specifications of heavy duty auto analytical equipment are given in **Annexure – 6 A**. Automation of analytical procedures of some of the equipments is given in **Annexure- 6 B.** A safe laboratory building should

have suitable separate rooms for different purposes and for performing different operations as described below, and accordingly a floor plan is given in **Annexure-7.** Grades of standard chemicals and glass wares are given at **Annexure-8.**

Room 1. Reception/sample receipt/dispatch of reports.
Room 2. Sample storage and preparation room.
Room 3. Nitrogen digestion/distillation room (with fume hood for digestion).
Room 4. Instrument room to house:

- Atomic absorption spectrophotometer (AAS)
- Flame photometer
- Spectrophotometer
- pH meter, conductivity meter
- Ovens
- Centrifuge
- Balances
- Water still

Room 5. Chemical analysis room:

- To prepare reagents, chemicals and to carry out their standardization.
- To carry out extraction of soil samples with appropriate chemicals/reagents.
- To carry out titration, colour development, filtration, etc.
- All other types of chemical work.

Room 6. Storage room for chemicals and spare equipment.
Room 7. Mechanical analysis room
Room 8. Conference / training/ meeting room

A soil testing laboratories also analyses irrigation water samples which are received from the farmers, particularly to check the quality of new irrigation source such as, digging up of a new tube well. A soil testing lab may be required to analyse some water samples. Hence, in **Chapter 8,** procedure for taking water samples and the analytical facilities, methods and important indices to judge the quality of irrigation water have been provided.

Laboratory Safety Measures

Special Care is required while operating equipment, handling the chemicals and in waste disposal.

Equipment

Electrical cables, plugs and tubing need proper check to avoid accident. Various types of gas cylinders needed in the laboratory like acetylene, nitrous oxide and LPG may be kept under watch and properly sealed/capped and may be stored in ventilated cupboards.

Chemical reagents

Hazardous chemicals may be stored in plastic bottles. While working with chemicals such as perchloric acid, fume hood may be used. Chemicals may be properly labelled indicating their hazardous nature.

Bottles with inflammable substances need to be stored in stainless steel containers.

Waste disposal

Cyanides, chromates, arsenic, selenium, cobalt and molybdate are very commonly used but hazardous chemicals and should never be disposed off in the laboratory sink but collected in a metal container for proper disposal at the specified places.

General rules and requirements – Dos and Don'ts in a laboratory

- Learn safety rules and use of first aid kits. Keep the first aid kit handy at a conspicuous working place in the laboratory.
- Personal safety aids such as laboratory coat, hand protection gloves, safety glasses, face shield and proper footwear should be used while working in the laboratory.
- Observe normal laboratory safety practice in connecting equipment with power supply, in handling chemicals and preparing solutions of reagents. All electrical work must be done by qualified personnel.
- Maintain instrument manual and log book for each equipment to avoid mishandling, accident and damage to equipment.
- Calibrate the equipment periodically.
- Carryout standardization of reagents daily before use.
- Always carry out a blank sample analysis with each batch.
- Ensure rinsing of pipette before use with the next solution.
- Do not return the liquid reagents back into the bottle after they are taken out for use.
- Do not put readily soluble substances directly into volumetric flax but first transfer into a beaker, dissolve and then put in the flask.
- Store oxidizing chemicals like iodine and silver nitrate only in amber colour bottles.
- Keep the working tables/space clean. Clean up spillage immediately.
- Wash hands after handling toxic / hazardous chemical.
- Never suck the chemicals with mouth but use automatic pipetting device.
- Use forceps / tongs to remove containers from the hot plates/ovens/furnaces.
- Do not use laboratory glassware for eating/drinking.
- Use fume hood while handling concentrated acids, bases and hazardous chemicals.
- Never open a centrifuge cover until the machine has stopped.

- Add acid to water and not water to acid while diluting the acid.
- Always put labels on bottles, vessels and wash bottles containing reagents, solutions, samples and water.
- Handle perchloric acid in fume hoods.
- Do not heat glasswares and inflammable chemicals directly on the flame.
- Read the labels of the bottles before opening them.
- Do not hold stopper between fingers while pouring liquid from bottle, nor put it on the working table but on a clean watch glass.

Laboratory Quality Assurance/Control

For the uniformity of expression and understanding, the definitions of the terms quality, quality-assurance and quality control as defined by the International Standardization Organization (ISO) and also those compiled in FAO Soils Bulletin 74 and the analytical methods as described in FAO Fertilizer and Plant Nutrition Bulletin No.19 have been largely adopted, as being standard methods and almost universally accepted.

Quality

The Quality has been defined as "the total features and characteristics of a product or service that bear on its ability to satisfy stated and implied need." A product can be stated to possess good quality, if it meets the predetermined parameters. In case of an analytical laboratory, the quality of the laboratory may be considered adequate and acceptable if it has the capacity to deliver the analytical results on a product within the specified limits of errors and as per other agreed conditions of cost and time of analysis so as to enable an acceptable judgement on the product quality.

Quality Assurance

As per ISO, it means "the assembly of all planned and systematic action necessary to provide adequate confidence that a product, a process or service will satisfy given quality requirements". The results of these actions are checked by another independent laboratory/person to conform the pronouncement on the quality of a product by a given laboratory. This could be referred as inter-laboratory check.

Quality Control

Quality control is an important part of quality assurance which is defined by ISO as "the operational techniques and activities that are used to satisfy quality requirements". Quality assessment or evaluation is necessary to see if the activities performed to verify the quality are effective. **Thus, an effective check on all the activities and processes in a laboratory can only ensure that the results pronounced on a product quality are within the acceptable parameters of accuracy.**

In quality control system, the following steps are involved, which when implemented properly, ensure that the results delivered are acceptable and verifiable by another laboratory.

- Check on the performance of the instruments.
- Calibration or standardization of instruments and chemicals.
- Adoption of sample check system as a batch control within the laboratory.
- External check: inter-laboratory exchange programme.

To ensure obtaining accurate and acceptable results of analysis on a sample, the laboratory has to run in a well regulated manner where the equipment are properly calibrated and the methods and techniques employed are scientifically sound which will give reproducible results. For ensuring the high standards of quality, Good Laboratory Practice (GLP) has to be followed. The GLP can be defined as "the organizational process and the conditions under which laboratory studies are planned, performed, monitored, recorded and reported". Thus, the GLP expects a laboratory to work according to a system of procedures and protocols whereas the procedures are also specified as the Standard Operating Procedure (SOP).

Standard Operating Procedure (SOP)

As per Reeuwijk and Houba (1998), a Standard Operating Procedure (SOP) is a document which describes the regularly recurring operations relevant to the quality of the investigation. The purpose of a SOP is to carry out the operation correctly and always in the same manner. A SOP should be available at the place where the work is done. If, for justifiable reasons, any deviation is allowed from SOP, the deviated procedure may be fully documented.

In a laboratory, SOP may be prepared for:

- Safety precaution.
- Procedure for operating instruments.
- Analytical methods and preparation of reagents.
- Registration of samples.

To sum up, all the operations have to be properly documented so as no chance is left for adhocism in any manner.

Error, Precision, Accuracy and Detection Limit

Error

Error is an important component of the analysis. In any analysis, when the quantity is measured with the greatest exactness that the instrument, method and observer are capable of, it is found that the results of successive determination differ among themselves to a greater or lesser extent. The average value is accepted as most probable. This may not

always be true value. In some cases, the difference in the successive values may be small, in some cases it may be large, the reliability of the result depends upon the magnitude of this difference. There could be a number of factors responsible for this difference which is also referred as 'error'. The error in absolute term is the difference between the observed or measured value and the true or most probable value of the quantity measured. The absolute-error is a measure of the accuracy of the measurement. The accuracy of a determination may, therefore, be defined as the concordance between it and the true or most probable value. The relative error is the absolute error divided by the true or most probable value.

The error may be caused due to any deviation from the prescribed steps required to be taken in analysis. The purity of chemicals, their concentration/strength and the accuracy of the instruments and the skill of the technician are important factors.

Precision and accuracy

In analysis, other important terms to be understood are precision and accuracy. Precision is defined as the concordance of a series of measurements of the same quantity. The mean deviation or the relative mean deviation is a measure of precision. In quantitative analysis, the precision of a measurement rarely exceeds 1 to 2 parts per thousand.

Accuracy expresses the correctness of a measurement, while precision expresses the reproducibility of a measurement. Precision always accompanies accuracy, but a high degree of precision does not imply accuracy. In ensuring high accuracy in analysis, accurate preparation of reagents including their perfect standardization is critical. Not only this, even the purity of chemicals is important. For all estimation, where actual measurement of a constituent of the sample in terms of the "precipitate formation" or formation of "coloured compound" or "concentration in the solvent" is a part of steps in estimation, chemical reagents involved in such aspects must always be of high purity which is referred as AR-grade (Analytical Reagent).

Detection limit

In the analysis for trace elements in soils, plants and fertilizers and for environmental monitoring, need arises to measure very low contents of analytes. Modern equipments are capable of such estimation. However, while selecting an equipment and the testing method for such purpose, it is important to have information about the lowest limits upto which analytes can be detected or determined with sufficient confidence. Such limits are called as detection limits or lower limits of detection.

The capacity of the equipment and the method may be such that it can detect the traces of analyte in the sample. In quantitative terms, the lowest contents of such analyte may be decided through appropriate

research as the values of interpretable significance. The service laboratories are generally provided with such limits.

Quality Control of Analytical Procedures

Independent Standards

The ultimate aim of the quality control measures is to ensure the production of analytical data with a minimum of error and with consistency. Once, an appropriate method is selected, its execution has to be done with utmost care. To check and verify the accuracy of analysis, independent standards are used in the system. The extent of deviation of analytical value on a standard sample indicates the accuracy of the analysis. Independent standard can be prepared in the laboratory from pure chemicals. When new standard is prepared, the remainder of the old ones always have to be measured as a mutual check. If the results are not within the acceptable levels of accuracy, the process of calibration, preparation of standard curve and the preparation of reagents may be repeated till acceptable results are obtained on the standard sample. After assuring this, analysis on unknown sample has to be started.

Apart from independent standard, certified reference samples can also be used as 'standard'. Such samples are obtained from other selected laboratories where the analysis on a prepared standard is carried out by more than one laboratory and such samples along with the accompanied analytical values are used as a check to ensure the accuracy of analysis.

Use of blank

A blank determination is an analysis without the analyte or attribute or in other words, an analysis without a sample by going through all steps of the procedure with the reagents only. **Use of blank accounts for any contamination in the chemicals used in actual analysis**. The 'estimate' of the blank is subtracted from the estimates of the samples. The use of 'sequence control' samples is made in long batches in automated analysis. Generally two samples, one with a low content of analyte and another with very high content of known analyte (but the contents falling within the working range of the method) are used as standards to monitor the accuracy of analysis.

Blind sample

A sample with known content of analyte. This sample is inserted by the head of the laboratory in batches and times unknown to the analyst. Various types of sample material may serve as blind samples such as control samples or sufficiently large leftover of test samples (analysed several times). It is essential that analyst is aware of the possible presence of a blind sample but is not able to recognize the material as such.

Validation of procedures of analysis

Validation is the process of determining the performance characteristics of a method / procedure. It is a pre-requisite for judgement of the suitability of produced analytical data for the intended use. This implies that a method may be valid in one situation and invalid in another. If a method is very precise and accurate but expensive for adoption, it may be used only when the data with that order of precision are needed. The data may be inadequate, if the method is less accurate than required. Two types of validation are followed.

Validation of own procedure

In-house validation of method or procedure by individual user laboratory is a common practice. Many laboratories use their own version of even well established method for reasons of efficiency, cost and convenience. A change in liquid solid ratio in extraction procedures for available soil nutrients and shaking time etc. result in changed value, hence need validation. Such changes are often introduced to consider local conditions, cost of analysis, required accuracy and efficiency.

Validation of such changes is the part of quality control in the laboratory. It is also a kind of research project, hence all types of the laboratories may not be in a position to modify the standard method. They should follow the given method as accepted and practiced by most other laboratories.

Apart from validation of methods, a system of internal quality control is required to be followed by the laboratories to ensure that they are capable of producing reliable analytical data with minimum of error. This requires continuous monitoring of the operation and systematic day to day checking of the produced data to decide whether these are reliable enough to be released.

Following steps need to be taken for internal quality control:

- Use a blank and a control (standard) sample of known composition along with the samples under analysis.
- Round off the analytical values to the 2nd decimal place. The value of 3rd decimal place may be omitted if less than 5. If it is more than 5, the value of second decimal may be raised by 1.

Since the quality control systems rely heavily on control samples, the sample preparation may be done with great care to ensure that the:

- Sample is homogenous.
- Sample material is stable.

- Sample has uniform and correct particle size as sieved through a standard sieve.
- Relevant information such as properties of the sample and the concentration of the analyte are available.

The samples under analysis may also be processed / prepared in such a way that it has similar particle size and homogeneity as that of the standard (control) sample.

As and when an error is noticed in the analysis through internal check, corrective measures should be taken. The error can be due to calculation or typing. If not, it requires thorough check on sample identification, standards, chemicals, pipettes, dispensers, glassware, calibration procedure and equipment. Standard may be old or wrongly prepared. Pipette may indicate wrong volume, glassware may not be properly cleaned and the equipment may be defective or the sample intake tube may be clogged in case of flame photometer or Atomic Absorption Spectrophotometer. Source of error may be detected and samples be analyzed again.

Validation of the Standard Procedure

This refers to the validation of new or existing method and procedures intended to be used in many laboratories including procedures accepted by national system or ISO. This involves an inter-laboratory programme of testing the method by a member of selected renowned laboratories according to a protocol issued to all participants. Validation is not only relevant when non-standard procedures are used but just as well when validated standard procedures are used and even more so when variants of standard procedures are introduced. The results of validation tests should be recorded in a validation report from which the suitability of a method for a certain purpose can be deduced.

Inter-laboratory sample and data exchange programme:

If an error is suspected in the procedure and uncertainty cannot readily be solved, it is not uncommon to have the sample analysed in another laboratory of the same system/organisation. The results of the other laboratory may or may not be biased, hence doubt may persist. The sample check by another accredited laboratory may be necessary and useful to resolve the problem.

An accredited laboratory should participate at least in one inter-laboratory exchange programme. Such programmes do exist locally, regionally, nationally and internationally. The laboratory exchange programme exists for method performance studies and laboratory performance studies.

In such exchange programme, some laboratories or the organizations have devised the system where periodically samples of known composition are sent to the participating laboratory without disclosing the results. The participating laboratory will analyse the sample by a given method and find out the results. It provides a possibility for assessing the accuracy of the method being used by a laboratory, and also about the adoption of the method suggested by the lead laboratory. Some of Such Programmes are:

- International Plant Analytical Exchange (IPE) Programme, and
- International Soil Analytical Exchange (ISE) Programme.

They come under the Wageningen Evaluating Programme for Analytical Laboratories (WEPAL) of the Wageningen Agricultural University, the Netherlands. Other programmes run by the Wageningen Agricultural University are:

- International Sediment Exchange for Tests on Organic Contaminants (SETOC).
- International Manure and Refuse Sample Exchange Programme (MARSEP).

For quality check, each laboratory will benefit if it becomes part of some sample/method check and evaluation programme. The system of self-check within the laboratory also has to be regularly followed.

Preparation of Reagent Solutions and their Standardization

Chemical reagents are manufactured and marketed in different grades of purity. In general the purest reagents are marketed as "Analytical Reagent" or AR grade. Further, the markings may be 'LR' meaning laboratory reagent or "CP", meaning chemically pure. The strength of chemicals is expressed as normality or molarity. It is, therefore, useful to have some information about the strength of important acids and alkali most commonly used in the chemical laboratories **(Table 12).** Acids and alkali are basic chemicals required in a laboratory.

TABLE-12

Strength of commonly used acids and alkali

S.N	Reagent /chemical	Normality (aprox.)	Molarity (aprox.)	Formula weight	% by weight (aprox.)	Specific Gravity (aprox.)	ml required for 1N/litre solution	ml required for 1M/litrsolu
1.	Nitric Acid	16	16.0	63	70	1.42	63.7	63.7
2.	Sulphuric Acid	35	17.5	98	98	1.84	28.0	56
3.	Hydrochloric Acid	11.6	11.6	36.5	37	1.19	82.6	82.6
4.	Phosphoric Acid	45	15	98	85	1.71	22.7	68.1
5.	Perchloric Acid	10.5	10.5	100.5	65	1.60	108.7	108.7
6.	Ammonium hydroxide	15	15	35	28	0.90	67.6	67.6

Some important terms which are often used in a laboratory for chemical analysis are defined / explained below:

Molarity

One molar (M) solution contains one mole or one molecular weight in grams of a substance in each litre of the solution. Molar method of expressing concentration is useful due to the fact that the equal volumes of equimolar solutions contain equal number of molecules.

Normality

The normality of a solution is the number of gram equivalents of the solute per litre of the solution. It is usually designated by letter N. Semi-normal, penti-normal, desi-normal, centi-normal and milli-normal solutions are often required, these are written (shortly) as 0.5N, 0.2N, 0.1N, 0.01N and 0.001N, respectively. However, molar expression is preferred because 'odd' normalities such as 0.121N are clumsily represented in fractional form.

The definition of normal solution utilizes the term 'equivalent weight'. This quantity varies with the type of reaction, and hence it is difficult to give a clear definition of equivalent weight which will cover all reactions. It often happens that the same compound possess different equivalent weights in different chemical reactions. A situation may arise where a solution has normal concentration when employed for one purpose and a different normality when used in another chemical reaction. Hence, the system of molarity is preferred.

Equivalent weight (Eq W)

The equivalent weight of a substance is the weight in grams which in its reaction corresponds to a gram atom of hydrogen or of hydroxyl or half a gram atom of oxygen or a gram atom of univalent ion. When one equivalent weight of a substance is dissolved in one litre, it gives I N solution. Equivalent and molecular weights of some important compounds are given in **Annexure- 9.**

Milliequivalent weight (mEq W)

Equivalent weight (Eq W) when expressed as milli-equivalent weight (mEq W), means the equivalent weight in grams divided by 1000. It is commonly expressed by "me". It is the most convenient value because it is the weight of a substance contained in or equivalent to one ml of I N solution. It is, therefore, a unit which is common to both volumes and weights, making it possible to convert the volume of a solution to its equivalent weight and the weight of a substance to its equivalent volume of solution.

Number of mEq = Volume x Normality

Buffer solutions

Solutions containing a weak acid and its salt or weak base and its salt (e.g. CH_3COOH + CH_3COONa) and (NH_4OH + NH_4Cl) possess the characteristic property to resist changes in pH when some acid or base is added in them. Such solutions are referred to as buffer solutions. Following are important properties of a buffer solution:

- It has a definite pH value.
- Its pH value does not alter on keeping for a long time.
- Its pH value is only slightly altered when strong base or strong acid is added.

It may be noted that because of the above property, readily prepared buffer solutions of known pH are used to check the accuracy of pH meters being used in the laboratory.

Titration

It is a process of determining the volume of a substance required to just complete the reaction with a known amount of other substance. The solution of known strength used in the titration is called titrant. The substance to be determined in the solution is called titrate.

The completion of the reaction is judged with the help of appropriate indicator.

Indicator

A substance which indicates the end point on completion of the reaction is called as indicator. Most commonly used indicators in volumetric analysis are:

- internal indicator
- external indicator
- self indicator

i.) Internal indicator

The indicators like methyl red, methyl orange, phenolphthalein and diphenylamine which are added in the solution where reaction occurs, are called internal indicators. On completion of the reaction of titrant on titrate, a colour change takes place due to the presence of indicator, which also helps in knowing that the titration is complete. Typical examples of colour change due to pH change is solutions are given in **Annexure-11**. The internal indicators used in acid - alkali neutralization solutions are methyl orange, phenolphthalein and bromothymol blue.

The indicator used in precipitation reactions like titration of neutral solution of NaCl (or chlorine ion) with silver nitrate ($AgNO_3$) solution is K_2CrO_4. On the completion of titration reaction of $AgNO_3$ with chlorine, when no more chlorine is available for reaction with silver ion to form AgCl, the chromium ions combine with Ag^{2+} ions to form sparingly soluble Ag_2CrO_4, which is brick red in colour. It indicates that chlorine has been completely titrated and end point has occurred.

Redox indicators are also commonly used. These are substances which possess different colours in the oxidized and reduced forms. Diphenylamine has blue violet colour under oxidation state and colourless in reduced condition. Ferrocin gives blue colour under oxidation state and red colour under reduced condition.

ii) External indicator

Some indicators are used outside the titration mixture. Potassium ferricyanide is used as an external indicator in the titration of potassium dichromate and ferrous sulphate in acid medium. In this titration, few drops of indicator are placed on a white porcelain tile. A glass rod dipped in the solution being titrated is taken out and brought in contact with the drops of indicator placed on white tile. In the beginning deep blue colour is noticed which turns greenish on completion of titration.

iii) Self indicator

The titrant, after completion of the reaction leaves its own colour due to its slight excess in minute quantities. In $KMnO_4$ titration with ferrous sulphate, the addition of $KMnO_4$ starts reacting with $FeSO_4$ which is colourless. On completion of titration, slight excess presence of $KMnO_4$

gives pink colour to the solution which acts as a self indicator and points to the completion of the titration.

Standard solution

The solution of accurately known strength (or concentration) is called a standard solution. It contains a definite number of gram equivalent or gram mole per litre of solution. If it contains 1 gram equivalent weight of a substance/compound, it is 1N solution. If it contains 2 gram equivalent weights of the compound, it is 2N.

All titrametric methods depend upon standard solutions which contain known amounts (exact) of the reagents in unit volume of the solution. A solution is prepared, having approximately the desired concentration. This solution is then standardized by titrating it with another substance which can be obtained in highly purified form. Thus, potassium permanganate solution can be standardized against sodium oxalate which can be obtained in a high degree of purity, since it is easily dried and is non-hygroscopic. Such substance, whose weight and purity is stable, is called as 'Primary Standard'. A primary standard must have the following characteristics:

- It must be obtainable in a pure form or in a state of known purity.
- It must react in one way only under the condition of titration and there must be no side reactions.
- It must be non-hygroscopic. Salt hydrates are generally not suitable as primary standards.
- Normally, it should have a large equivalent weight so as to reduce the error in weighing.
- An acid or a base should preferably be strong, that is, they should have a high dissociation constant for being used as standards.

Primary standard solution is one which can be prepared directly by weighing and with which other solutions of approximate strength can be titrated and standardized. Some Primary standards are given below:

Acids	1. Potassium hydrogen phthalate 2. Benjoic acid
Bases	1. Sodium carbonate 2. Borax
Oxidizing agents	1. Potassium dichromate 2. Potassium bromate
Reducing agents	1. Sodium oxalate 2. Potassium Ferro cyanide

Others 1. Sodium chloride
2. Potassium chloride

Secondary Standard Solutions are those which are prepared by dissolving a little more than the gram equivalent weight of the substance per litre of the solution and then their exact standardization is done with primary standard solution. Some Secondary standards are given below:

Acid 1. Sulphuric acid
2. Hydrochloric acid

Base 1. Sodium hydroxide.

Standard solutions of all the reagents required in a laboratory must be prepared and kept ready before taking up any analysis. However, their strength should be periodically checked or fresh reagents be prepared before analysis.

In all titrations involving acidimetry and alkalimetry, standard solutions are required. These may be prepared either from standard substances by direct weighing or by standardizing a solution of approximate normality of materials by titrating against a prepared standard. The methods of preparation of standard solutions of some non-primary standard substances of common use are given below.

Standardization of hydrochloric acid (HCl):

The concentrated H Cl is approximately 11N. Therefore, to prepare a standard solution, say decinormal (0.1N) of the acid, it is diluted roughly one hundred times. Take 10 ml of acid and make approximately 1 litre by dilution with distilled water. Titrate this acid against 0.1N Na_2CO_3 (Primary standard) using methyl orange as indicator. Colour changes from pink to yellow when acid is neutralized. Suppose 10 ml of acid and 12ml of Na_2CO_3 are consumed in the titration.

Acid	**Alkali**
$V_1 \times N_1$	$= V_2 \times N_2$
$10 \times N_1$	$= 12 \times 0.1$
$10\ N_1$	$= 1.2$
N_1	$= 0.12$

Normality of acid is 0.12

Similarly, normality of sulphuric acid can be worked out. H_2SO_4 needs to be diluted about 360 times to get approximately 0.1N because it has a normality of approximately 35. Then titrate against standard Na_2CO_3 to find out exact normality of H_2SO_4.

Standardization of sodium hydroxide (NaOH):

As per above method, the normality of HCl/H_2SO_4 has been fixed. Therefore, to find out the normality of sodium hydroxide, titration is carried out by using any one of these standard acids to determine the normality of the sodium hydroxide. For working out molarity, molar standard solutions are used.

In case of the standardization of NaOH or any other alkali, potassium hydrogen phthalate can also be used as a primary standard instead of going through the titration with secondary standards. It can be decided depending upon the availability of chemicals in the laboratory.

Conversion Factors

In a laboratory exercise, various units of measurement, such as area, volume, mass, length, pressure, temperature etc. are required to be converted from Si to non-Si unit and vice-versa. In this regard, **Annexure-12** is given as a handy ready reconkner for the use by lab technicians.

4.6.2. Soil Sampling

The method and procedure for obtaining soil samples vary according to the purpose of sampling. Analysis of soil samples may be needed for engineering and agricultural purposes. In this publication, soil sampling for agricultural purpose is described which is done for soil fertility evaluation and fertilizer recommendations for crops.

The results of even very carefully conducted soil analysis are as good as the soil sample itself. Thus, the efficiency of soil testing service depends upon the care and skill with which soil samples are collected. Non-representative samples constitute the largest single source of error in a soil fertility programme. It is to be noted that the most important phase of soil analysis is accomplished not in a laboratory but in the field where soils are sampled.

Soils vary from place to place. In view of this, efforts should be made to take the samples in such a way that it is fully representative of the field. Only one to ten gram of soil is used for each chemical determination and represents as accurately as possible the entire surface 0-22 cm of soil, weighing about 2 million kg/ha.

Sampling tools and accessories

Depending upon the purpose and precision required, following tools may be needed for taking soil samples.

- Soil auger- it may be a tube auger, post hole or screw type auger or even a spade for taking samples.

- A clean bucket or a tray or a clean cloth for mixing the soil and sub sampling.
- Cloth bags of specific size.
- Copying pencil for markings and tags for tying cloth bags.
- Soil sample information sheet.

Selection of a sampling unit

A visual survey of the field should precede the actual sampling. Note the variation in slope, colour, texture, management and cropping pattern by traversing the field. Demarcate the field into uniform portions, each of which must be sampled separately. If all these conditions are similar, one field can be treated as a single sampling unit. Such unit should not exceed 1 to 2 hectares, and it must be an area to which a farmer is willing to give separate attention. The unit of sampling is a compromise between the expenditure, labour and time on one hand and precision on the other. In view of limited soil testing facilities, it has been suggested to adopt an alternate approach where a sample may be collected from an area of 20-50 ha to be called as composite area soil sample and analyse the same for making a common recommendation for the whole area.

Sampling procedure

Prepare a map of the area to be covered in a survey showing different sampling unit boundaries. A plan of the number of samples and manner of composite sampling is entered on the map, different fields being designated by letters A, B, C etc. Each area is traverse separately. A slice of the plough-layer is cut at intervals of 15 to 20 steps or according to the area to be covered. Generally 10 to 20 spots must be taken for one composite sample depending on the size of the field.

Scrap away surface liter; obtain a uniformly thick slice of soil from the surface to the plough depth from each place. A V-shaped cut is made with a spade to remove 1 to 2 cm slice of soil. The sample may be collected on the blade of the spade and put in a clean bucket. In this way collect samples from all the spots marked for one sampling unit. In case of hard soil, samples are taken with the help of augur from the plough depth and collected in the bucket.

Pour the soil from the bucket on a piece of clean paper or cloth and mix thoroughly. Spread the soil evenly and divide it into 4 quarters. Reject two opposite quarters and mix the rest of the soil again. Repeat the process till left with about half kg of the soil, collect it and put in a clean cloth bag. Each bag should be properly marked to identify the sample.

The bag used for sampling must always be clean and free from any contamination. If the same bag is to be used for second time, turn it inside out and remove the soil particles. Write the details of the sample in the information sheet (a suggested format is given in **Annex 10**). Put a

copy of this information sheet in the bag. Tie the mouth of the bag carefully.

Precautions

- Do not sample unusual area like unevenly fertilized, marshy, old path, old channel, old bunds, area near the tree, site of previous compost piles and other unrepresentative sites.
- For a soft and moist soil, the tube auger or spade is considered satisfactory. For harder soil, a screw auger may be more convenient.
- Where crops have been planted in rows, collect samples from the middle of the rows so as to avoid the area where fertilizer has been band placed.
- Avoid any type of contamination at all stages. Soil samples should never be kept in the store along with fertilizer materials and detergents. Contamination is likely when the soil samples are spread out to dry in the vicinity of stored fertilizers or on floor where fertilizers were stored previously.
- Before putting soil samples in bags, they should be examined for cleanliness as well as for strength.
- Information sheet should be clearly written with copying pencil.

Sampling of salt affected soils

Salt affected soils may be sampled in two ways. Surface samples should be taken in the same way as for soil fertility analysis. These samples are used to determine gypsum requirement of the soil. For reclamation purpose, it is necessary to know the characteristics of lower soil depth also. Such soils are, therefore, sampled depth wise up to one meter. The samples may be removed from one to two spots per 0.4 hectare if the soil is uniformly salt affected. If patches are conspicuous then all big patches should be sampled separately. Soil is sampled depth wise separately (about ½ kg from each depth) for 0-15 cm, 15-30 cm, 30-60 cm and 60-100 cm soil depths. If a stony layer is encountered during sampling, such a layer should be sampled separately and its depth noted. This is very important and must not be ignored.

Soil samples can be removed by a spade or if the auger is used then care should be taken to note the depth of 'concretion' (stones) or other impermeable layer (hard pan). If the soil shows evidence of profile development or distinct stratification, samples should be taken horizon wise. If a pit is dug and horizons are absent then mark the vertical side of the pit at 15, 30, 60 and 100 cm depth from the surface and collect about ½ kg. soil from every layer, cutting uniform slices of soil separately. In addition to the above sampling, one surface soil sample should be taken as in the case of normal soil sampling for fertilizer recommendation.

Pack the samples and label the bags in the same way as is done for normal soil sampling, giving additional information about the depth of the

sample. The sheet accompanying the sample must include the information on nature of soil, hardness and permeability of soil, salinity cause and source, if known, relief, seasonal rainfall, irrigation and frequency of water logging, water table, soil management history, crop species and conditions of plant cover and depth of the hard pan or concretion. As the salt concentration may vary greatly with vertical or horizontal distance and with moisture and time, account must be kept about time of irrigation, amount of irrigation or rain received prior to sampling.

Despatch of Soil Samples to the Laboratory

Before sending soil samples to the testing laboratory by a farmer, it should be ensured that proper identification marks are present on the sample bags as well as labels placed in the bags. It is essential that it should be written by copying pencil and not with ink because the ink will smudge and become illegible. The best way is to get the soil sampling bags from soil testing laboratory with most of the information printed or stencilled on them with indelible ink.

Compare the number and details on the bag with the dispatch list. The serial numbers of different places should be distinguished by putting the identification mark specific for each centre. This may be in alphabets, say one for district and another for block/county and third for the village.

Pack the samples properly. Wooden boxes are most suitable for long transport. Sample bags may be packed only in clean bags never used for fertilizer or detergent packing.

Farmers may bring soil samples directly to the laboratory. Most of the samples are, however, sent to the laboratories through the field extension staff. An organized assembly-processing despatch system is required to ensure prompt delivery of samples to the laboratory.

Preparation of soil samples for analysis

Handling in the laboratory

As soon as the samples are received at the soil testing laboratory, they should be checked with the accompanying information list. If the soil testing laboratory staffs have collected the samples themselves, then adequate field notes might have been kept. All unidentifiable samples should be discarded. Information regarding samples should be entered in a register and each sample be given a laboratory number, in addition to sample number, which helps to distinguish if more than one source of samples is involved.

Drying of samples

Samples received in the laboratory may be moist. These should be dried in wooden or enamelled trays. Care should be taken to maintain the identity of each sample at all stages of preparation. During drying, the trays can be numbered or a plastic tag could be attached. The soils are allowed to dry in the air. Alternatively, the trays may be placed in racks in a hot air cabinet whose temperature should not exceed 35^0 C and relative humidity should be between 30 and 60%. Oven drying a soil can cause profound change in the sample. This step is not recommended as a preparatory procedure in spite of its convenience. Drying has negligible effect on total N content but the nitrate content in the soil changes with time and temperature. Microbial population is affected due to drying at high temperature. With excessive drying, soil potassium may be released or fixed depending upon the original level of exchangeable potassium. Exchangeable potassium will be increased if its original level was less than 1 meq/100 g soil (1 cmol/kg) and *vice-versa*, but the effect depends upon the nature of clay minerals in the soil. In general, excessive drying, such as oven drying of the soil, affects the availability of most of the nutrients present in the sample and should be avoided. Only air drying is recommended.

Nitrate, nitrite and ammonium determinations must be carried out on samples brought straight from the field. These samples should not be dried. However, the results are expressed on oven dry basis by separately estimating moisture content in the samples.

Post drying care

After drying, the samples are taken to the preparation room which is separate from the main laboratory. Air dried samples are ground with a wooden pestle and mortar so that the soil aggregate are crushed but the soil particles do not break down. Samples of heavy clay soils may have to be ground with an end runner grinding mill fitted with a pestle of hard wood and rubber lining to the mortar. Pebbles, concretions and stones should not be broken during grinding.

After grinding, the soil is screened through a 2 mm sieve. The practice of passing only a portion of the ground sample through the sieve and discarding the remainder is erroneous. This introduces positive bias in the sample as the rejected part may include soil elements with differential fertility. The entire sample should, therefore, be passed through the sieve except for concretions and pebbles of more than 2 mm. The coarse portion on the sieve should be returned to the mortar for further grinding. Repeat sieving and grinding till all aggregate particles are fine enough to pass the sieve and only pebbles, organic residues and concretions remain out.

If the soil is to be analyzed for trace elements, containers made of copper, zinc and brass must be avoided during grinding and handling. Sieves of different sizes can be obtained in stainless steel. Aluminium or plastic sieves are useful alternative for general purposes.

After the sample is passed through the sieve, it must be again mixed thoroughly.

The soil samples should be stored in cardboard boxes in wooden drawers. These boxes should be numbered and arranged in rows in the wooden drawers, which are in turn fitted in a cabinet in the soil sample room.

4.6.3 . Analytical methods for estimation of physical properties and available nutrients

1. Soil Texture
2. Soil Structure
3. Cation Exchange Capacity (CEC)
4. Soil Moisture
5. Water Holding Capacity (WHC)
6. pH
7. Lime Requirement
8. Soil Electrical Conductivity and gypsum requirement

8.a. Electrical Conductivity
8.b. Gypsum requirement

9. Organic Carbon
10. Total Nitrogen
11. Mineralizable N
12. Inorganic N – NO3 & NH4
13. Available Phosphorus
14. Available Potassium
15. Available Sulphur
16. Exchangeable Calcium and Magnesium

16.a. Calcium by Versenate (EDTA) Method
16.b. Calcium plus Magnesium by Versenate (EDTA) Method

17. Micronutrients

17.a. Available Zinc, Copper, Iron, Manganese
17.b. Available Boron
17.c. Available Molybdenum

1. Soil Texture

Soil texture or particle size distribution is a stable soil characteristic which influences physical and chemical properties of the soil. The sizes of the soil particles have a direct relationship with the surface area of the particles. Soil particles remain aggregated due to various types of binding forces and factors which include the content of organic matter, other colloidal substances present in the soil, oxides of iron and aluminium and the hydration of clay particles etc. To estimate the content of various sizes of soil particles, the soil sample has to be brought into dispersed state by removing various types of binding forces.

In the dispersed soil samples, the soil particles settle down at a differential settling rate according to their size. In the estimation of soil texture, particles below 2mm diameter are separately determined which constitute sand, silt and clay. Each one of them is characterized as below:

Coarse sand:	2.0 – 0.2 mm diameter
Fine sand:	0.2 – 0.02 mm diameter
Silt:	0.02 – 0.002 mm diameter
Clay:	< 0.002 mm diameter

The soil sample is dispersed by removing the binding force in soil particles. The settling rate of dispersed particles in water is measured. Large particles are known to settle out of suspension more rapidly than do small particles. This is because larger particles have less specific area and hence have lesser buoyancy than smaller particles. Stoke's law (1851) is used to express the relationship. The law stipulates that the resistance offered by the liquid to the fall of the particle varies with the radius of the sphere and not with the surface. Accordingly, the formula was given as below:

$$V = \frac{2}{9}\left(\frac{dp - d}{\eta}\right) gr^2$$

Where, V is the velocity of the fall in centimeter per second, g is the acceleration due to gravity, dp is the density of the particle, d is the density of the liquid, r is the radius of the particle in centimeter, and η is the absolute viscosity of the liquid. It is obvious that the velocity of fall of the particles with the same density in a given liquid will increase with the square of the radius.

With the above principle in view, the particle size distribution is estimated by measuring the amount of different sizes of soil particles present at different calibrated depths in the cylinder containing suspended soil sample.

Generally, two methods are most commonly used for estimation of particle size or soil texture:

- International Pipette method
- Bouyoucos Hydrometer method

Hydrometer method is most commonly used since it is less time consuming and easy to follow in a service laboratory. Dispersion is obtained by using Calgon (Sodium haxametaphosphate).

Hydrometer method

Apparatus

- Balance
- Cylinder - 1 litre and 1.5 litre

- Glass beaker – 1 litre
- Metal stirrer with 1500 rpm speed
- Bouyoucos hydrometer
- Oven
- Thermometer C^0

Reagent

- sodium hexametaphosphate solution containing 50 g salt per litre of water

Procedure

1. Weigh 50 g oven dried fine textured soil (100 g for coarse textured soil) into a baffled stirring cup. Fill the cup to its half with distilled water and add 10 ml of sodium hexametaphosphate solution.
2. Place the cup on stirrer and stir until soil aggregates are broken down. This usually requires 3-4 minutes for coarse textured soils and 7-8 minutes for fine textured clay.
3. Quantitatively transfer stirred mixture to the settling cylinder by washing the cup with distilled water. Fill the cylinder to the lower mark with distilled water after placing the hydrometer in the liquid. If 100 g of coarse textured sample was used, fill to the upper mark on the settling cylinder.
4. Remove hydrometer and shake the suspension vigorously in a back and forth manner. Avoid creating circular currents in the liquid as they will influence the settling rate.
5. Place the cylinder on a table and record the time. After 20 seconds, carefully insert the hydrometer and read the hydrometer at the end of 40 seconds.
6. Repeat step 4 and 5 to obtain hydrometer readings within 0.5 g differences from each other. The hydrometer is calibrated to read grams of soil material in suspension.
7. Record the hydrometer readings on the data sheet (given below).
8. Measure the temperature of the suspension. For each degree above 20° C add 0.36 to the hydrometer reading, and for each degree below 20° C, subtract 0.36 from the hydrometer reading. This is the corrected hydrometer reading.
9. Reshake the suspension and place the cylinder on a table where it will not be disturbed. Take a hydrometer reading exactly two hours later. Correct for temperature as described above.
10. From the percentage of sand, silt and clay calculated on the Data Sheet, use the diagram for textural triangle to determine the textural class of the soil.

Table 13 :Data sheet for recording of hydrometer readings:

1	Soil sample identification number	_____
2	Soil weight (g)	_____
3	Forty second hydrometer reading (g)	_____
4	Temperature of suspension (C^0)	_____
5	Corrected 40-second hydrometer reading (g)	_____
6	Two hours hydrometer reading (g)	_____
7	Temperature of suspension (C^0)	_____
8	Corrected 2-hour hydrometer reading (g)	_____
9	Grams of sand (the sand settles to the bottom of the cylinder within 40 seconds, therefore, the 40-second corrected hydrometer reading actually gives the grams of silt and clay in suspension. The weight of sand in the sample is obtained by subtracting line 5 from line 2).	_____
10	Grams of clay (the corrected hydrometer reading at the end of two hours represents grams of clay in the suspension since all sand and silt has already settled by this time).	_____
11	Percent sand (line 9 ÷ line 2) × 100	_____
12	Percent clay (line 10 ÷ line 2) × 100	_____
13	Percent silt (find the silt by difference. Subtract the sum of the percent sand and clay from 100).	_____
14	Soil class (as per Figure 1)	_____

FIGURE 1

Soil textural classes according to proportions of sand, silt and clay

2. Soil Structure

Soil structure is defined as the arrangement of the soil particles. With regard to structure, soil particles refer not only to sand, silt and clay but also to the aggregate or structural elements, which have been formed by the aggregation of smaller mechanical fractions. The word 'particle' therefore, refers to any unit that is part of the make up of the soil, whether primary unit (sand, silt or clay fraction) or a secondary (aggregate) particle.

The size, shape and character of the soil structure varies, which could be cube like, prism like or platter like. On the basis of size, the soil structure is classified as follows:

- Very coarse: >10 mm
- Coarse: 5-10 mm
- Medium: 2-5 mm
- Fine: 1-2 mm
- Very fine: <1 mm

Depending upon the stability of the aggregate and the ease of separation, the structure is characterized as follows:

- Poorly developed
- Weekly developed
- Moderately developed
- Well developed
- Highly developed

The soil structure or aggregate consists of an intermediate grouping of number of primary particles into a secondary unit. The important factors which facilitate the aggregation of soil particles are:

- Clay particles and types of clay minerals
- Cations such as Calcium
- Organic matter
- Colloidal matter such as oxides of iron and aluminium
- Plant roots
- Soil microbes and their types where fungi being most effective

Soil structure influences the extent of pore space in the soil, water holding capacity, aeration, root movement and the nutrient availability. The better and more stable soil aggregates are considered as a desirable soil property with regard to plant growth. Determination of soil structure is, therefore, an important exercise in soil fertility evaluation programme. An aggregate analysis aims to measure the percentage of water stable secondary particles in the soil and the extent to which the finer mechanical separates are aggregated into coarser fractions.

The determination of aggregate or clod size distribution involves procedures that depend on the disintegration of soil into clods and

aggregates. The resulting aggregate size distribution depends on the manner and condition in which the disintegration is brought about. For the measurements to have practical significance, the disruptive forces causing disintegration should closely compare with the forces expected in the field. The field condition particularly with respect to soil moisture should be compared with the moisture condition adopted for soil disintegration in the laboratory. The sampling of soil and subsequent disintegration of clods in relevance to seed bed preparation for upland crops should be carried out under air dry conditions for dry sieve analysis. A rotary sieve shaker would be ideal for dry sieving. Similarly the processes of wetting, disruption of dry aggregates and screening of aggregates should be compared to the disruptive actions of water and mechanical forces of tillage under wetland conditions. Although vacuum wetting of dry soil largely simulates the process of wetting in-situ, particularly in the subsurface layers, the surface soil clods, however, experience large scale disruption when they are immersed in water at atmospheric pressure. The re-productivity of the size distribution of clods should naturally be the criterion for deciding the method of wetting by either vacuum wetting or immersion in water. The immersion wetting is more closer to wetting of surface soil by irrigation.

After wetting, aggregates of different sizes can be obtained through several methods like sedimentation, elutriation and sieving. However, sieving under water compares more closely with the disruptive actions of water and other mechanical forces as experienced during wetland rice field preparation.

Dry Aggregates Analysis (Gupta and Ghil Dyal, 1998)

The size distribution of dry clods is measured by Dry Sieving Analysis performed on air dry bulk soil sample either manually or with the help of a rotary sieve shaker.

Apparatus

- Nest of sieves, 20 cm in diameter and 5 cm in height, with screens having 25.0, 10.0, 5.0, 2.0, 1.0, 0.5 and 0.25 mm size round openings with a pan and a lid
- Rotary sieve shaker
- Aluminium cans
- Balance
- Spade
- Brush
- Polyethelene bags
- Labels

Procedure

Bulk soil sample is collected from the tilled field with the help of a 20 cm diameter and 10 cm height ring. The ring is placed on the tilled soil and pressed until in level with the surface. The loose soil within the ring is removed and collected in a polyethelene bag.

One label indicating the depth and soil profile is put inside the bag and the other label is tied with the bag. The soil samples are then brought to the laboratory and air dried.

Spread the soil on a sheet of paper and prepare the subsamples by 'quartering'. The mixed soil material is coned in the center of the mixing sheet with care to make it symmetrical with respect to fine and coarse soil material. The cone is flattened and divided through the center with a flat metal spatula or metal sheet, one-half being moved to the side quantitatively. Each one-half is further divided into halves; the four quarters being separated into separate piles or 'quarters'. The sub-samples from two of these 'quarters' are weighed and used for clod size and aggregate distribution analysis as duplicates. The weighed soil sample is transferred to the top sieve of the nest of sieves having 5.0, 2.0, 1.0, 0.5 and 0.25 mm diameter round openings and a pan at the bottom. Cover the top sieve with the lid and place the nest of sieves on a rotary shaker. Switch on the shaker for 10 minutes, and then remove the sieves, and collect the soil retained on each screen in the pre-weighed aluminium cans, with the help of a small brush, and weigh the cans with the soil.

If the percentage of dry aggregates on 5 mm sieve exceeds 25%, transfer these aggregates to a nest of sieves with 25.0, 10.0 and 5.0 mm sieves along with a pan. Cover the top sieve containing the aggregates with a lid and place the nest of sieves on the rotary sieve shaker. Switch on the motor for 10 minutes and proceed as above for the estimation of aggregate size distribution. Analyse the duplicate sample following the same procedure and calculate the percent distribution of dry aggregates retained on each sieve.

Duplicate 100 g sample is dried in an oven for 24 hours at 105^{0} C to calculate the oven dry weight of the soil sample.

Calculation

i. Weight of Aggregates in each sieve group = (Wt. of Aggregates + Can) – Wt. of Can

ii. Percent distribution of Aggregates in each size group

$$\frac{\text{Weight of Aggregates in each size group}}{\text{Total weight of soil}} \times 100$$

iii. $$\text{Oven - dry wt. of Aggregate}(\%) = \frac{\text{Air - dry wt.}(\%) \times 100}{100 + \text{Moisture}\%}$$

Wet Aggregate Analysis (Gupta and Ghil Dyal, 1998)

Apparatus

- A mechanical oscillator powered by a gear reduction motor having amplitude of oscillation 3.8 cm and frequence of oscillator 30-35 cycles per minute
- Two sets of sieves, each having 20 cm diameter and 5 cm height with screen openings of 5.0, 2.0, 1.0, 0.5, 0.25 and 0.1 mm diameter
- Two buckner funnels 15 cm in diameter with rubber stoppers
- Two vacuum flasks of one litre capacity
- Suction pump or aspirator
- Rubber policeman
- Twelve aluminium cans
- Perforated cans
- Sand bath
- Filter papers

Reagents

- 5% Sodium hexametaphosphate
- 4% Sodium hydroxide

Procedure

Among the different procedures adopted, wetting the samples under vaccum is suggested because the rate of wetting influences slaking of crumbs. The time of sieving ranges from 10 minutes to 30 minutes depending upon the type of wetting. Ten minutes pre-shaking of the soil sample in a reciprocating shaker or end-to-end shaker has been suggested by Baver and Rhodes (1932) for fine textured soil.

The technique used by Yoder (1936) and subsequently improved by the Soil Science Society of America's Committee on Physical Analysis is generally used for determining the size distribution of water stable aggregates. The soil sample is taken when it is moist and friable. It is broken by applying mild stress into smaller aggregates which can pass through 8 mm screen. The sieved soil sample is taken on a watch glass for wetting by either vacuum soaking or immersion method. For vacuum wetting, the sample is placed in a vacuum desiccator containing de-aerated water at the bottom. The desiccator is evacuated until the pressure inside drops to about 3 mm and water starts boiling. Water is then allowed to enter through the top of the desiccator and to flow into the watch-glass

holding the sample. Enough water is added to cover the soil sample. Soil sample is then taken out of desiccator.

Prepare four soil samples of 25 g each, Place a set of duplicate sample in an oven for the determination of moisture content. Another set of saturated duplicate soil sample is then transferred to the top sieve of the nest of sieves (5.0, 2.0, 1.0, 0.5, 0.25 and 0.1 mm) and spread with the help of a glass rod and a slow jet of water. The bottom pan is then removed, and the nest of sieves is attached to the Yoder type wet sieve shaker. Fill the drum (which holds the set of sieves) with salt-free water at 20-25^0 C to a level somewhat below that of the screen in the top sieve of the nest of sieves, when the sieves are in the highest position. Then lower the nest of sieves to wet the soil for 10 minutes. Bring the nest of sieves to the initial position and adjust the level of water so that the screen in the top sieve is covered with water in its highest position. Now switch on the mechanical oscillator to move the nest of sieves up and down with a frequency of 30-35 cycles per minute and a stroke of 3.8 cm. Sieving is done for 10 minutes. Remove the nest of screens from the water and allow it to drain for some time. Transfer the soil resting on each screen with a stream of distilled water and brush into a Buckner funnel having a pre-weighed filter paper and connected to a suction pump. Transfer the soil along with the filter paper into an aluminium can and dry at 105^0 C for 24 hours. Weigh the soil nearest to 0.01 g.

Transfer the oven dry soil aggregates from all the cans of a set into the dispersion cup. Add dispersing agent (10 ml of 5% solution of sodium hexametaphosphate for normal and Ca-saturated soils, or 10 ml of 4% solution of sodium hydroxide for acid soil) and enough distilled water to fill the cup within 4 cm of the rim, and then stir the suspension for 10 minutes. Wash the suspension on an identical set of sieves as used previously by means of a stream of tap water and a brush and transfer it to aluminium cans. The sand in each can is oven dried and weighed in the same manner as above. Calculate the percent distribution of soil particles (aggregates and the sand) and the sand particles retained on each sieve.

Calculation

Size distribution of soil particles (aggregate + sand):

Soil particles in each size group (%) = $$\frac{W_{od}\ (ag+s)\ i \times 100}{W_{od}}$$

Sand particle in each size group (%) = $$\frac{Wod\ (s)\ i \times 100}{Wod}$$

where,

W_{od} is the oven dried weight of the aggregates (ag) and Sand (s), and i is the size group.

3. Cation Exchange Capacity (CEC)

The total number of exchangeable cations a soil can hold is called cation exchange capacity (CEC). The higher the CEC, the more cations it can

retain. It can be expressed in terms of milliequivalents/100 g of soil (me/100 g) or centimoles of positive charge per kg of soil (cmol/kg), which is numerically equal to me/100 g. The CEC of the soil depends on the kind of clay and organic matter present.

Apparatus

- Centrifuge
- 50 ml round bottom centrifuge tubes
- Mechanical shaker
- Flame Photometer and accessories which include Propane, Lithium and Sodium standards

Reagents

- Sodium acetate (NaOAc) 1.0M: Dissolve 136.08 g of sodium acetate trihydrate in distilled water and bring the volume to 1 litre. Adjust the pH to about 8.2.
- Ethanol 95%.
- Ammonium acetate (NH_4OAc) 1.0M: Dissolve 77.09 g of ammonium acetate in distilled water and dilute to approx. 900 ml. Adjust pH to 7.0 with dilute ammonium hydroxide or acetic acid as required and make up the volume to 1 litre.
- Standard solution of NaCl: Dissolve 5.845 g of AR grade NaCl in 1.0M ammonium acetate and make the volume to 1 litre. It will give 100 meq/litre of sodium in stock solution. From this solution take 0, 1, 2, 5, 7.5 and 10 ml and make up the volume to 100 ml each with the ammonium acetate. It will give 0, 1, 2, 5, 7.5 and 10 meq/litre of sodium.

Procedure

1. Weigh accurately 5 g soil and transfer the sample to a 50 ml centrifuge tube.
2. Add 25 ml of 1.0M sodium acetate solution to the tube, stopper and shake in a mechanical shaker for 5 minutes.
3. Centrifuge at 2000 rpm for 5 minutes or until the supernatant liquid is clear.
4. Decant the liquid completely and repeat the extraction three more times. Discard the decants.
5. Repeat steps 2 – 4 with ethanol or isopropyl alcohol until the EC of the decant reads less than 40 mS/cm(usually it takes 4 to 5 washings).
6. To displace the adsorbed Na, repeat steps 2 – 4 using the ammonium acetate solution. Collect the decant in 100 ml volumetric flask fitted with a funnel and filter paper. Make up to volume with ammonium acetate solution.
7. To determine sodium concentration by flame photometry, prepare a series of Na standard solutions in the range of 0 – 10 meq/litre of Na. Prepare a standard curve by plotting sodium concentration on x-axis and flamephotometric readings on y-axis. Unknown sample extract is fed on the flamephotometer and the reading is taken, corresponding to which the concentration of sodium is read from the standard curve. For

better results, add LiCl in each standard to yield a final concentration of about 5 meq/litre of LiCl.

Calculation

Ammonium acetate extractable Na which is exchangeable Na in meq/100 g soil =

$$\frac{\text{Na coc. of extract in meq/litre (Y) x 100}}{\text{Wt. of soil in g (5)}} \text{ x } \frac{\text{Vol. of extract in ml (100)}}{1000} = \frac{\text{Y x 10}}{5} = 2\,\text{Y}$$

This displaced Na is actually a measure of the Cation Exchange Capacity (CEC) of the soil. So, the me Na/100 g soil is actually me exchangeable cations (Ca, Mg, Na and K)/100 g soil.

4. Soil Moisture

Gravimetric method of moisture estimation is most widely used where the soil sample is placed in an oven at 105^0 C and dried to a constant weight. The difference in weight is considered to be water present in the soil sample.

Apparatus

- Aluminium Moisture Box
- Oven
- Desiccator

Procedure

1. Take 100 g of soil sample in the aluminium moisture box and keep in the oven after removing the lid of the box.
2. The sample is kept at 105^0 C till it attains a constant weight. It may take 24-36 hours.
3. Cool the sample, first in the switched off oven and then in a desiccator.
4. Take the weight of the cooled moisture box. The loss in weight is equal to moisture contained in 100 g soil sample.

Calculation

$$\text{Moisture}(\%) = \frac{\text{Loss in weight}}{\text{Oven - dry weight of soil}} x\,100$$

The corresponding moisture correction factor *(mcf)* for analytical results or the multiplication factor for the amount of sample to be weighed in for analysis is:

$$\text{Moisture correction factor} = \frac{100 + \%\ \text{moisture}}{100}$$

5. Water Holding Capacity (WHC)

Veihmeyer and Hendrickson (1931) defined the field capacity or the water holding capacity as the amount of water held in the soil after the excess gravitational water has drained away and after the rate of downward movement of water has materially ceased. Stage of field capacity is attained in the field after 48 to 72 hours of saturation. It is the upper limit of plant available soil moisture.

Apparatus

- Polythene sheets
- Spade
- Soil auger
- Moisture boxes/cans
- Balance
- Oven

Procedure

1. Select a uniform plot measuring 5 m x 5 m.
2. Remove weeds, pebbles etc. and make bunds around the plot.
3. Fill sufficient water in the plot to completely saturate the soil.
4. Cover the plot area with a polythene sheet to check evaporation.
5. Take soil sample from the centre of the plot from the desired layer, starting after 24 hours of saturation and determine moisutre content daily till the values of successive days are nearly equal.
6. Record the weight as below:
   - Weight of empty moisture box = X
   - Weight of moisture box + moist soil = Y
   - Weight of moisture box + oven dry soil = Z
   - Repeat above on next day and so on till a constant Z value is reached.

Calculation

Moisture content in soil = Y – Z
Weight of oven dry soil = Z – X

$$\text{Percentage of moisture in soil (1st day)} = \left(\frac{Y-Z}{Z-X}\right) x\,100 = a$$

Percentage of moisture on succeeding days = a_1, a_2, etc.

Plot the daily readings on a graph paper. The lowest reading is taken as a value of field capacity of the soil.

6. pH

The soil pH is the negative logarithm of the active hydrogen ion (H^+) conc. in the soil solution. It is the measure of soil sodicity, acidity or neutrality. It is a simple but very important estimation for soils, since soil pH influences to a great extent the availability of nutrients to crops. It also affects microbial population in soils. Most nutrient elements are available in the pH range of 5.5 to 6.5.

In various chemical estimations, pH regulation is critical. Specific colours as observed in the presence of various pH indicators and the colour changes due to pH change are shown in **Annexure-11**. The procedure for measurement of soil pH is given below.

Apparatus

- pH meter with a range of 0-14 pH
- Pipette/dispenser
- Beaker
- Glass rod

Reagent

- Buffer solutions of pH 4, 7 and 9
- Calcium chloride solution (0.01M): Dissolve 14.7 g $CaCl_2.2H_2O$ in 10 litre of water to obtain 0.01M solution.

Procedure

1. Calibrate the pH meter, using 2 buffer solutions, one should be the buffer with neutral pH (7.0) and the other should be chosen based on the range of pH in the soil. Take the buffer solution in the beaker. Insert the electrode alternately in the beakers containing 2 buffer solutions and adjust the pH. The instrument indicating pH as per the buffers is ready to test the samples
2. Weigh10.0g of soil sample into 50 or 100 ml beaker, add 20ml of $CaCl_2$ solution (use water instead of $CaCl_2$ solution throughout the procedure if water is used as a suspension medium).
3. Allow the soil to absorb $CaCl_2$ solution without stirring, then thoroughly stir for 10 second using a glass rod.
4. Stir the suspension for 30 minutes and record the pH on the calibrated pH meter.

Table 14 : Based on soil pH values, following types of soil reactions are distinguished:

PH Range	Soil Reaction Rating
<4.6	Extremely acid
4.6-5.5	Strongly acid
5.6-6.5	Moderately acid

Table 4 in terms of $CaCO_3$. The decrease of buffer pH by 0.1 unit is equivalent to 1 meq of H^+ in 100 ml buffer solution. The lime requirement varies with the type of soils and their cation exchange capacity.

TABLE 16 Lime requirement for different pH targets

Measured pH of soil buffer suspension ↓ pH →	Lime Requirement in tonnes/ha as $CaCO_3$ for bringing soil pH to different levels		
	6.0	6.4	6.8
6.7	2.43	2.92	3.40
6.6	3.40	4.13	4.62
6.5	4.37	5.35	6.07
6.4	5.59	6.56	7.53
6.3	6.65	7.78	8.99
6.2	7.52	8.93	10.21
6.1	8.5	10.21	11.66
6.0	9.48	11.42	13.12

Practically, pH of acid soils may not be raised beyond 6.4/6.5.

8. Soil Electrical Conductivity(EC) and Gypsum requirement

The soils having pH value more than 8.0-8.5 may have the following special features:

- Presence of excessive amounts of soluble salts.
- Presence of excessive amounts of sodium on the exchange complex.

The chemical properties of salt affected soils are summarized in **Table 17.**

TABLE 17

Chemical characteristics of saline, non-saline sodic and saline sodic soils

Soil	EC (dS/m)	ESP	pH
Saline	>4.0	<15	<8.5
Sodic (non-saline)	<4.0	>15	>8.5
Saline Sodic	>4.0	>15	<8.5

Source: Richards, 1954

Such soils are generally not considered suitable for growing most of the crops unless treated with suitable amendment materials. However, there are salt tolerant crops which could be grown on these soils.

To determine the quality of these soils, the following estimations are required:

- PH (as described before)
- Salt content or electrical conductivity
- Exchangeable sodium or gypsum requirement

8a. Electrical Conductivity (EC)

The electrical conductivity (EC) is a measure of the ionic transport in a solution between the anode and cathode. This means, the EC is normally considered to be a measurement of the dissolved salts in a solution. Like a metallic conductor, they obey Ohm's law.

Since the EC depends on the number of ions in the solution, it is important to know the soil/water ratio used. The EC of a soil is conventionally based on the measurement of the EC in the soil solution extract from a saturated soil paste, as it has been found that the ratio of the soil solution in saturated soil paste is approximately two-three times higher than that at field capacity.

As the determination of EC of soil solution from a saturated soil paste is cumbersome and demands 400-500 g soil sample for the determination, a less complex method is normally used. Generally a 1:2 soil/water suspension is used.

Apparatus

- EC meter
- Beakers (25 ml), erlenmeyer flasks (250 ml) and pipettes
- Filter paper

Reagent

- 0.01M Potassium chloride solution: Dry a small quantity of AR grade potassium chloride at 60^0 C for two hours. Weight 0.7456 g of it and dissolve in freshly prepared distilled water and make the volume to one litre. This solution gives an electrical conductivity of 1411.8×10^{-3} i.e. 1.412 mS/cm at 25^0C. For best result, select a conductivity standard (KCl solution) close to the sample value.

Procedure

1. Take 40 g soil into 250 ml Erlenmeyer flask, add 80 ml of distilled water, stopper the flask and shake on reciprocating shaker for one hour. Filter through Whatman No.1 filter paper. The filtrate is ready for measurement of conductivity.
2. Wash the conductivity electrode with distilled water and rinse with standard KCl solution.

3. Pour some KCl solution into a 25 ml beaker and dip the electrode in the solution. Adjust the conductivity meter to read 1.412 mS/cm, corrected to 25^0 C.
4. Wash the electrode and dip it in the soil extract.
5. Record the digital display corrected to 25^0 C. The reading in mS/cm of electrical conductivity is a measure of the soluble salt content in the extract, and an indication of salinity status of this soil (Table 18). The conductivity can also be expressed as mmhos/cm.

TABLE 18 General interpretation of EC values

Soil	EC (mS/cm)	Total salt content (%)	Crop reaction
1. Salt free	0-2	<0.15	Salinity effect negligible, except for more sensitive crops
2. Slightly saline	4-8	0.15-0.35	Yield of many crops restricted
3. Moderately saline	8-15	0.35-0.65	Only tolerant crops yield satisfactorily
4. Highly saline	>15	>0.65	Only very tolerant crops yield satisfactorily

8b. Gypsum requirement (Schoonover, 1952)

In the estimation of gypsum requirement of saline-sodic/sodic soils, the attempt is to measure the quantity of gypsum (Calcium sulphate) required to replace the sodium from the exchange complex. The sodium so replaced with calcium of gypsum is removed through leaching of the soil. The soils treated with gypsum become dominated with calcium in the exchange complex.

When Calcium of the gypsum is exchanged with sodium, there is reduction in the calcium concentration in the solution. The quantity of calcium reduced is equivalent to the calcium exchanged with sodium. It is equivalent to gypsum requirement of the soil when 'Ca' is expressed as $CaSO_4$.

Apparatus

- Mechanical shaker
- Burette – 50 ml
- Pipettes – 100 ml and 5 ml

Reagents

- Saturated gypsum (calcium sulphate) solution: Add 5 g of chemically pure $CaSO_4.2H_2O$ to one litre of distilled water. Shake vigorously for 10

minutes using a mechanical shaker and filter through Whatman No.1 filter paper.

- 0.01N $CaCl_2$ solution: Dissolve exactly 0.5 g of AR grade $CaCO_3$ powder in about 10 ml of 1:3 diluted HCl. When completely dissolved, transfer to 1 litre volumetric flask and dilute to the mark with distilled water. $CaCl_2$ salt should not be used as it is highly hygroscopic.
- 0.01N Versenate solution: Dissolve 2.0 g of pure EDTA – disodium salt and 0.05 g of magnesium chloride (AR grade) in about 50 ml of water and dilute to 1 litre. Titrate a portion of this against 0.01N of $CaCl_2$ solution to standardize.
- Eriochrome Black T (EBT) indicator: Dissolve 0.5 g of EBT dye and 4.5 g of hydroxylamine hydrochloride in 100 ml of 95% ethanol. Store in a stoppered bottle or flask.
- Ammonium hydroxide-ammonium chloride buffer: Dissolve 67.5 g of pure ammonium chloride in 570 ml of conc. ammonium hydroxide and dilute to 1 litre. Adjust the pH at 10 using dilute HCl or dilute NH_4OH.

Procedure

1. Weigh 5 g of air-dry soil in 250 ml conical flask.
2. Add 100 ml of the saturated gypsum solution. Firmly put a rubber stopper and shake for 5 minutes.
3. Filter the contents through Whatman No.1 filter paper.
4. Transfer 5 ml aliquot of the clear filtrate into a 100 or 150 ml porcelain dish.
5. Add 1 ml of the ammonium hydroxide-ammonium chloride buffer solution and 2 to 3 drops of Eriochrome black T indicator.
6. Take 0.01N versenate solution in a 50 ml burette and titrate the contents in the dish until the wine red colour starts changing to sky blue. Volume of versenate used = B.
7. Run a blank using 5 ml of saturated gypsum solution in place of sample aliquot. Volume of versenate solution used = A.

Calculation

Gypsum requirement (tonnes/ha) = (A – B) x N x 382

where,
A = ml of EDTA (versenate) used for blank titration
B = ml of ETDA used for soil extract
N = Normality of EDTA solution

9. Organic Carbon/Organic Matter

Organic matter estimation in the soil can be done by different methods. Loss of weight on ignition can be used as a direct measure of the organic matter contained in the soil. It can also be expressed as the content of organic carbon in the soil. It is generally assumed that on an average organic matter contains about 58% organic carbon. Organic

matter/organic carbon can also be estimated by volumetric and colorimetric methods. However, the use of potassium dichromate ($K_2Cr_2O_7$) involved in these estimations is considered as a limitation because of its hazardous nature. Soil organic matter content can be used as an index of N availability (potential of a soil to supply N to plants) because the content of N in soil organic matter is relatively constant.

Loss of weight on ignition

Apparatus

- Sieve
- Beaker
- Oven
- Muffle furnace

Procedure

1. Weigh 5.0 to 10.0 g (to the nearest 0.01 g) sieved (2 mm) soil into an ashing vessel (50 ml beaker or other suitable vessel).

2. Place the ashing vessel with soil into a drying oven set at 105^0 C and dry for 4 hours. Remove the ashing vessel from the drying oven and place in a dry atmosphere. When cooled, weigh to the nearest 0.01 g. Place the ashing vessel with soil into a muffle furnace and bring the temperature to 400^0 C . Ash in the furnace for 4 hours. Remove the ashing vessel from the muffle furnace, cool in a dry atmosphere and weigh to the nearest 0.01 g.

Calculation

$$\text{Percent organic matter (OM)} = \frac{(W_1 - W_2)}{W_1} \text{ x } 100$$

Percent organic C = % OM x 0.58

Where,

W_1 is the weight of soil at 105^0 C and W_2 is the weight of soil at 400^0 C.

Volumetric method (Walkley and Black, 1934)

Apparatus

- Conical flask - 500 ml
- Pipettes - 2, 10 and 20 ml
- Burette - 50 ml

Reagents

- Phosphoric acid – 85%
- Sodium fluoride solution – 2%
- Sulphuric acid – 96 % containing 1.25% Ag_2SO_4
- Standard 0.1667M $K_2Cr_2O_7$: Dissolve 49.04 g of $K_2Cr_2O_7$ in water and dilute to 1 litre
- Standard 0.5M $FeSO_4$ solution: Dissolve 140 g Ferrous Sulphate in 800 ml water, add 20 ml concentrated H_2SO_4 and make up the volume to 1 litre
- Diphenylamine indicator: Dissolve 0.5 g reagent grade diphenylamine in 20 ml water and 100 ml concentrated H_2SO_4.

Procedure

1. Weigh 1.0 g of the prepared soil sample in 500 ml conical flask.
2. Add 10 ml of 0.1667M $K_2Cr_2O_7$ solution and 20 ml concentrated H_2SO_4 containing Ag_2SO_4.
3. Mix thoroughly and allow the reaction to complete for 30 minutes.
4. Dilute the reaction mixture with 200 ml water and 10 ml H_3PO_4.
5. Add 10 ml of NaF solution and 2 ml of diphenylamine indicator.
6. Titrate the solution with standard 0.5M $FeSO_4$ solution to a brilliant green colour.
7. A blank without sample is run simultaneously.

Calculation

Percent organic Carbon (X) =

$$\frac{10\,(S - T) \times 0.003}{S} \times \frac{100}{\text{Wt. of soil}}$$

Since one gram of soil is used, this equation simplifies to:

$$\frac{3(S - T)}{S}$$

Where,
S = ml $FeSO_4$ solution required for blank
T = ml $FeSO_4$ solution required for soil sample
3 = Eq W of C (weight of C is 12, valency is 4, hence Eq W is 12÷4 = 3.0)
0.003 = weight of C (1 000 ml 0.1667M $K_2Cr_2O_7$ = 3 g C. Thus, 1 ml 0.1667M $K_2Cr_2O_7$ = 0.003 g C)
Organic Carbon recovery is estimated to be about 77%. Therefore, actual

amount of organic carbon (Y) will be:

$$\text{Percent value of organic carbon obtained} \times \frac{100}{77}$$

6.6-6.9	Slightly acid
7.0	Neutral
7.1-8.5	Moderately alkaline
>8.5	Strongly alkaline

The acidic soils need to be limed before they can be put to normal agricultural production. The alkali soils need to be treated with gypsum to remove the excessive content of sodium.

7. Lime Requirement

Crop yields are normally high in soils with pH values between 6.0 and 7.5. Lime is added to raise the pH of acid soils, and the amount of lime required to raise the pH to an optimum level is called as Lime Requirement. A number of methods are available for the determination of lime requirement. The Woodruff and the Shoemaker *et al* methods are discussed here which are based on the use of a buffer solution, whose pH undergoes change when treated with acid soils. The pH of buffer solution will gradually decrease when H^+ ion concentration increases. When H^+ increases by 1 meq in 100 ml buffer solution, pH value will decrease by 0.1 unit.

Buffer solutions needs to be prepared afresh. A 0.05M solution of AR grade potassium hydrogen pthalate (molecular weight 204.22) gives a pH of 4.0 at 25^0 C and it can be used as a buffer.

Woodruff's Method (Woodruff, 1948)

Apparatus

- pH meter
- Automatic pipettes

Reagent

- Woodruff's buffer solution: Dissolve 10 g calcium acetate [$Ca(CH_3COOH)_2$], 12 g para-nitrophenol, 10 g salicylic acid and 1.2 g sodium hydroxide in distilled water. Adjust pH to 7.0 with acetic acid or sodium hydroxide, transfer to 1 litre volumetric flask and make the volume to the mark with distilled water.

Procedure

1. Take 10 g soil sample in a clean 50 ml beaker.
2. Add 10 ml of distilled water, stir and wait for 30 minutes.
3. Determine pH value in soil suspension.
4. If the pH value is less than 5.0 (average of 4.5 and 5.5 to have one value), add 10 ml Woodruff's buffer solution, stir and wait for 30 minutes before determining new pH value.

The amount of lime required to raise the pH for agricultural purpose is shown in **Table 15.**

TABLE 15 pH and quantity of lime required to reduce soil acidity (tonnes/ha)

pH (after buffer)	$CaCO_3$	$Ca(OH)_2$	Marl	Limestone	Dolomite
6.5	6.00	4.68	7.20	9.00	6.54
6.4	7.20	5.62	8.64	10.80	7.85
6.3	8.40	6.55	10.08	12.60	9.16
6.2	9.60	7.49	11.52	14.40	10.46
6.1	10.80	8.42	12.96	16.20	11.77
6.0	12.00	9.36	14.40	18.00	13.08
5.9	13.20	10.30	15.84	19.80	14.39
5.8	14.40	11.23	17.28	21.60	15.70
5.7	15.60	12.17	18.72	23.40	17.00
5.6	16.80	13.10	20.16	25.20	18.31

The soils between pH 6.6 and 7.5 are practically considered as nearly neutral. Such soils do not need to be treated with lime or gypsum. Even in case of soils, which are acidic and alkaline beyond these limits, growing of acid loving and salt tolerant crops may be considered. Only highly acidic soils and the soils with high alkalinity need to be treated with chemical amendments since this operation is quite expensive.

Shoemaker Method (Shoemaker *et al.*, 1961)

Apparatus

- pH meter
- Automatic pipettes - 10 and 20 ml

Reagent

- Extractant Buffer: Dissolve 1.8 g of nitrophenol, 2.5 ml triethanolamine, 3.0 g potassium chromate (K_2CrO_4), 2.0 g calcium acetate and 53.1 g calcium chloride in a litre of water. Adjust pH to 7.5 with NaOH.

Procedure

1. Take 5.0 g soil sample in a 50 ml beaker.
2. Add 5 ml distilled water and 10 ml extractant buffer.
3. Shake continuously for 10 minutes or intermittently for 20 minutes and read the pH of the soil buffer suspension with glass electrode. The pH of the buffer solution is reduced, depending upon the extent of soil acidity.

For various levels of measured pH of soil buffer suspesion, the amount of lime required to raise the soil pH to 6.0, 6.4 and 6.8 is given in

Or Percentage value of organic carbon x 1.3

Percent Organic matter =
Y x 1.724 (organic matter contains 58 % organic carbon, hence 100/58 = 1.724)

Note: Published organic C to total organic matter conversion factor for surface soils vary from 1.724 to 2.0. A value of 1.724 is commonly used, although whenever possible the appropriate factor be determined experimentally for each type of soil.

Colorimetric method (Datta *et al.*, 1962)

Apparatus

- Spectrophotometer
- Conical flask -100 ml
- Pipettes - 2 , 5 and 10 ml

Reagents

- Standard potassium dichromate 1/6M (1N)
- Concentrated sulphuric acid containing 1.25% Ag_2SO_4
- Sucrose (AR quality)

Procedure

1. **Preparation of standard curve:** Sucrose is used as a primary standard as carbon source. Take different quantities of sucrose (1 mg to 20 mg) in 100 ml flasks. Add 10 ml standard $K_2Cr_2O_7$ and 20 ml of concentrated H_2SO_4 in each flask. Swirl the flasks and leave for 30 minutes. A blank is also prepared in the similar way without adding sucrose. Green colour develops which is read on spectrophotometer at 660 nm, after adjusting the blank to zero. The reading so obtained is plotted against mg of sucrose as carbon source (carbon = wt. of sucrose x 0.42 because carbon content of sucrose is 42%) or against mg C directly. A standard curve as prepared for estimation of organic carbon by Motsara and Roy (2008) while setting up a soil testing laboratory in DPR Korea is given in Figure 2, as an example, which shows the accuracy of the method (the r^2 is as high as 0.991). For convenience, the curve is shown directly against C content, which has been derived from mg sucrose used in preparing the standard curve.

Procedure

1. Take 1 g of soil in 100 ml conical flask.
2. Add 10 ml of 0.1667M $K_2Cr_2O_7$ and 20 ml of conc. H_2SO_4 containing 1.25 percent of Ag_2SO_4.
3. Stir the reaction mixture and allow to stand for 30 minutes.
4. The green colour of chromium sulphate so developed is read on a spectrophotometer at 660 nm after setting the blank, prepared in the similar manner, at zero.

Calculation

The carbon content of the sample is found out from the standard curve

which shows the carbon content (mg of carbon v/s spectrophotometer

readings as absorbance).

Percent C = mg C observed x 100/1000 (observed reading is for 1 g soil, expressed as mg).
Percent OM = %C x 1.724

10. Total Nitrogen (Kjeldahl Method)

Total N includes all forms of inorganic N, like NH_4 –N, NO3 -N and also NH_2 (Urea) –N, and the organic N compounds like proteins, amino acids and other derivatives. Depending upon the form of N present in a particular sample, specific method is to be adopted for getting the total nitrogen value. While the organic N materials can be converted into simple inorganic ammoniacal salt by digestion with sulphuric acid, for reducing nitrates into ammoniacal form, use of salicylic acid or Devarda's alloy is

made in the modified Kjeldahl method At the end of digestion, all organic and inorganic salts are converted into ammonium form which is distilled and estimated by using standard acid.

As the precision of the method depends upon complete conversion of organic N into NH_4 - N, the digestion temperature and time, solid:acid ratio and the type of catalyst used have an important bearing on the method. The ideal temperature for digestion is 320° – 370° C. At lower temperature, the digestion may not be complete, while above 410° C, the loss of NH_3 may occur. The salt:acid (weight:volume) ratio should not be less than 1:1 at the end of digestion. Commonly used catalysts to hasten the digestion process are $CuSO_4$ or Hg. Potassium sulphate is added to raise the boiling point of the acid so that loss of acid by volatilization is prevented.

Apparatus

- Kjeldahl digestion and distillation unit
- Conical flasks
- Burettes
- Pipettes

Reagents

- Sulphuric acid – H_2SO_4 (93-98%)
- Copper sulphate – $CuSO_4H_2O$ (AR grade)
- Potassium sulphate or anhydrous sodium sulphate (AR grade)
- 35% sodium hydroxide solution: Dissolve 350 g solid NaOH in water and dilute to one litre
- 0.1M NaOH: Prepare 0.1M NaOH by dissolving 4.0 g NaOH in water and make volume to 1 litre. Standardize against 0.1N potassium hydrogen phthalate or standard H_2SO_4
- 0.1M HCl or 0.1M H_2SO_4: Prepare approximately 0.1M acid solution and standardize against 0.1M sodium carbonate
- Methyl red indicator
- Salicyclic acid for reducing NO_3 to NH_4, if present in the sample
- Devarda's alloy for reducing NO_3 to NH_4, if present in the sample.

Procedure

1. Weigh 1 g sample of soil. Place in Kjeldahl flask .
2. Add 0.7 g copper sulphate, 1.5 g K_2SO_4 and 30 ml H_2SO_4.
3. Heat gently until frothing ceases. If necessary, add small amount of paraffin or glass beads to reduce frothing.
4. Boil briskly until solution is clear and then continue digestion for at least 30 minutes.
5. Remove the flask from the heater and cool, add 50 ml water and transfer to distilling flask.
6. Take accurately 20–25 ml standard acid (0.1M HCl or 0.1M H_2SO_4) in the receiving conical flask so that there will be an excess of at least 5 ml of the acid. Add 2-3 drops of methyl red indicator. Add enough water to cover the end of the condenser outlet tubes.

7. Add 30 ml of 35% NaOH in the distilling flask in such a way that the contents do not mix.
8. Heat the contents to distil the ammonia for about 30-40 minutes.
9. Remove receiving flask and rinse outlet tube into receiving flask with a small amount of distilled water.
10. Titrate excess acid in the distillate with 0.1M NaOH.
11. Determine blank on reagents using same quantity of standard acid in a receiving conical flask.

Calculation

$$\text{Percent N} = \frac{1.401\ (V_1M_1 - V_2M_2) - (V_3M_1 - V_4M_2)}{W} \times df$$

Where,

V_1 - ml of standard acid taken in receiving flask for samples
V_2 - ml of standard NaOH used in titration
V_3 - ml of standard acid taken to receiving flask for blank
V_4 - ml of standard NaOH used in titrating blank
M_1 - Molarity of standard acid
M_2 - Molarity of standard NaOH
W - Weight of sample taken (1 g)

df - Dilution factor of sample (if 1 g was taken for estimation, the dilution factor will be 100).

Note : 1000 ml of 0.1 M HCl or 0.1 M H2SO4 = 1.401 g Nitrogen

Precautions

- The material after digestion should not solidify.
- No NH_4 should be lost during distillation.
- If the indicator changes colour during distillation, determination must be repeated using either a smaller sample weight or a larger volume of standard acid.

11. Mineralizable Nitrogen (Subbia And Asija, 1956)

In case of soils, mineralizable N (also organic C) is estimated as an index of available nitrogen content and not the total nitrogen content. The easily mineralizable nitrogen is estimated using alkaline $KMnO_4$, which oxidizes and hydrolyses the organic matter present in the soil. The liberated ammonia is condensed and absorbed in boric acid, which is titrated against standard acid. The method has been widely adopted to get a reliable index of nitrogen availability in soil due to its rapidity and reproducibility. The process of oxidative hydrolysis is, however, a progressive one and thus, a uniform time and heating temperature should be allowed for best results. Use of glass beads checks bumping while liquid paraffin checks frothing during heating as is recommended in total N estimation by Kjeldahl method.

Apparatus

- .Nitrogen distillation unit, preferably with six regulating heating elements.
- Conical flasks, pipettes, burette, etc.

Reagents

- 0.32% $KMnO_4$: Dissolve 3.2 g of $KMnO_4$ in water and make the volume to one litre.
- 2.5% NaOH: Dissolve 25 g of sodium hydroxide pellets in water and make the volume to one litre.
- 2% Boric acid: Dissolve 20 g of boric acid powder in warm water by stirring and dilute to one litre.
- Mixed Indicator: Dissolve 0.066 g of methyl red and 0.099 g of bromocresol green in 100 ml of ethyl alcohol. Add 20 ml of this mixed indicator to each litre of 2% boric acid solution.
- 0.1M Potassium Hydrogen Phthalate: Dissolve 20.422 g of the salt in water and dilute to one litre. This is a primary standard and does not require standardization.
- 0.02M H_2SO_4: Prepare approximately 0.1M H_2SO_4 by adding 5.6 ml of conc. H_2SO_4 to about 1 litre of distilled water. From this, prepare 0.02M H_2SO_4 by diluting a suitable volume (20 ml made to 100 ml) with distilled water. Standardize it against 0.1M NaOH solution.
- 0.1M NaOH. Dissolve 4gm NaOH in 100 ml distilled water. Standardize against potassium hydrogen phthalate.

Procedure

1. Weigh 20 g of soil sample in a 800 ml Kjeldahl flask.
2. Moisten the soil with about 10 ml of distilled water, wash down the soil, if any, adhering to the neck of the flask.
3. Add 100 ml of 0.32% of $KMnO_4$ solution.
4. Add a few glass beads or broken pieces of glass rod.
5. Add 2-3 ml of paraffin liquid, avoiding contact with upper part of the neck of the flask.
6. Measure 20 ml of 2% boric acid containing mixed indicator in a 250 ml conical flask and place it under the receiver tube. Dip the receiver tube in the boric acid.
7. Run tap water through the condenser.
8. Add 100 ml of 2.5% NaOH solution and immediately attach to the rubber stopper fitted in the alkali trap.
9. Switch the heaters on and continue distillation until about 100 ml of distillate is collected.
10. First remove the conical flask containing distillate and then switch of the heater to avoid back suction.
11. Titrate the distillate against 0.02M H_2SO_4 taken in burette until pink colour starts appearing.
12. Run a blank without soil.

13. Carefully remove the Kjeldahl flask after cooling and drain the contents in the sink.

Calculation

Volume of acid used to neutralize ammonia in the sample = A – B ml

N content in the test sample = (A – B) x 0.56mg

Percent Nitrogen (A – B) x 0.56 x 5

Where,

A = Volume of 0.02M H_2SO_4 used in titration against ammonia absorbed in boric acid.

B = Volume of 0.02M sulphuric acid used in blank titration.

1 ml of 0.02M sulphuric acid = 0.56 mg N (1 000 ml of 1M H_2SO_4 = 14 g Nitrogen).

Wt. of soil sample = 20 g. Thus, factor for converting into % Nitrogen = 100/20 = **5**

Caution

- Check all the joints of the Kjeldahl apparatus to prevent any leakage and loss of ammonia.
- Hot Kjeldahl flasks should neither be washed immediately with cold water nor allowed to cool for long to avoid deposits to settle at the bottom which are difficult to remove.
- In case frothing takes place and passes through to the boric acid, such samples should be discarded and fresh distillation done.
- Opening ammonia bottles in the laboratory should be strictly prohibited while distillation is on. The titration should be carried out in ammonia free atmosphere.
- In case the titration is not to be carried out immediately, the distillate should be stored in ammonia free cupboards after tightly stoppering the flasks

12. Inorganic Nitrogen - NO_3^- And NH_4^+

Inorganic N in soil is present predominantly as NO_3^- and NH_4^+. Nitrite is seldom present in detectable amount, and its determination is normally unwarranted except in neutral to alkaline soils following the application of NH_4 or NH_4-forming fertilizers.

Nitrate is highly soluble in water, and a number of solutions including water have been used as extractants. These include saturated 0.35% $CaSO_4$ $2H_2O$ solution, 0.03M NH_4F, 0.015M H_2SO_4, 0.01M $CaCl_2$, 0.5M $NaHCO_3$ (pH 8.5), 0.01M $CuSO_4$, 0.01M $CuSO_4$ containing Ag_2SO_4 and 2.0M KCl.

Exchangeable NH_4 is defined as NH_4 that can be extracted at room temperature with a neutral K salt solution. Various molarities have been used, such as 0.05M K_2SO_4, 0.1M KCl, 1.0M KCl, and 2.0M KCl.

The potential of a soil to mineralize N as measured by N availability indexes (OM, OC and even total N) is fairly constant from year to year, making it unnecessary to make that type of determination each year. However, it is still necessary to take into consideration the initial amount of available N (inorganic N: NO_3 – N and/or NH_4 – N) in the rooting zone at or near planting time for better prediction of N fertilizer need. In contrast to N index tests, this type tests must be made each year, specially when there is possibility of residual inorganic N remaining from previous application or fallow period.

The methods for the determination of NO_3 - N and NH_4 - N are even more diverse than the methods of extraction (Keeney and Nelson 1982). These range from specific ion electrode to colorimetric techniques, microdiffusion, steam distillation, and flow injection analysis. Steam distillation is still a preferred method when using ^{15}N. However, for routine analysis phenoldisulfonic acid method for NO3 and indophenol blue method for NH4 estimation have been described

Nitrate by phenoldisulfonic acid method

One of the major difficulties in estimating NO_3 in soils by colorimetric methods is obtaining a clear colourless extract with low contents of organic and inorganic substances which interfere with the colorimetric method. In arid and salt affected soils, chloride (Cl) is the major anion which interferes with color development of the phenoldisulfonic acid method. Therefore, if chloride concentration is more than 15 µg/g soil, it should be removed before analysis by the use of Ag_2SO_4 to precipitate chloride as AgCl. The Ag_2SO_4 is added to the extract or to the reagent used for extraction, and the AgCl is removed by filtration or centrifugation after precipitation of the excess Ag_2SO_4 by basic reagent such as $Ca(OH)_2$ or $MgCO_3$. It is necessary to remove the excess Ag^+ before analysis of the extract because it also interferes with the phenoldisulfonic acid method of determining NO_3.

Apparatus

- Reciprocating shaker
- Heavy-duty hot plate
- Spectrophotometer
- Dispenser
- Erlenmeyer flask
- Beakers
- Glass rod

Reagents

- Phenoldisulfonic acid (phenol 2,4-disulfonic acid): Transfer 70 ml pure liquid phenol (carbolic acid) to an 800 ml Kjeldahl flask. Add 450 ml

concentrated H_2SO_4 while shaking. Add 225 ml fuming H_2SO_4 (13-15% SO_3). Mix well. Place Kjeldahl flask (loosely stoppered) in a beaker containing boiling water and heat for 2 hours. Store resulting phenoldisulfonic acid [$C_6H_3OH(HSO_3)_2$] solution in a glass-stoppered bottle.

- Dilute ammonium hydroxide solution (about 7.5M NH_4OH): Mix one part NH_4OH (specif gravity 0.90) with one part H_2O.
- Copper sulfate solution (0.5M): Dissolve 125 g $CuSO_4.5H_2O$ in 1 litre of distilled water.
- Silver sulfate solution (0.6%): Dissolve 6.0 g Ag_2SO_4 in 1 litre of distilled water. Heat or shake well until all salt is dissolved.
- Nitrate-extracting solution: Mix 200 ml of 0.5M copper sulfate solution and 1 litre 0.6% silver sulfate solution and dilute to 10 litres with water. Mix well.
- Standard nitrate solution (100 µg NO_3-N/ml, stock solution): Dissolve 0.7221 g KNO_3 (oven dried at 105° C) in water and dilute to 1 litre. Mix thoroughly.
- Standard nitrate solution (10 µg NO_3-N/ml; working solution): Dilute 100 ml of 100 µgNO3-N/ml stock solution to 1 litre with water. Mix well.
- Calcium hydroxide, AR grade powder (free of NO_3).
- Magnesium carbonate, AR grade powder (free of NO_3).

Procedure

1. Place about 5 g soil in an Erlenmeyer flask.
2. Add 25 ml of nitrate-extracting solution.
3. Shake contents for 10 minutes.
4. Add about 0.2 g $Ca(OH)_2$ and shake for 5 minutes.
5. Add about 0.5 g $MgCO_3$ and shake for 10-15 minutes.
6. Allow to settle for a few minutes.
7. Filter through a Whatman filter paper No. 42.
8. Pipette 10 ml of clear filtrate into a 100 ml beaker. Evaporate to dryness on a hot plate at low heat in a fume hood (free of HNO_3 fumes). Do not continue heating beyond dryness.
9. When completely dry, cool residue, add 2 ml phenoldisulfonic acid rapidly (from a burette having the tip cut off) covering the residue quickly. Rotate the beaker so that reagent comes in contact with all residual salt. Allow to stand for 10-15 minutes.
10. Add 16.5 ml cold water. Rotate the beaker to dissolve residue (or stir with a glass rod until all residue is in solution).

11. After the beaker gets cool, add 15 ml.dilute NH_4OH slowly until the solution is distinctly alkaline as indicated by the development of a stable yellow color.
12. Add 16.5 ml water (volume becomes 50 ml). Mix thoroughly.
13. Read concentration of NO_3-N at 415 nm, using the standard curve.
14. Preparation of standard curve: Take 0, 2, 5, 8, and 10 ml of the 10 µg NO_3/ml working solution in respective 100 ml beakers, add 10 ml NO_3-extracting solution and evaporate to dryness. Follow steps 9 to 13, using these standard solutions having 0, 0.4, 1.0, 1.6 and 2.0 µg NO_3-N/ml. Prepare a standard curve to be used for estimation of NO_3 in the sample.

Calculation

$$NO_3 - N \text{ in test soln } (\mu g/ml) = \frac{\text{Vol. after colour development (ml)}}{\text{Vol. evaporated (ml)}} \times \frac{\text{Vol. of extracting soln. (ml)}}{\text{Wt. of oven-dried soil (g)}}$$

Ammonium by indophenol blue method

The phenol reacts with NH_4 in the presence of an oxidizing agent such as hypochlorite to form a coloured complex in alkaline condition. The addition of sodium nitroprusside as a catalyst in the reaction between phenol and NH_4 increases the sensitivity of the method by many folds. The addition of EDTA is necessary to complex divalent and trivalent cations present in the extract. Otherwise, it forms precipitate at the pH of 11.4 – 12 used for color development, and this turbidity would interfere with formation of the phenol – NH_4 complex.

The first step is the extraction of exchangeable Ammonium.

Apparatus

- Erlenmeyer flask
- Volumetric flask
- Shaker
- Spectrophotometer

Reagents

- Potassium chloride (KCl) solution, 2M: Dissolve 150 g AR grade KCl in 1 litre distilled water.
- Standard ammonium (NH_4^+) solution: Dissolve 0.4717 g of ammonium sulfate $(NH_4)_2SO_4$ in water, and dilute to a volume of 1 litre. If pure dry $(NH_4)_2SO_4$ is used, the solution contains 100 µg of NH_4-N/ml. Store the solution in a refrigerator. Immediately before use, dilute 4 ml of this stock NH_4^+ solution to 200 ml. The resulting working solution contains 2 µg of NH_4-N/ml. Accordingly, various concentrations of standard solution to be made for the standard curve.

- Phenol-nitroprusside reagent: Dissolve 7 g of phenol and 34 mg of sodium nitroprusside [disodium pentacyanonitrosylferrate, $Na_2Fe(CN)_5NO.2H_2O$] in 80 ml of NH_4^+-free water and dilute to 100 ml. Mix well, and store in a dark-colored bottle in the refrigerator.
- Buffered hypochlorite reagent: Dissolve 1.480 g of sodium hydroxide (NaOH) in 70 ml of NH_4^+-free water, add 4.98 g of sodium monohydrogen phosphate (Na_2HPO_4) and 20 ml of sodium hypochlorite (NaOCl) solution (5-5.25% NaOCl). Use less or more hypochlorite solution if the concentration is higher or lower, respectively than that is indicated above. Check the pH to ensure a value between 11.4 and 12.2. Add a small amount of additional NaOH if required to raise the pH. Dilute to a final volume of 100 ml.
- Ethylenediaminetetraacetic acid (EDTA) reagent: Dissolve 6 g of ethylenediamine tetraacetic acid disodium salt (EDTA disodium) in 80 ml of deionized water, adjust to pH 7, mix well, and dilute to a final volume of 100 ml.

Extraction

1. Place 10 g of soil in a 250 ml wide-mouth Erlenmeyer flask and add 100 ml of 2M KCl.
2. Put stopper and shake the flask on a mechanical shaker for 1 hour.
3. Allow the soil-KCl suspension to settle (about 30 min) till the supernatant is clear.
4. If the KCl extract can not be analyzed within 24 hours, then filter the soil-KCl suspension (Whatman no. 42 filter paper) and store in the refrigerator.

   Aliquots from this extract is used for the NH_4 estimation.

Estimation

1. Pipette an aliquot (not more than 5 ml) of the filtered 2M KCl extract containing between 0.5 and 12 μg of NH_4-N into a 25 ml volumetric flask. Aliquots of ≤ 3 ml normally contain sufficient NH_4-N for quantification.
2. Add 1 ml of the EDTA reagent, and mix the content of the flask.
3. Allow the content to stand for 1 minute, then add 2 ml of the phenol-nitroprusside reagent, followed by 4 ml of the buffered hypochlorite reagent, and immediately dilute the flask to volume (25 ml) with NH_4^+ - free water and mix well.
4. Place the flask in a water bath maintained at 40° C, and allow it to remain for 30 min.
5. Remove the flask from the bath, cool to room temperature, and determine the absorbance of the coloured complex at a wavelength of 636 nm against a reagent blank solution.
6. Determine the NH_4-N concentration of the sample by reference to a calibration curve plotted from the results obtained with 25 ml standard samples containing 0, 2, 4, 6, 8, 10, and 12 μg of NH_4-N/ml.
7. To prepare this curve, add an appropriate amount of 2M KCl solution (same volume as that used for aliquots of soil extract, i.e. about 5 ml))

to a series of 25-ml volumetric flasks. Add 0, 1, 2, 3, 4, 5, and 6 ml of the 2 μg NH_4-N/ml solution to the flasks, and measure the intensity of blue color developed with these standards by the procedure described above for the analysis of unknown extracts.

Calculation

NH_4-N in the sample as noted from the standard curve= A (μg/ml)

$$\mu g \text{ of } NH_4 - N \text{ in 1 g soil} = \frac{A \times 100 (\text{total vol. of extract})}{5 (\text{vol. of extract estimated})} \times \frac{1}{10 (\text{wt. of soil})} = 2A$$

where,

Weight of the soil taken for estimation = 10 g

Total volume of extract = 100 ml

Volume of extract taken for estimation = 5 ml

13. Available Phosphorus

Two methods are most commonly used for determination of available phosphorus in soils: Bray's Method No.1 for acidic soils and Olsen's Method for neutral and alkaline soils.

In these methods, specific coloured compounds are formed with the addition of appropriate reagents in the solution, the intensity of which is proportionate to the concentration of the element being estimated. The colour intensity is measured spectrophotometrically. In spectrophotometric analysis, light of definite wavelength (not exceeding say 0.1 to 1.0 nm in band width) extending to the ultraviolet region of the spectrum constitutes the light source. The photoelectric cells in spectrophotometer measure the light transmitted by the solution.

A spectrophotometer, as its name implies, is really two instruments in one cabinet – a spectrometer and a photometer. A spectrometer is a device for producing coloured light of any selected colour (or wavelength) and, when employed as part of a spectrophotometer, is usually termed as monochromator and is generally calibrated in wavelengths (nm). A photometer is a device for measuring the intensity of the light, and when incorporated in a spectrophotometer is used to measure the intensity of the monochromatic beam produced by the associated monochromator. Generally, the photometric measurement is made first with a reference liquid and then with a coloured sample contained in similar cells interposed in the light beam: the ratio of the two intensity measurements being a measure of the opacity of the sample at the wavelength of the test.

The approximate wavelength ranges of complementary colours are given in Table 19.

TABLE 19 Wavelengths and corresponding colour ranges

Wavelength (nm)	Hue	Complementary Hue
<400	Ultraviolet	
400-435	Violet	Yellow green
435-480	Blue	Yellow
480-490	Greenish blue	Orange
490-500	Bluish green	Red
500-560	Green	Purple
560-580	Yellowish green	Violet
580-595	Yellow	Blue
595-610	Orange	Greenish blue
610-750	Red	Bluish green
>760	Infra-red	

Source: Vogel, 1961

White light covers the entire visible spectrum 400-760 nm

Bray's method No. 1 (Bray and Kurtz, 1945) for acid soils

Apparatus

- Spectrophotometer
- Pipette - 2 ml, 5 ml, 10 ml and 20 ml
- Bearkers/flasks - 25 ml, 50 ml, 100 ml and 500 ml

Reagents

- Bray Extractant No 1 (0.03M NH_4F in 0.025M HCL): Dissolve 2.22 g of NH_4F in 200 ml of distilled water, filter, and add to the filtrate 1.8 litres of water containing 4 ml of concentrated HCl, make up the volume to 2 litres with distilled water.
- Molybdate reagent: Dissolve 1.50 g $(NH_4)_2MoO_4$ in 300 ml distilled water. Add the solution to 350 ml of 10M HCl solution gradually with stirring. Dilute to 1 litre with distilled water.
- Stannous chloride solution (Stock Solution): Dissolve 10 g $SnCl_2$ $2H_2O$ in 25 ml of concentrated HCl. Add a piece of pure metallic tin and store the solution in a glass stoppered bottle.
- Stannous chloride solution (Working Solution): Dilute 1 ml of the stock solution of stannous chloride to 66.0 ml with distilled water just before use. Prepare fresh dilute solution every working day.

Procedure

1. Preparation of the Standard Curve: Dissolve 0.1916 g of pure dry KH_2PO_4 in 1 litre of distilled water. This solution contains 0.10 mg P_2O_5/ml. Preserve this as a stock standard solution of phosphate. Take 10 ml of this solution and dilute it to 1 litre with distilled water. This solution contains 1 µg P_2O_5/ml (0.001 mg P_2O_5/ml). Take 1, 2, 4, 6 and 10 ml of this solution in separate 25 ml flasks. Add to each, 5 ml of the

extractant solution, 5 ml of the molybdate reagent and dilute with distilled water to about 20 ml. Add 1 ml dilute $SnCl_2$ solution, shake again and dilute to the 25 ml mark. After 10 minutes, read the blue colour of the solution on the spectrophotometer at 660 nm wavelength. Plot the absorbance reading against µg P_2O_5 and join the points.

2. Extraction: Add 50 ml of the Bray's extractant No. 1 to the 100 ml conical flask containing 5 g soil sample. Shake for 5 minutes and filter.
3. Development of colour: Take 5 ml of the filtered soil extract with a bulb pipette in a 25 ml measuring flask; deliver 5 ml of the molybdate reagent with an automatic pipette, dilute to about 20 ml with distilled water, shake and add 1 ml of the dilute $SnCl_2$ solution with a bulb pipette. Fill to the 25 ml mark and shake thoroughly. Read the blue colour after 10 minutes on the spectrophotometer at 660 nm wavelength after setting the instrument to zero with the blank prepared similarly but without the soil.

Calculation

$$P(kg/ha) = \frac{A}{1000000} \times \frac{50}{5} \times \frac{2000000}{5} = 4A$$

Where,

Weight of the soil taken	= 5 g
Volume of the extract	= 50 ml
Volume of the extract taken for estimation	= 5 ml
Volume made for estimation (dilution = 5 times)	= 25 ml

Amount of P observed in the sample on the standard curve= A (µg).
Wt. of 1 ha of soil upto a depth of 22 cm is taken as 2 million kg.

As an example, the standard curve prepared by Motsara and Roy (2008) for estimation of available P by Bray's method while establishing a soil testing laboratory in DPR Korea is given in Figure 3

FIGURE 3
Standard curve for P on spectrophotometer

$\frac{Mg}{Pb}$ = $\frac{1.25}{0.2} = 6.25$ $\frac{3.75}{0.60} = 6.25$ $\frac{7.50}{1.20} = 6.25$
$\frac{2.50}{0.4} = 6.25$ $\frac{5.00}{0.80} = 6.25$ $\frac{8.75}{1.40} = 6.25$

Olsen's method (Olsen, et al, 1954) for alkaline soils

Apparatus

Same as for Bray's Method No. 1.

Reagents

- Bicarbonate extractant: Dissolve 42 g Sodium bicarbonate in 1 litre of distilled water and adjust the pH to 8.5 by addition of dilute NaOH or HCl. Filter, if necessary.
- Activated carbon – Darco G 60.
- Molybdate reagent: Same as for the Bray's Method No. 1.
- Stannous chloride solution: Same as in Bray's Method No. 1.

Procedure

1. Preparation of the standard curve: Procedure is the same as in Bray's Method No. 1.
2. Extraction: Add 50 ml of the bicarbonate extractant to 100 ml conical flask, containing 2.5 g soil sample. Add 1 g activated carbon. Shake for 30 minutes on the mechanical shaker and filter.
3. Development of Colour: Procedure same as described under the Bray's Method No. 1.

Calculation

Same as described under the Bray's Method No. 1.

Caution:

In spite of all precautions, intensity of blue colour changes slightly with every batch of molybdate reagent. It is imperative to check standard curve every day by using 2 or 3 dilutions of the standard phosphate solution. If the standard curve does not tally, draw a new standard curve with fresh molybdate reagent.

14. Available Potassium

Flame photometeric method (Toth and Prince, 1949)

Potassium present in the soil is extracted with neutral ammonium acetate of 1 molarity. This is considered as plant available K in the soils. It is estimated with the help of flame photometer. This is a well-accepted method.

Apparatus

- Multiple Dispenser or automatic pipette – 25 ml

- Flasks and beakers - 100 ml
- Flame Photometer

Reagents

- Molar neutral ammonium acetate solution: Dissolve 77 g of ammonium acetate ($NH_4C_2H_3O_2$) in 1 litre of water. Check the pH with bromothymol blue or with a pH meter. If not neutral, add either ammonium hydroxide or acetic acid as per the need to neutralize it to pH 7.0.
- Standard potassium solution: Dissolve 1.908 g pure KCl in 1 litre of distilled water. This solution contains 1 mg K/ml . Take 100 ml of this solution and dilute to 1 litre with ammonium acetate solution. This gives 0.1 mg K/ml as stock solution.
- Working potassium standard solutions: Take 0, 5, 10, 15 and 20 ml of the stock solution separately and dilute each to 100 ml with the M ammonium acetate solution. These solutions contain 0, 5, 10, 15 and 20 μg K/ml, respectively.

Procedure

1. Preparation of the Standard Curve: Set up the flame photometer by atomizing 0 and 20 μg K/ml solutions alternatively to 0 and 100 reading. Atomize intermediate working standard solutions and record the readings. Plot these readings against the respective potassium contents and connect the points with a straight line to obtain a standard curve.
2. Extraction: Add 25 ml of the ammonium acetate extractant to conical flask fixed in a wooden rack containing 5 g soil sample. Shake for 5 minutes and filter.
3. Determine potash in the filtrate with the flame photometer.

Calculation

$$K(kg/ha) = A \times \frac{25}{5} \times \frac{2000000}{1000000} = 10A$$

Where,

A = content of K (μg) in the sample, as read from the standard curve:

Weight of 1 ha of soil upto a plough depth of 22 cm is approx. 2 million kg.

Example

While setting up the soil testing laboratory at DPR Korea, following standard curve (Figure 4) was prepared by Motsara and Roy (2008) for estimation of K on flame photometer following the above method.

15. Available Sulphur

Available sulphur in mineral soils occurs mainly as adsorbed SO_4 ions. Phosphate ions (as monacalcium 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 those of Na or K as Ca prevents deflocculation in heavy textured soils and leads to easy filtration. SO_4 in the extract can be estimated turbidimetrically using a spectrophotometer. A major problem arises when the amount of extracted sulphur is too low to be measured. To overcome this problem, seed solution of known S concentration is added to the extract to raise the concentration to easily detectable level.

Barium sulphate precipitation method is described here.

Apparatus

- Spectrophotometer
- Mechanical shaker
- Volumetric flask

Reagents

- Mono-calcium phosphate extracting solution (500 mg P/litre): Dissolve 2.035 g of $Ca(H_2PO_4)_2.H_2O$ in 1 litre of water.

- Gum acacia-acetic acid solution: Dissolve 5g of chemically pure gum acacia powder in 500 ml of hot water and filter in hot condition through Whatman No.42 filter paper. Cool and dilute to one litre with dilute acetic acid.
- Barium chloride: Pass AR grade $BaCl_2$ salt through 1 mm sieve and store for use.
- Standard stock solution (2000 mg S/litre): Dissolve 10.89g of oven-dried AR grade potassium sulphate in 1 litre water.
- Standard working solution (10 mg S/litre): Measure exactly 2.5 ml of the stock solution and dilute to 500 ml.
- Barium sulphate seed suspension: Dissolve 18 g of AR grade $BaCl_2$ in 44 ml of hot water and add 0.5 ml of the standard stock solution. Heat the content to boiling and then cool quickly. Add 4 ml of gum acacia-acetic acid solution to it. Prepare a fresh seed suspension for estimation everyday.
- Dilute nitric acid (approx 25%): Dilute 250 ml of AR grade conc. HNO_3 to one litre.
- Acetic-phosphoric acid: Mix 900 ml of AR grade glacial acetic acid with 300 ml of H_3PO_4 (AR grade).

Procedure

1. Weigh 20 g of soil sample in a 250 ml conical flask. Add 100 ml of the monocalcium phosphate extracting solution (500 mg P/litre) and shake for one hour. Filter through Whatman No.42 filter paper.
2. Take 10 ml of the clear filtrate into a 25 ml volumetric flask.
3. 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, if required.
4. 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.
5. After 10 minutes, invert 10 times. Again after 5 minutes invert for 5 times.
6. Allow to stand for 15 minutes and then add 1 ml of gum acacia-acetic acid solution.
7. Make up the volume to 25 ml, invert 3 times and keep aside for 90 minutes.
8. Invert 10 times and measure the turbidity intensity at 440 nm (blue filter).
9. Run a blank side by side.
10. Preparation of standard curve:
    - Put 2.5, 5.0, 7.5, 10.0, 12.5, 15.0 ml of the working standard solution (10 mg S/litre) into a series of 25 ml volumetric flasks to obtain 25, 50, 75, 100, 125 and 150 µg S.
    - Proceed to develop turbidity as described above for sample aliquots.
    - Read the turbidity intensity and prepare the curve by plotting readings against sulphur concentrations (in µg in the final volume of 25 ml).

Calculation

$$\text{Available Sulphur } (SO_4^- - S) \text{ in soil (mg/kg)} = \frac{W \times 100}{10 \times 20} = \frac{W}{2}$$

Where,
W stands for the quantity of sulphur in mg as obtained on X-axis against an absorbance reading (Y-axis) on standard curve
20 is the weight of the soil sample in g
100 is the volume of the extractant in ml
10 is the volume of extractant in ml in which turbidity is developed.

16. Determination of exchangeable Calcium and Magnesium

Exchangeable cations are usually determined in a neutral normal ammonium acetate extract of soil. Extraction is carried out by shaking the soil : extractant mixture followed by filtration or centifugation. Calcium and magnesium are determined either by EDTA titration method or by atomic absorption spectrophotometer after the removal of ammonium acetate and organic matter.

It may be noted that in soils appreciable amount of soluble calcium and magnesium may be present. Hence, these water soluble cations are estimated in the 1:2 soil water extract and deducted from ammonium acetate extractable calcium and magnesium (since ammonium acetate also extracts water soluble cations) to obtain exchangeable calcium and magnesium. To obtain soil water extract, generally 25g soil and 50ml of water suspension is shaken for 30 minutes on a mechanical shaker and filtered. The method of estimation in the water extract (water soluble cations) and ammonium acetate extract (exchangeable cation) is same.

The EDTA titration method developed by Chang and Bray (1951) is preferred on account of its accuracy, simplicity and speed.

The method is based on the principle that calcium, magnesium and a number of other cations form stable complexes with versenate (ethylendiaminetetraacetic acid disodium salt) at different pH. The interference of Cu, Zn, Fe, Mn is prevented by the use of 2% NaCN solution or carbamate. Usually in irrigation waters and water extracts of soil, the quantities of interfering ions are negligible and can be neglected.

A known volume of standard calcium solution is titrated with standard versenate 0.01N solution using muroxide (ammonium purpurate) indicator in the presence of NaOH solution. The end point is a change of colour from orange red to purple at pH 12 when the whole of calcium forms a complex with EDTA.

16.a. Calcium by Versenate (EDTA) method

Apparatus

- Shaker
- Porcelain dish
- Beakers
- Volumetric/conical flask.

Reagents

- Ammonium chloride – ammonium hydroxide buffer solution: Dissolve 67.5 g ammonium chloride in 570 ml of conc. ammonium hydroxide and make to 1 litre.
- Standard 0.01N calcium solution: Take accurately 0.5 g of pure calcium carbonate and dissolve it in 10 ml of 3N HCl. Boil to expel CO_2 and then make the volume to 1 litre with distilled water.
- EDTA solution (0.01N): Take 2.0 g of versenate, dissolve in distilled water and make the volume to 1 litre. Titrate it with 0.01N calcium solution and make necessary dilution so that its normality is exactly equal to 0.01N.
- Muroxide indicator powder: Take 0.2 g of muroxide (also known as ammonium purpurate) and mix it with 40 g of powdered potassium sulphate. This indicator should not be stored in the form of solution, otherwise it gets oxidized.
- Sodium diethyl dithiocarbamate crystals: It is used to remove the interference of other metal ions.
- Sodium hydroxide 4N: Prepare 16% soda solution by dissolving 160 g of pure sodium hydroxide in water and make the volume to 1 litre. This will give pH 12.

Procedure

1. Take 5 g air dried soil sample in 150 ml conical flask and add 25 ml of neutral normal ammonium acetate. Shake on mechanical shaker for 5 minutes and filter through Whatman filter paper No.1.
2. Take a suitable aliquot (5 or 10 ml) and add 2-3 crystals of carbamate and 5 ml of 16% NaOH solution.
3. Add 40-50 mg of the indicator powder. Titrate it with 0.01N EDTA solution till the colour gradually changes from orange red to reddish violet (purple). It is advised to add a drop of EDTA solution at an interval of 5 to 10 seconds, as the change of colour is not instantaneous.
4. The end point must be compared with a blank reading. If the solution is over titrated, it should be back titrated with standard calcium solution and exact volume used is thus found.
5. Note the volume of EDTA used for titration.

Calculation

If N_1 is normality of Ca^{++} and V_1 is volume of aliquot taken and N_2V_2 are the normality and volume of EDTA used, respectively, then,

$N_1V_1 = N_2V_2$

$$\text{Or } N_1 = \frac{N_2V_2}{V_1} = \frac{\text{Normality of EDTA x Vol.of EDTA}}{\text{ml of aliquot taken}}$$

Here N_1 (Normality) = equivalent of Ca^{2+} present in one litre of aliquot.

$$\text{Hence, } Ca^{2+} \text{ me/litre} = \frac{\text{Noramlity of EDTA x Vol.of EDTA x 1000}}{\text{ml of aliquot taken}}$$

When expressed on soil weight basis,

$$Ca^{2+} \text{ me/100 g soil} = \frac{100}{\text{wt.of soil}} \text{ x } \frac{\text{extract volume}}{1000} \text{ x Ca as me/litre}$$

16.b. Calcium plus Magnesium by Versenate (EDTA) method

Magnesium in solution can be titrated with 0.01N EDTA using Eriochrome black T dye as indicator at pH 10 in the presence of ammonium chloride and ammonium hydroxide buffer. At the end point, colour changes from wine red to blue or green. When calcium is also present in the solution this titration will estimate both calcium and magnesium. Beyond pH 10 magnesium is not bound strongly to Erichrome black T indicator to give a distinct end point.

Apparatus

- Shaker
- Porcelain dish
- Beaker
- Volumetric/conical flask

Reagents

- EDTA or Versenate solution (0.01N): Same as in calcium determination.
- Ammonium chloride-ammonium hydroxide buffer solution: Same as in calcium determination.
- Eriochrome black T indicator: Take 100 ml of ethanol and dissolve 4.5 g of hydroxyl amine hydrochloride in it. Add 0.5 g of the indicator and prepare solution. Hydroxylamine hydrochloride removes the interference of manganese by keeping it in lower valency state (Mn^{2+}). Or mix thoroughly 0.5 g of the indicator with 50 g of ammonium chloride.

- Sodium cyanide solution (2%) or sodium diethyl dithiocarbamate crystals. This is used to remove the interference of copper, cobalt and nickel.

Procedure

1. Take 5 g air dried soil in 150 ml flask, add 25 ml of neutral normal ammonium acetate solution and shake on a mechanical shaker for 5 minutes and filter through Whatman No.1 filter paper.
2. Pipette out 5 ml of aliquot containing not more than 0.1 meq of Ca plus Mg. If the solution has a higher concentration, it should be diluted.
3. Add 2 to 5 crystals of carbamate and 5 ml of ammonium chloride-ammonium hydroxide buffer solution. Add 3-4 drops of Eriochrome black T indicator.
4. Titrate this solution with 0.01N versenate till the colour changes to bright blue or green and no tinge of wine red colour remains.

Calculation

If N_1 and V_1 are normality (concentration of Ca^{2+}+Mg^{2+}) and volume of aliquot taken and N_2V_2 are the normality and volume of EDTA used respectively, then,

$N_1V_1 = N_2V_2$
Or N_1 =

$$\frac{N_2V_2}{V_1} = \frac{\text{Normality of EDTA x Vol. of EDTA}}{\text{ml of aliquot taken}}$$

Here N_1 (Normality) = equivalents of Ca^{2+} plus Mg^{2+} present in one litre of aliquot.

$$\text{Hence, } Ca^{2+} \text{ plus } Mg^{2+} \text{ me/litre} = \frac{\text{Normality of EDTA x Vol. of EDTA x 1000}}{\text{ml of aliquot taken}}$$

Meliequivalent (me) of Mg^{++} = me (Ca^{++} + Mg^{++}) – me of Ca^{++}

When expressed on soil weight basis.

$$Ca^{++} + Mg^{++} \text{ me/100 g soil} = \frac{100}{\text{wt. of soil}} \times \frac{\text{extract volume}}{1000} \times Ca^{++} + Mg \text{ me/litre}$$

17. Micronutrients

For estimation of micronutrients also, it is the plant available form which is critical and not the total content. The major objective of soil test for micronutrients is to determine whether a soil can supply adequate micronutrients for optimum crop production or whether nutrient deficiencies are expected in crops grown on such soils. Most commonly

studied micronutrients are Zn, Cu, Fe, Mn, B and Mo and the same have been dealt with here.

Micronutrients are present in different forms in the soil. Among the most deficient ones is Zn, which is present as divalent cation Zn^{2+}. Maize, citrus, legumes, cotton and rice are especially sensitive to zinc deficiency. Iron is present mostly in sparingly soluble ferric oxide form, which occur as coatings of aggregate or as separate constituent of the clay fraction. Soil redox potential and pH affect the availability of iron. The form of iron that is predominantly taken up by plants is the Fe^{2+}. Uptake of Fe is inhibited by phosphate levels due to the formation of insoluble iron phosphate. Manganese, chemically behaves in the soils the same way as Fe. Soil Mn originates primarily from the decomposition of ferromagnesian rocks. It is taken up by the plants as Mn^{2+} ions, although it exists in many oxidation states. Manganese and phosphate are mutually antagonistic. Copper like zinc exists in soils mainly as divalent ions Cu^{2+}. It is usually adsorbed by the clay minerals or associated with organic matter, although they have little or no effect on its availability to crops. High phosphate fertilization can induce Cu deficiency. Molybdenum mostly occurs as MoO_3, MoO_5 and MoO_2. These oxides are slowly transformed to soluble molybdates (MoO_4) which is the form taken up by plants. Boron deficiency occurs mostly in the light textured acid soils when they are leached heavily through irrigation or heavy rainfall.

Different extractants have been developed for assessing plant available nutrient (element) content in the soils. The elements so extracted can be estimated quantitatively through chemical methods or instrumental techniques. Commonly used extractant for different elements are given in **Table 20.**

TABLE 20 Commonly used extractants for micronutrients

Element	Extractants
Zinc	EDTA + Ammonium Acetate, EDTA + Ammonium Carbonate, DTPA + $CaCl_2$, HCl, HNO_3 and Dithiozone + Ammonium Acetate
Copper	EDTA, EDTA + Ammonium Acetate, Ammonium Bicarobnate + DTPA,HCl and HNO_3
Iron	EDTA, DTPA, EDTA + Ammonium Acetate, HCl and HNO_3
Manganese	Hydroquinone, Ammonium Phosphate, DTPA and EDTA + Ammonium Acetate
Boron	Hot water and Manitol + $CaCl_2$
Molybdenum	Ammonium Oxalate, Ammonium Acetate, Ammonium Fluoride and Water

TABLE 22 Specifications of hollow cathode lamps for micronutrient estimation on AAS

Specifications	Zn	Cu	Fe	Mn
Lamp current (m A°)	5	3	7	5
Wave length (nm)	213.9	324.8	248.3	279.5
Linear range (mg/l)	0.4-1.5	1.0-5.0	2.0-9.0	1.0-3.6
Slit width (nm)	0-2	0-2	0-2	0-2
Integration time (sec)	2.0	2.0	2.0	2.0
Flame	Air Acetylene			

Running parameters which are specific to a particular model are given in the software provided with the equipment manual. Accordingly, the current supply, wave length of hollow cathode lamp, integration time and anticipated estimation ranges are fixed. Hollow cathode and Deutorium lamps are required to be properly aligned before starting the equipment. After proper alignment and adjustment, standard curves are prepared to ensure that the concentration of the elements in solutions perfectly relates to the absorbance.

Preparation of standard solutions

Readymade standard solutions 1 000 µg/ml or 1 mg/ml (1 000 ppm) of dependable accuracy are supplied with the AAS and are also available with the suppliers of chemical reagents. If the standard solutions are to be prepared in the laboratory, either metal element foils of 100% purity or the standard chemical salts can be used. The quantities of chemical required to make 1 litre standard solution of 100 µg/ml for different elements are given in **Table 23**

TABLE 23 Specifications for preparing micronutrient standard solutions

Element	Conc. of stock solution (µg/ml)	Salt to be used	Quantity of salt required/litre
Zn	100	Zinc Sulphate ($ZnSO_4.7H_2O$)	0.4398
Cu	100	Copper Sulphate ($CuSO_4.5H_2O$)	0.3928
Fe	100	Ferrous Sulphate ($FeSO_4.7H_2O$) or Ferrous Ammonium Sulphate	0.4964 0.7028
Mn	100	Manganese Sulphate ($MnSO_4.H_2O$)	0.3075

In case of Zn, Cu and Fe, 1 000 μg/ml (1 000 ppm) standard solution are preferably prepared by dissolving 1.0 g pure metal wire and volume made to 1 litre as per the method described under each element. It is diluted to obtain the required concentration. In case of Mn, Mn $SO_4.H_2O$ is preferred.

Preparation of standard curves

i. Zinc

Reagents

- Standard Zinc Solution: Weigh 1.0 g of pure zinc metal in a beaker. Add 20 ml HCl (1:1). Keep for few hours allowing the metal to dissolve completely. Transfer the solution to 1 litre volumetric flask. Make up the volume with glass-distilled water. This is 1 000 μg/ml zinc solution. For preparation of standard curve, refer 1 000 μg/ml solution as solution **A**. Dilute 1 ml of standard A to 100 ml to get 10 μg/ml solution to be designated as standard **B.**

- Glass-distilled or demineralized acidified water of pH 2.5 $\pm$ 0.5: Dilute 1 ml of 10% sulphuric acid to one litre with glass-distilled or mineralized water and adjust the pH to 2.5 with a pH meter using 10% H_2SO_4 or NaOH. This solution is called acidified water.

- Working Zn standard solutions: Pipette 1, 2, 4, 6, 8 and 10 ml of standard **B** solution in 50 ml numbered volumetric flask and make the volume with DTPA solution to obtain 0.2, 0.4, 0.8, 1.2, 1.6 and 2.0 μg/ml zinc. Stopper the flasks and shake them well. Fresh standards should be prepared every time when a fresh lot of acidified water is prepared.

Procedure

1. Flaming the solutions: Atomise the standards on atomic absorption spectrophotometer at a wave-length of 213.8 nm (Zn line of the instrument).

2. Prepare a standard curve of known concentrations of zinc solution by plotting the absorbance values on Y–axis against their respective zinc concentration on X–axis.

Precautions

- Weighing must be done on an electronic balance.
- All the glass apparatus to be used should be washed first with dilute hydrochloric acid (1:4) and then with distilled water.
- The pipette should be rinsed with the same solution to be measured.
- The outer surface of the pipette should be wiped with filter paper after use.

- Glass-distilled or demineralized acidified water of pH 2.5 ± 0.5: Same as that done for Zn.
- Working Fe standard solutions: Pipette 10 ml of iron stock solution in 100 ml volumetric flask and dilute to volume with DTPA solution. This is 100 µg/ml iron solution. Take 2, 4, 8, 12 and 16 ml of 100 µg/ml solution and dilute each to 100 ml to obtain 2, 3, 8, 12 and 16 µg/ml of Fe solution.

Procedure

1. Flame the standards on an atomic absorption spectrophotometer at a wavelength of 248.3_nm (Fe line of the instrument).
2. Prepare the standard curve with the known concentration of copper on X-axis by plotting against absorbance value on Y-axis.

Example

A graph prepared on standardization of iron estimation by AAS while setting up of a soil and fertilizer testing laboratory at Vientiane, Laos by Motsara and Roy(2008)-FAO Buletin No.19 is reproduced below (Figure 7). It shows a r^2 value of 0.9814. From this graph, conc. of Fe in the soil sample was worked out.

iv Manganese

Reagents

- Standard Mn solution: Weigh 3.0751 g of AR grade manganese sulphate ($MnSO_4$ H_2O) on a clean watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and

funnel with acidified water and make the volume up to the mark. This solution will be 1 000 µg/ml Mn. A secondary dilution of 5 ml to 100 ml with acidified water gives a 50 µg/ml solution.

- Glass-distilled or de-mineralized acidified water of pH 2.5 ± 0.2: Same as that for Zn.
- Working Mn standard solutions: Standard curve is prepared by taking lower concentrations of Mn in the range of 0-10 µg/ml Take 1, 2, 4, 6 and 8 ml of 50 µg/ml solution and make up the volume with DTPA solution to 50 ml to obtain 1, 2, 4, 6 and 8 µg/ml working standards.

Procedure

1. Flame the standards on an atomic absorption spectrophotometer at a wavelength of 279.5 nm (Mn line of the instrument).
2. Prepare the standard curve with the known concentration of Mn on X-axis by plotting against absorbance value on Y-axis.

Example

A graph prepared on standardization of iron estimation by AAS while setting up of a soil and fertilizer testing laboratory at Vientiane, Laos by Motsara and Roy(2008) – FAO Bulletin No.19 is reproduced below (Figure 8). It shows an r^2 value of 0.9982. From this graph, conc. of Fe in the soil sample was worked out.

Procedure for extraction by DTPA

Once standard curves have been prepared, proceed for extraction by DTPA.

1. Take 10 g of soil sample in 100 ml narrow-mouth polypropylene bottle.

17a. Available Zinc, Copper, Iron and Manganese

Ethylene damine-teraacitic acid (EDTA) + Ammonium Acetate is commonly used for extraction of many elements. DTPA (Diethylenetriaminepentaacetic acid) is yet another common (universal) extractant and is widely used for simultaneous extraction of elements, like Zn, Cu, Fe and Mn. This extractant was advanced by Lindsay and Norvell (1978). Although, a specific extractant for an element which has higher correlation with plant availability may be preferred, the universal or the common extractant saves on the cost of chemicals and the time involved in estimation, especially in a service laboratory where a large number of samples need to be analysed with in a short period.. Therefore, DTPA being one such extractant has been described in this publication.

The estimation of elements in the extract is done with the help of Atomic Absorption Spectrophotometer (AAS). Critical limits for DTPA extractable micronutrient elements as proposed by Lindsay and Norvell (1978) are given in **Table 21.**

TABLE 21 Critical limits for DTPA extractable micronutrients

Availability	Micronutrients (µg/g soil)			
	Zn	Cu	Fe	Mn
Very low	0-0.5	0-0.1	0-2	0-0.5
Low	0.5-1	0.1-0.3	2-4	0.5-1.2
Medium	1-3	0.3-0.8	4-6	1.2-3.5
High	3-5	0.8-3	6-10	3.5-6
Very High	>5	>3	>10	>6

Principle of extraction

DTPA is an important and widely used chelating agent, which combines with free metal ions in the solution to form soluble complexes of elements. To avoid excessive dissolution of $CaCO_3$, which may release occluded micronutrients that are not available to crops in calcarious soils and may give erroneous results, the extractant is buffered in slightly alkaline pH. Triethanolamine (TEA) is used as buffer because it burns cleanly during atomization of extractant solution while estimating on AAS. The DTPA has a capacity to complex each of the micronutrient cations as 10 times of its atomic weight. The capacity ranges from 550 to 650 mg/kg depending upon the micronutrient cations.

Extracting solution (DTPA)

DTPA 0.005M, 0.01M $CaCl_2.2H_2O$ and 0.1M TEA extractant: Add 1.967 g DTPA and 13.3 ml TEA in 400 ml distilled water in a 500 ml flask. Take 1.47 g $CaCl_2.2H_2O$ in a separate 1 000 ml flask. Add 500 ml distilled water and shake to dissolve. Add DTPA+TEA mixture in $CaCl_2$ solution and make

the volume to 1 litre. pH should be adjusted to 7.3 by using 1M HCl before making the volume.

Principle of Estimation

The extracted elements can be estimated by various methods, which include volumetric analysis, spectrometry and atomic absorption spectroscopy. Volumetric methods such as EDTA and $KMnO_4$ titrations are used for estimation of zinc and Mn, and iron, respectively. Copper can be estimated by titration with $Na_2S_2O_3$. Spectrometric methods are deployed in estimation of specific colour developed due to the presence of an element, which forms coloured compound in the presence of specific chemicals under definite set of conditions. The colour intensity has to be linear with the concentration of the element in question. The interference due to any other element has to be eliminated. Such methods are dithiozone method for estimation of zinc, orthophenonthroline method for iron, potassium periodate method for manganese, carbamate method for copper. The chemical methods are generally cumbersome and time taking. Hence the most commonly employed method is atomic absorption spectrometry. Here, the interference by other elements is almost nil or negligible because the estimation is carried out for an element at a specific emission spectraline. In fact in AAS, traces of one element can be accurately determined in the presence of a high concentration of other elements.

Principle of Atomic Absorption Spectrophotometry

The procedure is based on flame absorption rather than flame emission and upon the fact that metal atoms absorb strongly at discrete characteristic wavelengths which coincides with the emission spectralines of a particular element. The liquid sample is atomized. The hollow cathode lamp which precedes the atomiser, emits the spectrum of the metal used to make the cathode. This beam traverse the flame and is focused on the entrance slit of a monochromator, which is set to read the intensity of the chosen spectral line. Light with this wavelength is absorbed by the metal in the flame and the degree of absorption being the function of the concentration of the metal in the flame, the concentration of the atoms in the dissolved material is determined. For elemental analysis, a working curve or a standard curve is prepared by measuring the signal or absorbance of a series of standards of known concentration of the element under estimation. From such curve, concentration of the element in unknown sample is estimated.

Atomic Absorption Spectroscopy can be successfully applied for estimation of Zn, Cu, Fe and Mn. For specific estimation on AAS, hollow cathode lamps, specific to specific elements are used. The specifications of relevant hollow cathode lamps are given in **Table 22.**

- After using the pipette, place them on a clean dry filter paper in order to prevent contamination.

Example

A graph prepared on standardization of zinc estimation by AAS while setting up of a soil and fertilizer testing laboratory at Vientiane, Laos by Motsara and Roy(2008) is reproduced below (Figure 5). It shows a r^2 value of 0.997. From this graph, conc. of zinc in the soil sample was worked out.

ii. Copper

Reagents

- Standard copper solution: Weigh 1 g of pure copper wire on a clean watch glass and transfer it to one litre flask. Add 30 ml of HNO_3 (1:1) and make up the mark. Stopper the flask and shake the solution well. This is 1 000 µg/ml Cu solution and should be stored in a clean bottle for further use. Dilute 1 ml of 1 000 µg/ml solution of copper to 100 ml to get 10 µg/ml standard copper solution.
- Glass-distilled or demineralized acidified water of pH 2.5 $\pm$ 0.5: Same as that done for Zn.
- Working Cu standard solutions: Pipette 2, 3, 4, 5, 6 and 7 ml of 10 µg/ml standard Cu solution in 50 ml numbered volumetric flasks and make the volume with DTPA solution to get 0.4, 0.6, 0.8, 1.0, 1.2 and

1.4 μg/ml copper. Stopper the flasks and shake them well. Prepare fresh standards every fortnight.

Procedure

1. Flame the standards on an atomic absorption spectrophotometer at a wavelength of 324.8_nm (Cu line of the instrument).
2. Prepare the standard curve with the known concentration of copper on X-axis by plotting against absorbance value on Y-axis.

Example

A graph prepared on standardization of copper estimation by AAS while setting up of a soil and fertilizer testing laboratory at Vientiane, Laos by Motsara and Roy(2008) is reproduced below (Figure 6). It shows a r^2 value of 0.9997. From this graph, conc. of Cu in the soil sample was worked out.

iii. Iron

Reagents

- Standard iron solution: Weigh accurately 1 g pure iron wire and put it in a beaker and add approximately 30 ml of 6M HCl and boil. Transfer it to one litre volumetric flask through the funnel giving several washings to the beaker and funnel with glass-distilled water. Make the volume up to the mark. Stopper the flask and shake the solution well. This is 1 000 μg/ml iron solution.

2. Add 20 ml of DTPA extracting solution.
3. Stopper the bottle and shake for 2 hours at room temperature (25^0 C).
4. Filter the content using filter paper No.1 or 42 and collect the filtrate in polypropylene bottles
5. Prepare a blank following all steps except taking a soil sample.

Note:

The extract so obtained is used for estimation of different micronutrients. For extraction of more accurate quantity of an element which has a higher degree of correlation with plant availability, there are element specific extractants. An extractant standardized/recommended for a given situation in a country may be used. The estimation procedure on AAS, however, remains unchanged.

Estimation on AAS

1. Select an element specific hollow cathode lamp and mount it on AAS.
2. Start the flame.
3. Set the instrument at zero by using blank solution.
4. Aspirate the standard solutions of different concentrations one by one and record the readings.
5. Prepare standard curve plotting the concentration of the element concerned and the corresponding absorbance in different standard samples (as described before).
6. When the operation is performed accurately, a straight line relationship is obtained between the concentration of the element and the absorbance on AAS with a correlation coefficient which may be nearly as high as 1.0.
7. Aspirate the soil extractant obtained for estimation of nutrient element in the given soil sample and observe the readings.
8. Find out the content of the element in the soil extract by observing its concentration on the standard curve against its absorbance

Calculation

Content of micronutrient in the sample (mg/kg) = C μg/ml x 2 (dilution factor).

Where,
Dilution factor = 2.0 (Soil sample taken = 10.0 g and DTPA used = 20 ml)
Absorbance reading on AAS of the soil extract being estimated for a particular element = X
Concentration of micronutrient as read from the standard curve for the given absorbance (X) = C μg/ml.

17b. Available boron

The most commonly used method for available B is hot water extraction of soil as developed by Berger and Truog (1939). A number of modified versions of this method have been proposed but the basic procedure remains the same.

Water soluble boron is the available form of boron. It is extracted from the soil by water suspension. In the extract, boron can be analysed by colorimetric methods using reagents such as Carmine, azomethine – H and most recently by inductively coupled plasma (ICP) and atomic emission spectrometry (Haubold *et al.*, 1988; Jeffrey and McCallum, 1988). Colourimetric method is however, preferable due to the fact that boron being a non-metal, use of AAS for its estimation pose some limitations.

The extraction method described here is the simple modification (Gupta, 1967) of the one developed by Berger and Truoug (1939) in which boiling soil with water is employed.

Extraction Procedure

1. Weight 25 g of soil in a quartz flask or beaker.
2. Add about 50 ml of double distilled water and about 0.5 g of activated charcoal.
3. The mixture is boiled for about 5 minutes and filtered through Whatman Filter Paper No.42.

i. Estimation by AAS

The specifications of relevant hollow cathode lamp is given below:

Lamp current (m A°)	5
Wave length (nm)	249.7
Linear range (µg/ml)	1-4
Slit width (nm)	0.02
Integration time (sec)	2.0
Flame	Acetylene Nitrous Oxide

Running parameters which are specific to a particular model are given in the software provided with the equipment manual. Accordingly, the current supply, wave length of hollow cathode lamp, integration time and anticipated estimation ranges are fixed. Hollow cathode and Deutorium lamps are required to be properly aligned before starting the equipment. After proper alignment and adjustment, standard curves are prepared to ensure that the concentration of the element in solutions perfectly relates to the absorbance.

Reagents

- Standard Boron Solution: Dissolve 8.819 g $Na_2B_4O_7 10H_2O$ in warm water. Dilute to 1 litre to get 1 000 µg/ml boron stock solution. Dilute 1 ml of standard to 100 ml to get 10 µg/ml boron.
- Working standards: Take 1, 2, 3, 4, 5, 6, 7 and 10 ml of 10,000 µg/ml solution and dilute each to 50 ml to get 200, 400, 600, 800, 1000, 1200, 1400 and 2000 µg/ml B.

Procedure

1. Atomise the working standards on atomic absorption spectrophotometer using acetylene-nitrous oxide as fuel instead of air acetylene fuel (as used for other micronutrients) at a wavelength of 249.7 nm.
2. Prepare a standard curve of known concentration of boron by plotting the absorbance values on Y-axis against their respective boron concentration on X-axis. Measure the absorbance of the soil sample extract and find out the boron content in the soil from the standard curve.

Calculation

Content of B in the soil (µg/g or mg/kg) = C x dilution factor (10)

Where :

- *C* (µg/ml) = concentration of B as read from the standard curve against the absorbance reading of the soil solution on the spectrophotometer;
- Dilution factor = 10, which is calculated as follows:

  - weight of the soil taken = 25 g;
  - volume of extractant (water) added = 50 ml;
  - first dilution = 2 times;
  - volume of the filtrate taken = 5 ml;
  - final volume of filtrate after colour development = 25 ml;
  - second dilution = 5 times;
  - total dilution = 2 x 5 = 10 times

ii. Estimation by colorimetric method

The extracted B in the filtered extract is determined by the azomethine-H colorimetric method.

Apparatus

- Analytical balance
- Flask or beaker
- Volumetric flask
- Funnels
- Whatman No.42 filter paper
- Spectrophotometer

Reagents

- Azomethine-H: Dissolve 0.45 g azomethine-H and 1.0 g L-ascorbic acid in about 100 ml deionized or double-distilled water. If solution is not clear, it should be heated gently in a water bath or under a hot water tap at about 30^0 C till it dissolves. Every week a fresh solution should be prepared and kept in a refrigerator.
- Buffer solution: Dissolve 250 g ammonium acetate in 500 ml deionized or double-distilled water and adjust the pH to about 5.5 by slowly adding approximately 100 ml glacial acetic acid, with constant stirring.
- EDTA solution (0.025 M): Dissolve 9.3 g EDTA in deionized or double-distilled water and make the volume upto 1 litre.
- Standard stock solution: Dissolve 0.8819g $Na_2 B_4O_7 10H_2O$ AR grade in a small volume of deionized water and make volume to 1 000 ml to obtain a stock solution of 100 µg B/ml.
- Working standard solution: Take 5 ml of stock solution in a 100 ml volumetric flask and dilute it to the mark. This solution contains 5 µg B/ml.

Procedure

1. Take 5 ml of the clear filtered extract in a 25 ml volumetric flask and add 2 ml buffer solution, 2 ml EDTA solution and 2 ml azomethine-H solution.
2. Mix the contents thoroughly after the addition of each reagent.
3. Let the solution stand for 1 hour to allow colour development. Then, the volume is made to the mark.
4. Intensity of colour is measured at 420 nm.
5. The colour thus developed has been found to be stable upto 3-4 hours.
6. Preparation of standard curve: Take 0, 0.25, 0.50, 1.0, 2.0 and 4.0 ml of 5 µg B/ml solution (working standard) to a series of 25 ml volumetric flasks. Add 2 ml each of buffer reagent, EDTA solution and azomethine-H solution. Mix the contents after each addition and allow to stand at room temperature for 30 minutes. Make the volume to 25 ml with deionized or double-distilled water and measure absorbance at 420 nm. This will give reading for standard solution having B concentration 0, 0.05, 0.10, 0.20, 0.40 and 0.80 µg B/ml.

Calculation

Content of B in the soil (μg/g or mg/kg) = C x Dilution factor (10)

Where,
C (μg/ml) = Concentration of B as read from the standard curve against the absorbance reading of the soil solution on the spectrophotometer.

Dilution factor = 10 which is calculated as follows:

- Weight of the soil taken = 25 g
- Volume of extractant (water) added = 50 ml
- First dilution = 2 times
- Volume of the filtrate taken = 5 ml
- Final volume of filtrate after colour development = 25 ml
- Second dilution = 5 times
- Total dilution = 2 x 5 = 10 times

Note:

1. The use of azomethine-H is an improvement over that of carmine, quinalizarin and curcumin, since the procedure involving this chemical does not require the use of concentrated acid.
2. The amount of charcoal added may vary with the organic matter content of the soil and should be just sufficient to produce a colourless extract after 5 min. of boiling on a hot plate. Excess amounts of charcoal can result in loss of extractable B from soils.

17c. Available molybdenum

Molybdenum (Mo) is a rare element in soils, and is present only in very small amounts in igneous and sedimentary rocks. The major inorganic source of Mo is molybdenite (MoS_2). The total Mo content in soils is perhaps the lowest of all the micronutrient elements, and is reported to range between 0.2 μg/g and 10 μg/g.

In the soil solution Mo exists mainly as $HMoO_4$ ion under acidic condition, and as MoO_4^{2-} ion under neutral to alkaline conditions. Because of the anionic nature of Mo, its anions will not be attracted much by the negatively charged colloids, and therefore, tend to be leached from the soils in humid region.

Molybdenum can be toxic due to greater solubility in alkaline soils of the arid and semi-arid regions, and deficient in acid soils of the humid regions.

In plants a deficiency of Mo is common at levels of 0.1 μg/g soil or less. Molybdenum toxicity (molybdenosis) is common when cattle graze forage plants with 10-20 μg Mo/g.

In case of Molybdenum, ammonium acetate and/or ammonium oxalate extraction is usually carried out. Estimations can be done both by AAS and colourimetric methods with preference for the latter due to the formation of oxide in the flame in case of estimation by AAS. Therefore, chemical method has also been described. Ammonium oxalate is considered as a better extractant. However, for estimation on AAS ammonium acetate is preferred as the oxalates pose a limitation on AAS unless removed by digesting with di-acid as is described in case of colorimetric estimation.

i. Estimation by AAS

The specifications of relevant hollow cathode lamp is given below:

Lamp current (m A°)	5
Wave length (nm)	313.3
Linear range (µg/ml)	1-4
Slit width (nm)	0.02
Integration time (sec)	2.0
Flame	Acetylene Nitrous Oxide

Running parameters which are specific to a particular model are given in the software provided with the equipment manual. Accordingly, the current supply, wave length of hollow cathode lamp, integration time and anticipated estimation ranges are fixed. Hollow cathode and Deutorium lamps are required to be properly aligned before starting the equipment. After proper alignment and adjustment, standard curves are prepared to ensure that the concentration of the element in solutions perfectly relates to the absorbance.

Apparatus

- Centrifuge and 50 ml centrifuge tubes
- Automatic shaker
- Atomic Absorption Spectrophotometer

Reagents

- Ammonium acetate solution (NH_4OAc) 1.0 M: Dissolve 77.09 g of ammonium acetate in 1 litre of distilled water and adjust pH to 7.0.
- Glass-distilled acidified water of pH 2.5: Same as that given under Zn estimation.
- Standard molybdenum solution: Dissolve 0.15 g of MoO_3 (molybdenum trioxide) in 100 ml 0.1M NaOH. Dilute to 1 litre to get 100 µg/ml Mo stock solution. Dilute 10 ml of the standard to 100 ml to get 10 µg/ml Mo.

- Working standard solutions: Take 1, 2, 3, 4, 5, 6, 7 and 10 ml of 10 µg/ml Mo standard solution and dilute each to 50 ml. This will give 0.2, 0.4, 0.6,0.8, 1.0, 1.2, 1.4 and 2.0 µg/ml Mo, respectively.

Procedure

1. Weigh accurately 5 g soil and transfer it into a 50 ml centrifuge tube.
2. Add 33 ml of 1M ammonium acetate solution to the tube, stopper and shake in a mechanical shaker for 5 minutes.
3. Centrifuge at 2000 rpm for 5 minutes or until the supernatant is clear.
4. Decant the solution into a 100 ml volumetric flask.
5. Repeat steps 2-4.
6. Make-up the volume to 100 ml with ammonium acetate.
7. Atomise the working standards on atomic absorption spectrophotometer at a wavelength of 313.5 nm. Prepare a standard curve of known concentration of molybdenum by plotting the absorbance values on Y-axis against their respective molybdenum concentration on X-axis.
8. Measure the absorbance of the soil sample extract and find out molybdenum content in the soil from the standard curve.

Calculation :

Content of Mo in the sample (mg / kg) = C µg / ml x 20 (dilution factor).

Where :

- C = Concentration of Mo in the sample, as read from the standard curve for the given absorbance;
- Dilution factor = 20.0(soil sample taken = 5 g and volume made to 100 ml).

ii. Estimation by colorimetric method

Apparatus

- Spectrophotometer
- Hot plate
- Refrigerator
- Water bath

Reagents

- 50% potassium iodide solution: Dissolve 50 g in 100 ml of double-distilled water (DDW).
- 50% ascorbic acid solution: Dissolve 50 g in 100 ml of DDW.
- 10% sodium hydroxide solution: Dissolve 10 g of NaOH in 100 ml of DDW.

- 10% thiourea solution: Dissolve 10 g in 100 ml of DDW and filter. Prepare fresh solution on the same day of use.
- Toluene-3, 4-dithiol solution (commonly called dithiol): Weigh 1.0 g of AR grade melted dithiol (51^0 C) in a 250 ml glass beaker. Add 100 ml of the 10% NaOH solution and warm the content upto 51^0 C with frequent stirring for 15 minutes. Add 1.8 ml of thioglycolic acid and store in a refrigerator.
- 10% tartaric acid: Dissolve 10 g in 100 ml of DDW.
- Iso-amyl acetate.
- Ethyl alcohol.
- Ferrous ammonium sulphate solution: Dissolve 63 g of the salt in about 500 ml of DDW and then make the volume to one litre.
- Nitric-perchloric acid mixture (4:1)
- Extracting reagent: Dissolve 24.9 g of AR grade ammonium oxalate and 12.6 g oxalic acid in water and make the volume to one litre.
- Standard stock solution (100 μg/ml Mo): Dissolve 0.150 g of AR grade MoO_3 in 100 ml of 0.1M NaOH, make slightly acidic with dilute HCl and make the volume to 1 litre.
- Working standard solution (1 μg/ml Mo): Dilute 10 ml of the stock solution to 1 litre.

Procedure

1. Weigh 25 g of air-dry soil sample in a 500 ml conical flask. Add 250 ml of the extracting solution (1:10 ratio) and shake for 10 hours.
2. Filter through Whatman No.50 filter paper. Collect 200 ml of the clear filtrate in a 250 ml glass beaker and evaporate to dryness on a water bath.
3. Heat the contents in the beaker at 500^0 C in a furnace for 5 hours to destroy organic matter and oxalates. Keep overnight.
4. Digest the contents with 5 ml of HNO_3-$HClO_4$ mixture (4:1), then with 10 ml of 4M H_2SO_4 and H_2O_2, each time bringing to dryness.
5. Add 10 ml of 0.1M HCl and filter. Wash the filter paper,first with 10 ml of 0.1M HCl and then with 10 ml of DDW until the volume of the filtrate is 100 ml.
6. Run a blank side by side (without soil).
7. Take 50 ml of the filtrate in 250 ml separatory funnels and add 0.25 ml of ferrous ammonium sulphate solution and 20 ml of DDW and shake vigorously.
8. Add excess of potassium iodide (KI) solution and clear the liberated iodine by adding ascorbic acid drop by drop while shaking vigorously.
9. Add one ml of tartaric acid and 2 ml of thiourea solution and shake vigorously.
10. Add 5 drops of dithiol solution and allow the mixture to stand for 30 minutes.
11. Add 10 ml of iso-amyl acetate and separate out the contents (green colour) in colorimeter tubes/cuvettes.
12. Read the colour intensity at 680 nm (red filter).

13. Preparation of standard curve: Measure 0, 2, 5, 10, 15 and 20 ml of the working standard Mo solution containing 1 mg/litre Mo in a series of 250 ml separatory funnels. Proceed for colour development as described above for sample aliquots. Read the colour intensity and prepare the standard curve by plotting Mo concentration against readings.

Calculation

Available Mo in soil (mg/g) = $$A x \frac{250}{200} x \frac{1}{25} = \frac{A}{20}$$

Where,
A = Mo concentration in µg / ml as obtained on X-axis against a sample reading.

4.6.4 Calibration of Soil test with crop response correlation

The soil testing and soil fertility management programmes have been given adequate importance for sustaining crop production and balanced fertilization in Indian Agriculture. Its implementation has gradually progressed and met with varying degree of success, depending upon areas and even according to the agency responsible for implementing the programme. In the initial stages based upon the simple fertilizer trials on farmers' fields and response to fertilizer application, soil fertility status was categorized into low, medium and high classes. The soil fertility management programme was strengthened with the setting up of All India Coordinated Research Project on Soil Test Crop Response Correlation and the Coordinated Research Project on Micronutrients in Soils and Plants in 1967 and the All India Coordinated Research Projects on long term fertilizer experiments in 1970. The emphasis on fertilizer prescription for a whole cropping system began by ICAR in 1980s. These programmes were launched during the initial period of green revolution when need for higher amounts and scientifically assessed fertilizer use for different crops and areas was recognized.

The soil-testing programme has remained an improvement of soil fertility evaluation activity in India. This has been the result of the work done by several soil testing laboratories in developing suitable methods for available nutrients, testing the suitability of these methods for making fertilizer use recommendation and obtaining the predictable yield responses. The soil fertility maps prepared by different authors are indicative of the fertility status of the soils and can thus be used for planning the types of fertilizer use and levels of nutrient required to be used in general. **Soil fertility maps, however, do not substitute for an individual soil analysis based fertilizer use recommendation which may have to take into account cropping system being followed by a farmer, level of fertilizer use in previous crops, and the management level of individual farmers**. Initially the agronomic field experiments were carried out on medium fertility soils. The rates of fertilizers were either reduced or increased by 30 to 50% empirically for soils rated as high or low

respectively. Ramamurthy and Velayutham (1971) reported an increase in the yield (11%) when the fertilizers were applied based on these adjustments over the general recommendation on fertilizer use made in a State.

Soil test based recommendations are often reported not to give response as stipulated. The following are some of the reasons identified for poor correlation between the soil test as estimated by the methods adopted in the soil testing laboratories and crop responses on a variety of soils.

1. The sample not being the true representative of the soil intended to be studied for correlation.
2. Improper selection of testing methods for different soils.
3. Imperfect analysis of the samples
4. Heterogeneity in the soils used for correlation work.
5. Heterogeneity in terms of crop management and agronomic conditions.
6. Nature of statistical functions chosen for correlation.

With the introduction of the fertilizer – responsive high-yielding varieties and intensification of multiple cropping under expanded irrigation facilities, the importance of balanced fertilizer use for increased crop production was well recognized by the Indian farmer. Since the fertilizer recommendations for crops based on simple field trials did not give the expected yield response; a need arose for the refinement of fertilizer prescription for varying soil test values for economic crop production. Against this background the following specific objectives were assigned to the ICAR's All India Coordinated Research Project on Soil Test Crop Response Correlation Studies which is operating at 17 centres in the country.

1. To establish relationships between soil test and crop response to fertilizers on representative soils in different agro-climatic regions of the country and from the results so obtained, to provide a basis for fertilizer recommendations for maximum profit per hectare.

2. To derive a basis for fertilizer recommendations for desired yield targets suited to the constraints of fertilizer availability and/or credit facilities to the farmers.

3. To devise a basis for making fertilizer recommendation for a whole cropping system based on initial soil test values.

The scientific support of All India Coordinated Research Project for obtaining soil test based crop response correlation data and also that from the Soils Departments of Agricultural Universities have helped making the soil testing program scientifically sound and more practicable in terms of getting predictable yield and thus helping the extension agencies in the

States for ensuring fertilizer use according to crop needs. However, the basis and criteria of standardization is not being adequately and fully communicated to the service laboratories for their adoption. In view of this, these laboratories, in many cases, are following the old criteria of classifying the soil nutrients into different classes derived from the field trials on local varieties of crops and corresponding fertilizer doses which are often low. In the changed situation where the crop varieties and fertilizer doses have undergone significant change, the rating chart or the basis of making fertilizer use recommendations by the soil testing lab. needed urgent change to achieve the objective. The Ministry of Agriculture had started to support STCR Project to compute the region wise/state wise latest basis of making fertilizer recommendations from the data generated under the scheme. Following this, the STCR Project is organising region wise training of soil testing staff and also a National Conference annually on soil testing to discuss such findings and disseminate the information among the soil testing staff so as to improve their skill. This has proved as a useful exercise. **However, the STCR project should provide block / district-wise ready reckner as a basis for making recommendations by the soil testing labs based on the soil test values obtained after soil analysis by the given method**.

4.6.5 Interpretation of soil test data and formulation of fertilizer recommendations

Since the Liebig era (1803 – 1873) to the mid-nineteenth century, many methods and approaches have been tried to obtain a precise or workable basis for predicting the fertilizer requirements of crops. An excess of a particular nutrient over another nutrient, which is in short supply could follow the phenomenon of the Law of Minimum (Liebig) which states:

- If a particular element is deficient, plant growth will be poor even when all other nutrient elements are abundant.
- If the deficient element is supplied, growth will increase.
- Increasing the particular element may not be helpful beyond this point as another element will be in short supply and limit the growth.

Some of the approaches followed for predicting the fertilizer requirement of the crops include :

(i) Generalized recommendations;

(ii) Soil test rating and fertilizer adjustment (Muhr et al., 1965)

(iii) Critical nutrient level in the soil or sufficiency concept (Krishnamoorthy et al., 963).

(iv) Fertilizer Recommendation for targeted yields (Truog, 1960)

(v) Fertilizer recommendation for a crop sequence (Ramamoorthy et al.,1971)

It shows that the efforts to refine the recommendation are going on as a continuous process.

I. Generalized recommendation :

These recommendations are made by the States after their bi-annual conference in association with the State Agricultural Universities. These are generally made as a single recommendation for the whole State for a crop. This is too general and does not include the properties of individual soil sample and associated agronomic and management factors of the individual farmer and his financial capability to purchase inputs.

II. Soil Test Rating and Fertilizer Adjustment :

In this approach, soil test value for a particular nutrient as obtained by a suitable analytical method, is categorized as low / medium/ high as per pre-determined rating chart prepared through soil test crop correlation studies carried out on the soil. General fertilizer dose is reduced or increased by 30 to 50% in case of high or low fertility status soil sample.

III. Critical Nutrient level in the soil or sufficiency concept :

In this approach, soil test value, for a particular nutrient, as obtained by a suitable analytical method, is categorized as low/ medium/high as per pre-determined rating chart prepared through soil test crop correlation studies carried out on the soil similar in the physic-co-chemical and biological properties, as the soil in question. The fertilizer nutrient application is then recommended for the given nutrient status of the soil. For the lower nutrient status, higher doses and vice-versa are recommended. For this approach, the rating chart and corresponding fertilizer doses are required to be continuously refined by STCR Project of ICAR as this is one of the objectives of the project. This approach is most commonly followed in soil testing labs.

IV. Fertilizer recommendation for a targeted yield:

The STCR project is concentrating on this approach which stipulates that the farmer will target his yield and thus would need recommendation for the targeted crop yield. The view against this approach is that the farmers generally do not target the yield in advance but would use fertilizer according to their resources. Thus the recommendation should be according to different stipulated levels of purchasing power of farmers rather than farmer targeting the yield in advance. In the yield targeting concept, the %age utilization of the applied fertilizer nutrient, and the contribution of soil nutrient is worked out for meeting the crop need as per the expected uptake of the nutrient by the crop (as per its composition) and for the stipulated yield target.

V. Fertilizer recommendation for the crop sequence :

In this approach, efforts are made to assess the requirement of nutrients for the crop rotation as a whole rather than for a single crop. The

assumptions become more completed in predicting the nutrient requirement for 2/3 crops at a time. The crop rotation itself may not remain fixed. Also the nutrient uptake and requirement for the succeeding crops may be greatly different from the assumptions made due to changing agro-climatic and seasonal changes for the subsequent crops.

In simple terms, it can be stated that the research laboratories including the soil-test-crop correlation project of ICAR are carrying out research work to establish a relationship between the soil available nutrient, applied nutrient (through fertilizer / organic manures, bio-fertilizer etc.) and crop response. In this exercise, soil nutrient is extracted through a particular chemical extractant where it is assumed that it has the similar extraction capacity which the plant roots will have and thus nutrient so extracted is assumed to be available to the crop plant. The applied fertilizer nutrient (Fertilizer / Manures) also contributes to the crop up take upto a certain amount of its content since whole of the applied nutrient is not possible to be absorbed by the crop. Numerous factors which influence the soil plant root environment such as moisture level, pH, micro-biological status, presence of various elements including beneficial and injurious to the crop, agronomic practices which influence the physical, biological and even chemical environment in the soil plant relationship determine the extent of availability of both the soil and applied plant nutrient.

It can be therefore, appreciated that the better correlation can be established and only through a large number of coordinated field trials by the research labs to formulate the recommendation for the fertilizer application on a given soil for a particular crop.

It may be noted that generally the percentage uptake of applied fertilizer nutrient (N,P,K) varies in different situations as follows:-

Nitrogen	- 30% to 60%
Phosphorus	– 10 to 30%
Potash	– 20 to 60%
Micronutrients Zinc	– 2 to 5%

The scope of improving the fertilizer use efficiency is enormous. A perfect soil test based fertilizer use recommendation and application of fertilizer nutrients in optimum combination and required doses can enhance the efficiency of fertilizer use and thus the profitability of fertilizer use in crops.

4.6.6. Preparation of soil fertility Maps, follow up and evaluation:

The individual soil test based fertilizer use recommendations are followed by the farmers. The data accumulated by the soil testing labs in each State can be made use for preparing soil fertility maps. These maps

may be prepared by the central / lead soil testing laboratory designated and equipped in each State.

While preparing soil fertility maps Parker's (1951) method of preparing nutrient index may be deployed as has been done by various authors in the past and explained in foregoing pages.

The maps may be prepared for :

(i) State as a whole by using soil analysis data accumulated by all the labs in the State. This map may be used for State level planning for fertilizer supply / procurement in the State and other policy related issues at the State level planning.

(ii) District level maps may be used for organizing fertilizer supply to the district. Block/village maps maybe used for recommending fertilizer use by the farmers in the block/village. **However, preference may be given to the village-wise map for making fertilizer use recommendation. The maps are not to substitute individual soil test based recommendation for a farmer. However, as stated earlier, there is a big gap between soil testing capacity and the number of farm holding, hence block/village level may be used for making fertilizer use recommendation.**

Taking various factors into consideration, it has been generally agreed among the scientists that the minimum number of the soil samples for a village / block level map may be **500.** Larger the number, better it is but the soil testing capacity is a limitation.

CHAPTER 5

SOIL TEST FIELD KIT

5.1 Principles of operation of Soil Test Kit

Soil test kits are tools used for rapid, on-the-spot, rough (approximate) determination of chemical properties of soils in the field. They are semi-quantitative devices in which the more elaborate laboratory analytical procedures of soil chemical analyses are simplified for rapid use in the field. Test kits are simple, quick and convenient to use, which make them very desirable as a means of diagnosing soil nutrient problems in certain circumstances.

The basis for rapid chemical testing is the quick calorimetric tests available for the levels of nitrate, phosphorus and potassium in soil extracts. The colour change is compared with calibrated reference colour charts or strips by observing with the naked eye. The colour hue on the colour chart that corresponds to or approximates the colour-change of the sample indicates the range of nutrient concentration in the sample, i.e. very low, low, medium or high nutrient levels in soil. In some brands of test kits, the ranges of nutrient concentrations designated as very low, low, medium and high are indicated numerically on the colour chart. Some brands, however, do not give such an indication. In any case very low, low, medium and high nutrient levels are interpreted as extremely deficient, deficient, fairly adequate and sufficient soil nutrient status respectively.

To remove the subjectivity of test-kit visual colour readings and thereby improve reproducibility and accuracy, reflectometers have been developed by some companies. This device replaces visual colour evaluation with quantitative assessment of colour change. The method combines the convenience of test-strip analysis with the precision of quantitative measurement. It is now available for pH, macronutrients, micronutrients and some heavy metals in soils.

Field test kits do not replace good laboratory analysis of soil since they are less accurate. However, their use becomes desirable under certain situations.

5.2 Uses of field Test Kit

i. *In lieu of a soil testing lab*: Although a test kit is not an alternative to a soil testing laboratory, it can fill this gap where such a laboratory is not available or not accessible to farmers. The kit can be used as a rough guide until a soil testing laboratory is available, functional and accessible

in the area. A soil management recommendation arising from test kit results is superior to a blanket recommendation.

ii. *To survey large areas quickly*: A very large area, e.g. a province or a region, can be surveyed for its soil nutrient status within a relatively short period.

For example, selected area can be subjected to quick soil tests using several teams of surveyors. Through this exercise, valuable information will be obtained about individual fields; in addition the overall picture of the soil nutrient status of the entire area will emerge. This gives the agricultural worker or the farmer an idea of the fertility need of the area.

iii. *To verify suspected fertility symptoms*: When an apparent deficiency symptom appears on a plant in the field, a soil test with a test kit can be made to ascertain quickly whether or not it is the suspected deficiency.

iv. *To call attention to the need for laboratory soil tests*: Test kits can be used quickly to determine whether a plant nutrient is deficient (very low to medium range), or adequate (medium to high range), in soil. If found deficient, the farmer is then advised to get the soil tested in a soil testing laboratory for accurate soil test values and soil management recommendations (In Quick Fertilizer Testing Kit, a similar approach is suggested).

v. *To supplement routine soil testing*: A test kit can be used as a monitoring tool to check if a fertilizer recommendation is adequate and appropriate for a particular field. Extension officers can follow up on the recommendations with field tests. If the recommended level does not give the expected (desired) plant growth and plant-tissue nutrient status, it could be because the farmer did not follow the recommendation or that the recommendation was based on an inaccurate soil-test result from the soil testing laboratory. Another soil sampling and laboratory test may be suggested following the test-kit evaluation.

vi. *To follow nutrient uptake in research fields*: Treatment plots of field calibration studies can be checked to monitor if and when the crops are taking up nutrients. This information can enhance the understanding and balanced interpretation of results of the studies. For example it can indicate the most active nutrient-uptake stage of crop growth when it is important to ensure fertilizer nutrient availability at the root surface.

5.3 Advantages of Soil Testing Kits

i. It is simple, quick and convenient to use. A test for N, P and K can be completed in less than five minutes once the sample has been collected. It is convenient because the kit is contained in a small box (pocket laboratory) which can easily be carried to even the most remote rural field locations.

ii.It is much cheaper than laboratory testing facilities; therefore it is available for the use of farmers.

iii.The test can be carried out on the spot where the problem exists and the facts and conditions related to the problem are fresh in mind. The field testing provides the kit user with immediate answers to nutrient problems.

iv. It cuts down on the cost of time, transportation and materials that may be needed to carry soil samples to the laboratory for analysis.

v. It provides a much better guide than blanket fertilizer recommendations that may be adopted in the absence of a functional and accessible soil testing laboratory. It can be used as a rough guide until soil testing laboratory facilities are available.

vi. It enables the literate and enlightened farmers to conduct their own on-the-spot analysis and interpretation of the test result without the assistance of an official extension agent. This is very important in India where the ratio of extension worker to farmer population is very low.

5.4 Limitations of field Test Kits

The following limitations of field test kits must be borne in mind when using the kits and interpreting the test results.

i. The results obtained are less accurate than laboratory test results because they are deduced by visual colour observations. The results will therefore be influenced by the sharpness of the eye (visibility) of individuals conducting the test. The recent development of reflectometery for colour strip analysis has, however, removed the subjectivity of visual colour evaluation.

ii. Analysis of Trace and secondary elements are not considered in most brands of test kits.

iii. Guidelines for interpretation of test results in some brands of kit do not state the nutrient quantities represented by qualitative (descriptive) statements of levels such as "low fertility" or "high fertility", based on field calibration of soils in a given climatic region.

iv. Phosphate reagents of most test kits go bad a few months after preparation. Yet it takes quite some time for kits to arrive after ordering from the source. Besides, most kits can do only 50 soil samples at a time with the provided solutions / reagents. Refills have to be continually ordered and this means that the user must depend on, and constantly be in contact with the manufacturer.

v. Mixing and subsampling moist soil in the field can hardly achieve satisfactory representative sampling. It is even more difficult with very clayey soils.

5.5 Soil Sampling and Preparation for Field Testing

Soil sampling for field testing follows the same procedure as sampling for laboratory testing (see section on Soil sampling). Accurate, representative sampling is as essential here as sampling for a regular routine soil testing programme.

Sample preparation for field tests requires more patience and thoroughness on the field than is normally required in a regular routine soil testing programme. Since the soil sample will not undergo the process of drying, grinding and sieving, hence, thorough mixing of the freshly collected sample is imperative. A clean plastic bucket is used to collect the composite sample that is then thoroughly mixed by hand before sub-sampling for field testing. As satisfactory mixing of undried fresh soil samples is difficult; but it is possible if carefully done, especially when one realizes the implications of testing non representative subsamples after careful representative sampling of the field. A part of the fresh soil sample is taken to laboratory to determine water loss on drying i.e. moisture content on an air-dried basis. This may be used to correct the field-test results to make them comparable to both laboratory analysis and other field-test values.

5.6 Testing of the sample

First Quick Soil Testing Kit was developed by IARI in early sixties. Following which, many Agricultural Universities /Scientific equipment manufacturers have developed soil testing kits having their own standards. Basic principle of estimation is same as in a given quantitative chemical analysis but the estimations are qualitative in nature and often based on colour development, which is expected to be corresponding to the quantity of the nutrient in question and is compared visually with the standard so fixed and not instrumentally estimated. Hence, qualitative in nature.

Quick Testing Kit, however, could not become popular in the country. During 1980, ICAR had taken up a scheme of training un-employed youth on the use of kits and then providing them with kits to test soils from farmer's field on nominal charges and advise the farmers about fertilizer use. The scheme did not become popular and was discontinued. Soil testing kit, however, is useful for a broad assessment of soil nutrient at a low cost. Progressive farmers and fertilizer dealers can make use of the kits.

CHAPTER 6

MOBILE SOIL TESTING LABORATORY (VAN)

6.1 Aims, objectives and its operation in the field

Soil Testing Service was initiated in India during 1955-56 with the establishment of 16 soil testing labs under the Indo-US Operational Agreement for Determination of Soil Fertility and Fertilizer Use. The programme has been gradually expanded with a setting up of more of soil testing laboratories. Since the stationary labs can not have direct contact with the farmers, it was felt, in the initial stages of the programme, to set up mobile soil testing labs also so that the samples could be collected and analysed on the spot in presence of farmers. The programme is being expanded and at present, there are 120 mobile labs in the country. The mobile soil testing laboratories are similar to the stationary laboratories with regard to staff, type of equipment, facilities and the testing methods for soil. The facilities of testing are mounted on a Mobile van suitably fabricated to house the equipment and facilities. The vans are also provided with the audio-visual facilities so as to address the farmers and show them films related to the agricultural development including fertilizer use etc. The films on sample collection and fertilizer use methods etc. are also shown.

The mobile labs are taken to the desired area. Samples are collected from the fields by associating the farmers with the sample collection. The analysis is carried out in the village by locating the van at a suitable site. Recommendations are handed over to the farmers personally in the village and details are also explained to them. Stationary labs often suffer from the lack of authenticity of the sample received but the mobile labs staff collect the sample themselves and thus assured of greater accuracy and authenticity of the sample.

In principle, mobile labs have greater advantage of being in direct contact with the farmers than the stationary labs but in actual practice, it is seen that the mobile vans/ labs run into operational problem more particularly with regard to the following:-

(i) Size of the fabricated van (vehicle) is rather large, being 740 cm(L) X 244 cm (W) X 295 cm(H), which gets into operational problem while running on small / kuccha roads in the interior of the villages in the remote areas.

(ii) The vehicle requires frequent repairs.

(iii) It is also observed that in some cases, it is being used as a means of transport only. Thus the mobile labs gets into disuse.

(iv) Smaller vehicles can not be alternative since a large number of equipment, facilities and movable working labs etc. are required to be taken in the van apart form providing the space for the staff of 8.

(v) Though provision is made to house the equipment in a secured manner in the van, still equipment get damaged / go out of order while the vehicle runs on uneven and kuccha roads. Many equipments are delicate hence cannot withstand the jerks when the vehicle moves. However, in spite of the shortcomings, the advantage of analysing the samples on the spot and making recommendations personally to the farmers, outways the shortcomings mentioned above. Hence, on balance it can be said that mobile labs are to be continued.

6.2 Design of a mobile van

Pre-fabricated mobile vans are supplied by the fabricators in the country to be used as mobile labs after furnishing and providing equipment. However, a plan of the mobile van along with its compartments for housing equipment and facilities is described below:-

A typical mobile soil testing laboratory is built up on a chassis having 4225 mm (165 inches) wheel base, full forward control with face grill, front and rear shock absorbers, auxiliary rear springs.

The body of a typical mobile van can be divided into two compartments:

a. Passenger compartment – to accommodate seven laboratory staff and Driver during transit.

b. Laboratory compartment with full laboratory fixtures and furnishing.

The total outside dimension of the body may be as follows :

Length	: 740 cm
Width	: 244 cm
Height (from ground)	: 295 cm

The passenger compartment has to have inside dimension as follows:

Length	: 215 cm

Width	: 226 cm
Height	: 190 cm

The laboratory compartment has to have inside dimension as follows :

Length	: 485 cm
Width	: 230 cm
Height	: 190 cm.

Besides the above, there are five compartments fabricated below the chassis. Out of these, two compartments are on the same side as the petrol tank. The longer of the two compartments is meant to be used for carrying the canvas tents with accessories and the shorter compartment is meant to be used for carrying folding steel furnitures. The three compartments on the side away from the petrol tank have been designed to carry sulphuric acid jars, petrol, kerosine and the portable folding sinks with stands.

In the passenger compartment, the seats for the passengers are to be built over suitably designed compartments fabricated with 16 SWG M.S. sheets. These seats are hinged to serve as lids of these fabricated compartments for safe-keeping of cooking utensils and other accessories for use of the field party during stay in camps.

Two wooden boxes may be provided in the passenger compartment for fixing the battery and carrying petromax and hurricane lanterns.

The laboratory compartment may have the following details:

1. Rear door (double) with handle and lock. Retractable steps are provided outside this door.
2. Two service doors. One on each side with tower bolts inside, handle and lock outside.
3. Windows with sliding safety glass panes and inside locking arrangements and net curtains – three on each side.
4. Work benches : One on each side built in with the body having length – 485 cm, width – 60 cm. and height – 90 cm. These work benches have below them specially designed compartments for housing the scientific instruments, glasswares and audio-visual equipments with suitable shock-absorbing lining and packing materials. The portions of the work benches above the service doors do not have compartments, in order to facilitate free movements of stands, chemicals or extractants from inside to outside and vice-versa according as the various extraction procedures and filtration are being carried out. These void portions of the working benches are suitably reinforced with angle iron pieces for mounting the shaking machine on one side and a portable air-compressor and a voltage stabilizer on the other.

5. Two fans are provided to the laboratory portion of the van at the rear wall and an exhaust fan is fitted on the partition between the passenger compartment and the laboratory compartment.
6. Four tube lights 40 watts 24" long are fitted to the ceiling of the laboratory compartment with detachable plexiglass cover.
7. Other electrical fittings: Three pin plug points with switch:
   5 Amp. – 6 (3 on each side just above work bench connected through voltage stabilizer).15 Amp. – 3 (to operate shaking machine and compressor and centrifuge)
   Main switch with 5 Amp. Fuge – one
   Main switch with 15 Amp. Fuge – one.
8. A rack of 197 cm length and 22 cm width is provided just below the roof for housing of the Projector screen, made of 11/2" aluminium angles and expanded metal base provided with canvas belt and buckle for tying the screen.

Besides the above, eight boxes made of teak boards 1 ¾ " thick may be provided. Four of these may have the following dimensions:

Length ..100 cm.
Width ...66 cm.
Height ..57 cm.

and the other four have the dimensions as :
Length ..100 cm.
Width ..66 cm.
Height ..37 cm

These boxes have to have arrangement for clamping when the smaller ones are placed above the bigger ones so that they form a single rigid unit during transit.

There is a soil sample storage box of the following dimensions:
Length ..75 cm
Width ..68 cm.
Height ..76 cm.

For carrying acid jars and kerosene and petrol, two foam rubber padded wooden boxes made of 1 ¾ " thick teak boards are provided.

In view of limited working space inside the laboratory portion of the van, provision is made for a sheltered space covered by canvas on one side of the van away from the petrol tank. This space is used for keeping the wooden boxes when the van is vacated and these boxes serve partially the purpose of laboratory working table for filtration etc. This has been considered necessary for providing more working space so that the daily targets of analysis can be easily achieved.

6.3 Staff requirement of the Mobile Soil Testing Laboratory

In order to make the mobile soil testing laboratories effective and to make them function at their maximum efficiency the work pattern for the handling of the soil samples must be well organised. Adequate and efficient staff forms the basis of such well organized work pattern. The staff requirement for a mobile soil testing laboratory having a sample analysis capacity of 10,000 soil samples per year is as follows :-

Sl.No.	Particulars	Number	Qualifications required
1.	Asstt. Soil Chemist	One	M.Sc.(Soil Science / Agri..Chemistry) or B.Sc.(Ag.) / Chemistry with 5 years experience in soil testing / soil survey / fertilizer testing
2.	Analytical Assistants	Three	B.Sc(Ag.) /Chemistry with 3 years experience in soil testing / soil survey / fertilizer testing
3.	Laboratory Attendants	Two	High School Pass
4.	Projector-Operator-cum- Typist	One	Trained in the relevant line
5.	Driver	One	With proper licence
	Total	**Eight**	

A mobile laboratory with recommended number of staff can collect and analysis at least 50 soil samples in an average working day of seven hours, including the time required for making out the fertilizer recommendations. The mobile

laboratory can operate in the field for a period of six months in a year, handling a total of 5,000 soil samples. The staff is also required to show films on sample collection / analysis and also other fertilizer use and promotion aspects. During the remaining part of the year, it can be stationed for work near the standard stationary laboratory, to which it is attached and in this way can analysis an additional 5,000 soil samples per year.

To avoid hardship to any one set of staff by having to stay out in the field for long periods, it is suggested that the staff appointed for the mobile laboratory and the staff in the standard stationary laboratory, be rotated periodically.

The sampling methods, processing the samples and their analysis is done by the same procedures and methods as described in case of stationary laboratory (**Chapter 4**). Preparation of analysing report

and framing of recommendations is also done in the identical manner as in case of stationary lab.

6.4. Details of required facilities

The following details are provided at **Annexure-14**

(i) Required equipment for different estimation.
(ii) Required chemical reagents and glass wares
(iii) Supporting scientific equipment.
(iv) Polyethylene, porcelain and wooden items
(v) Miscellaneous and audio visual aid.

CHAPTER 7

GENERAL OBSERVATIONS

7.1. Shortcomings in the soil testing programme

(i) Sample collection is not often very representative of the field intended to be given recommendation for fertilizer use. Farmer is often not involved in the sample collection. Thus he does not appreciate the importance, value and content of fertilizer use recommendations and does not follow it for various other reasons also.

(ii) Analysis reports / recommendations are not received by the farmers in advance for purchasing fertilizers. There is a big time lag between sample collection and receipt of report.

(iii) Laboratory equipment are often not calibrated. There is no system of inter-intra lab soil analysis check, hence, accuracy of analysis is not ascertained and soil analysis often may not be accurate, thus recommendation arising out of such an analysis is not expected to be sound.

(iv) The Incharge of the labs, many a times are, not soil scientists. Hence analysis and interpretation of results do not have adequate technical input.

(v) Quantity of the chemicals are often not supplied according to the sample analysing capacity while the labs are expected to work as per the target set for the year. This situation results in poor quality of work or under utilization of already existing low capacity.

(vi) There is no system of regular / periodical training of the lab staff, thus, the staff does not remain in touch with the latest available equipment / method of testing and formulating recommendation etc.

(vii) Soil Testing Service is free of any fee/ charge in most of the States. Only some States are charging nominal fee which does not call for the seriousness of farmers, hence, their involvement in the programme is not much.

8. Soil Testing labs do not get the feedback on the outcome of their recommendations and have no chance of improving / modifying the recommendation based on the outcome of various recommendations made in the past.

9. The initial system of attaching an agronomist with the soil testing labs, to maintain a linkage with the labs and the farmer to ensure implementation of recommendation has been discontinued.

10. Most important aspect is the use of old **'rating chart'** by many labs to classify samples into different categories of nutrient status. This aspect is most important requiring special attention of ICAR/STCR Project/State Governments to ensure that the rating charts provided to the laboratories are updated on priority basis.

7.2. Suggested remedial measures for improvement in the programme

(i) **Lab Management** :

(a) Each state may identify one better equipped and properly staffed laboratory in the State to be designated as a **central lab/lead lab**. This lab may maintain a working liaison with the Department of Soils in one of the agricultural universities in the State. This laboratory may be used as a training place for the technicians of the soil testing labs with the technical support from the university. This laboratory may work out a system of 'Check Sample' with the University lab to check and ensure that the capability and the practice of sample analysis is adequate in the soil testing labs. Periodically (annually) the university lab may check the analysis of 100th , 500th and 1000th sample in each of the laboratories in the State and record a certificate in this aspect. Adequate financial support may be given for such an analysis to the University lab.

(b) Equipment of the central lab may be periodically calibrated /got checked with the help and involvement of a soil scientist from the university. Such checks may be recorded. An arrangement may be worked out between State Agriculture Department and the Universities to identify a Senior Soil Scientist in the Universities who may work as a Coordinator / Counterpart to the Incharge of Lead Soil Testing Laboratories. Equipment of the other labs may be checked by the Incharge of the central soil testing lab.

(c) Automation of analysis may be introduced in the Central Lab **(Annexure-6B)**

(d) Equipment, chemicals, glassware and other miscellaneous lab items may be maintained as per the analysing capacity of the labs. List of equipment, chemicals and glassware required for a soil testing lab having a capacity to analyse 10,000 soil samples and 200 water samples /year is given at **Annexure-6.**

(ii) **Man Power:** Each laboratory may be provided with the required staff, according to its capacity. Each laboratory may be headed by a technical person having M.Sc. (Soil Science & Agri. Chemistry) as an essential qualification or B.Sc.(Ag.) with a minimum of 5 years experience of working in soil testing / soil Survey / fertilizer testing lab. **There should be no relaxation in this stipulation so that the technical flaw in the programme is removed**. At least new labs being set up with the central assistance may adhere to this requirement to start with, without exception or relaxation or else do not start a lab.

Requirement of staff in a soil testing lab having a capacity of analysing 10,000 soil samples / 200 water samples per year and that for a central/lead laboratory is given in **Annexure-2.** It may be noted

that the incharge of the central lab may be at the level of Joint Director of Agriculture in the State and at least 8 years of experience in the field of required specialization.

(iii) **Training :**

(a) Central lab may organize a 3 days annual orientation training of technician during lean period of sample analysis work in the laboratory.

(b) Orientation training of 'lab incharge' may be organized once in a year for a period of 3 days in the agricultural university.

(c) Incharge of the central lab may participate in the kharif/ rabi Conferences being organized by States to formulate various recommendations relating to input use / crop variety etc.

(iv) **Sample Collection** : Special care may be taken for collection of representative soil samples as outlined in para 4.6.2. Authenticity of the samples has to be ensured at all levels – starting from collection stage to its storage in the lab even after analysis.

(v) **Timely communication of report** : Since the reports are often not received in time by the farmers, when sent through usual postal system, a system of online communication of reports may be started by which the soil testing laboratory may send the report to the Block Development Officer (BDO) to at least cut the postal delays. The farmers often visit BDO's office for various other activities and may be able to collect reports. This however also presupposes that all the soil testing laboratories are provided with computer facilities. Keeping the cost in mind, the system of on-line communication reports may be started in the selected laboratories initially and then to cover all the labs.

(vi) **Feedback on recommendation** : The laboratories may be kept informed on the outcome of the recommendations made by them on fertilizer use at least on representative and typical case by case basis, e.g. where the recommendation has given as expected / better than expected results and where it has not given results as expected.

(vii) **Assign a post of Agronomist** at the level of Deputy Director of Agriculture in the State to the Central laboratory. This officer will ensure an effective and live linkage between the field and the laboratory.

(viii) **Adoption of village by the lab**:

To start with, each lab may adopt at least one nearby village from where samples may be collected by the laboratory staff and

recommendations are also communicated / handed over directly by the laboratory staff to the farmers and to follow the outcome of the programme. Each lab can take up one village as a mission to see the utility of the programme by itself and find out shortcomings so that the whole programme can be improved on the basis of such direct observation / study. Presently, the labs are literally cut off from the field and work in isolation of the whole programme.

(ix) **Charging Testing Fee :** All the states may start a system of charging a fee for sample analysis. Some States are already charging the fee which varies from Rs.5/- per sample to Rs.10/- per sample. Charging the fee will bring an accountability on the part of the lab to make a sound recommendation because farmers will participate in sample collection or at least will know that a sample has been collected and will be expected to appreciate the value of the report received on the basis of some cost borne by them. They will start asking the question if report is not received in time or is not found to be useful when the recommendation is followed as advised by the lab. Charging the fee will also help the states to supplement the requirement of funds by the laboratories. A minimum fee of Rs.20 per sample analysis may be charged. Estimated cost of analysis of a sample is approximately Rs.80 for physical parameters + NPK analysis while with the micronutrients it would be about Rs100 (Only chemicals and 20% of glass breakages are considered as part of the cost for this purpose).

(x) **Improvement in Rating Chart** : A very critical aspect of the programme is to improve and update area wise categorization of soil nutrient content into different classes such as low, medium and high or into larger number of categories such as, very low, low, medium, high and very high etc., and based on these categories, make fertilizer recommendation for the given soil / crop. This categorization is based on soil test crop correlation studies being carried out by STCR programme of ICAR. **The STCR is expected to provide the criteria preferably at block level and districts level.** A single basis for the state as a whole has a lot of limitations.

The geographical boundaries of a block / district may not necessarily correspond exactly to the soil type based agro ecological areas studied by STCR ; hence the recommendations may be applicable to more than a block. Therefore, laboratory-wise ready- reckner rating chart may be prepared since labs are also generally district-wise.

(xi) **Soil Fertility maps:**

Latest National level maps were prepared in May, 2002 for N,P and K status of state-wise soil fertility. These maps were based on a total of 3.67 million soil samples analysed by the laboratories in States during 1997-1999. Nearly 10 years have passed and it is expected that the appreciable changes may have occurred in the soil fertility to require updating of these maps. The ICAR has since been assigned the task for preparing National

level/ State-wise soil fertility maps. State-wise/ district-wise / block-wise and if possible village-wise map may also be prepared by the lead / central laboratory in the States, based on the soil analysis done in the respective State soil testing laboratories. The village-wise maps may be prepared if the number of soil samples is at least 500 representing a given area / village. For this, the formula as given by Parker (1951) and adopted by Indian scientists, as explained in the foregoing pages may be followed.

(xii) Soil Test based fertilizer use demonstrations may be carried out since a large number of farmers are still not aware about the benefits of the soil test based and balanced fertiliser use recommendations.

(xiii) **Strategies to use limited soil analysis facility fruitfully:**

(a) As per existing practice, analyse all the samples sent directly by the farmers, on a priority basis and make fertilizer use recommendations. Ensure that all such recommendations reach the farmers before sowing period starts. Follow up these recommendation and keep a record of success of the recommendation.

(b) Start the practice of taking "**composite samples**" by the extension agencies. Composite samples may represent farms / fields having broadly similar landscape, soil type, crop, management and the yield goals etc. Only one representative sample may be collected from such farms which may represent an area of 20 to 50 hectares comprising 10 to 20 farms or even more, all of them having one recommendation based on a composite sample collected and analysed from these farms. While doing so, if some piece of land is visually seen to be greatly different from the adjoining fields, such fields may be sampled separately and may not be made a part of the Composite-sample. The system of composite soil sample analysis may be made a focus of the programme along with samples being directly received from the farmers.

(c) Fertilizers may be used with the help of Fertility maps:

There is no substitute to making recommendation for each and every farm separately. This will, however, never be possible in foreseeable future for reasons of practicability and requirement of funds. Nearly equally meaningful recommendation, as could be based on a farmer specific soil analysis, for fertilizer use is possible if village-wise/block-wise fertility maps are prepared based on properly collected soil samples and accurately analysed by the laboratories. A reasonable assumption has to be made that broadly the similar looking soil having identical vegetation, topography and management etc., on a fairly large area, are similar in broad fertility status. In contrast to this, such farms in the village which are conspicuously different from one another may be sampled separately and analysed separately for making recommendation because their fertility level is expected to be different. The fertilizer use recommendation if made village-wise, combined with the recommendations for individual farms,

where individual samples were analysed, will be a practical approach to use fertilizers scientifically, keeping in view, the limitations of available facilities.

The state level recommendations made for a crop for the state as a whole may be replaced by above approach.

(xiv) **To Improve the quality of analysis / recommendations**

It is meaningful to set up more laboratories, but more than that, it is important to improve the quality of many of the existing labs, both in terms of providing modern equipment, qualified staff and by organizing periodical trainings for them. This will help the laboratories to improve the quality of the recommendations which will in turn increase the faith of farmers in soil test based recommendations.
Funds not being unlimited, if priority is given to the improvement of the existing labs, it will be money better spent than continuing with the poor / inadequately furnished labs having unqualified or untrained staff or persons with unrelated qualifications from where often technically unsound and delayed recommendations are made, as per often received comments on the existing programme

7.3 Farmers' acceptance of soil testing service:

The general impression about soil testing is that it is a rapid and not too accurate a method of assessing the plant food elements that are deficient in a soil and that if one applies these deficient elements, good yields are obtained. A close study will reveal that accurate soil testing is a complex set of scientific procedures involving accurate analytical methods. Each recommendation based on a soil test takes into account the values obtained by these accurate analyses, research work conducted on the crop in the particular soil, areas and the management practices of concerned farmers. The soil test with the resulting fertilizer recommendation is, therefore, actual connecting link between agronomic research and its practical application to the farmers' field. However, soil testing is not an end in itself. It is a means to an end. A farmer who follows only the soil test recommendation may not necessarily be assured of a good crop yields as they are also the result of application of other sound management practices, such as proper tillage, efficient water management, good seed and adequate plant protection measures. **Soil testing is essential, however, as the first step in obtaining high yields and maximum returns from the money invested in fertilisers**. The awareness of the farmers about the benefits of soil testing is too inadequate. In terms of samples received in the laboratories only 8-10 % of the samples are sent directly by the farmers for analysis. As per the High Powered Committee on Fertilizers Consumption (1987) only 6.74 % samples were received in the laboratories directly from the farmers. Special efforts are required to bring about farmers' awareness and make the programme a farmer oriented activity.

7.4 Role of Extension Workers in Soil Testing and Success of the Programme

Soil analysis and fertilizer recommendation is only a part of the soil testing service. To a good measure, the efficiency of the service depends upon the care and efforts put forth by extension workers and the farmers in collection and despatch of the samples to the laboratories and obtaining reports timely. Its effectiveness also depends upon the proper follow up in conveying the recommendations to the farmers, including the actual use of fertilizer according to the recommendations. The role of extension service, soil chemists and the agronomists in the field is important.

The published data have established a superiority of soil test based fertilizer recommendation beyond any shade of doubt over generalised recommendations made by the states. It is ironical that inspite of the proven benefits of the soil testing service for the farmers, the service is suffering both from technological aspect and due to inadequate and untrained manpower. Weakness of the programme in its various aspects as discussed above needs improvement.

7.5 Soil Health Cards

The soil test data are made use for making fertilizer use recommendation by the soil testing laboratories. These data are also used for preparation of soil fertility maps. The soil analysis basically aims at assessing the fertility status of the soil. This information along with the additional information on the farmer's land may be presented to the farmers in the form of soil health cards. The additional information may relate to the relevant revenue record of farmer's field. The soil health card so issued to the farmers may be periodically updated so as the farmers are aware about the changing fertility status of their land. This card may also be useful to the farmers in getting loans for agriculture purposes where agricultural value of the land may be one of the factors. A format of soil health card is at **Annexure -13.** The states may make suitable modification to the aspects which may be relevant to a specific state.

7.6. Government Policy on soil testing and financial support

It is well recognized that the soil test based fertilizer use, results in balanced, efficient and profitable fertilizer use. Soil testing programme is, therefore, being supported by Govt. of India through different plan periods and have also advised the states to provide support to this activity, from time to time.

The Union Ministry of Agriculture had set-up mobile vans during 1970s, provided Atomic Absorption Spectrophotometers during 1980s, gave financial aid during 7th plan for strengthening some of the existing labs and also to set up new labs. Support was given for organising training of the soil testing staff under macro management scheme throughout the country

during 1992-95. During XI Plan, the Union Ministry is providing a substantial support to set up new soil testing laboratories, mobile soil testing vans and to strengthen the existing laboratories. Funding pattern is at **Annexure-1.**

Governments' recent policy change on fertilizer subsidy w.e.f. 01.04.2010, stipulates that fertilizers subsidy will be worked out on the basis of their nutrient content. This would ensure that special attention is paid on the individual soil nutrient deficiency and application of fertilizers on the basis of such deficit nutrient. It would further require the formulation of fertilizer products according to the needs of nutrients in a given soil / crop. This would be possible only when the soil testing labs are in a position to give information on soil nutrient deficiencies on smaller area basis, say village-wise, if not on individual farmer's basis. This will further emphasise on the need of strengthening the soil testing service in the country both in quality and capacity. In the new policy of giving nutrient based fertilizer subsidy, a specific emphasis on 'Nutrient' will focus on nutrient-wise soil deficiency and the production and promotion of fertilizers according to the need of such deficient nutrient. This will call for greater attention on the use of soil nutrient deficiency based fertilizers. However, this policy will ensure that no fertilizer gets less or more emphasis than the other due to any consideration such as, production technology or use of raw material and thus, on the basis of cost of production etc. It will ensure uniformity of subsidy in all types of fertilizers.

Type of soil and crop are two most important factors which govern the need for type and amounts of nutrient required in a given area. Hence, apart from soil testing, priority may be given to the type of crops. A study (Chanda, 2008) on fertilizer use by various crops reveals that food crops consume 72% of the total fertilizer nutrients used in the country. The crop-wise fertilizer use is as under :-

S.No.	Name of the Crop	% of total fertilizer consumed
1.	Paddy	37
2.	Wheat	24
3.	Coarse Cereals	8
4.	Pulses	3
	Total	**72**
5.	Oil Seeds	9
6.	Fruits	2
7.	Sugarcane	5
8.	Vegetables	4
9.	Cotton	3
10.	Others	5
	Total	**28**

Individual farmer and individual crop may continue to be given attention but from the overall perspective, paddy and wheat continue to be dominant crops consuming over 60% of the total fertilizers used in a country. Hence, to reduce the fertilizer subsidy, special attention of improving the fertilizer use efficiency may be given to these crops.

If the fertilizer industry will venture to produce and promote the products on the basis of requirement of specific soil nutrient deficiency, the industry will have to get into the soil testing programme in a big way and generate such information as a measure of good supplement to soil testing programme basically being run by the Government. The fertilizer industry may adopt at least one district in a State and ensure and monitor that the fertilizer in the adopted district is used on the basis of plant nutrient deficiency as determined through accurate soil testing.

CHAPTER 8

WATER ANALYSIS

Irrigation water, irrespective of its source, always contains some soluble salts. The suitability of waters for a specific purpose depends upon the types and amounts of dissolved salts. Some of the dissolved salts or other constituents may be useful for crops but the quality or suitability of waters for irrigation purposes is assessed in terms of the presence of undesirable constituents and only in a limited situations, the irrigation waters is judged as a source of plant nutrients. Some of the dissolved ions such as NO_3 are useful for crops.

The limits of purity established for drinking water, water to be used for industrial purposes and for agriculture are different. It may, therefore, be possible that a water which is not good for drinking and industrial use, may be quite suitable for irrigation. This publication deals with irrigation waters only. The most important characteristic that determine the quality of irrigation waters are:

1. pH
2. Total concentration of soluble salts judged through electrical conductivity (EC).
3. Relative proportion of sodium to other cations such as Ca and Mg referred as Sodium Adsorption Ratio (SAR).
4. Concentration of boron or other elements that may be toxic to plants
5. Concentration of carbonates and bi-carbonate as related to the concentration of calcium plus magnesium referred as Residual Sodium Carbonate (RSC).
6. Content of anions such as chloride, sulphate and nitrate.

The analytical data on the above parameters are used to describe the quality of irrigation water taking standards fixed for each aspect as an index.

Some waters coming from industries as effluents and domestic waste water as sewage may contain some specific plant nutrients also. These waters may be useful for irrigation of field crops if assessed for toxic/pollutant metals, and organic and microbial constituents with regard to their suitability or otherwise. Determination of organic constituents is generally carried out under two categories: i) organic substances which quantify an aggregate amount of organic carbon, and ii) individual or specific organic substances such as benzene, DDT, methane, phenol, endosulfan, etc. Important determinations are the chemical oxygen demand

(COD) which gives the total organic substances and the bio-chemical oxygen demand (BOD) which gives the amount of total biodegradable organic substances in the water sample.

To keep the scope of this publication tailored to Service laboratories, only parameters of practical utility to judge the quality of commonly used irrigation waters are described. Thus, the aspects pertaining to the use of effluents and sewage waters have not been covered. Similarly, the aspect of assessing water as a carrier of plant nutrients also has not been covered.

8.1 Important indices of practical utility to judge irrigation water quality

The following standards are laid down :

i). Electrical Conductivity (EC)

The concentration of total salt content in irrigation waters, estimated in terms of electrical conductivity (EC), is taken as the most important parameter for judging the suitability of irrigation waters. Generally, all irrigation waters having less than 2.25 mS/cm conductivity are considered suitable except in some unusual situations like very sensitive crops and highly clay soils having poor permeability. Ideal value is less than 0.75 mS/cm and the waters widely used have the values between 0.75-2.25 mS/cm (Richards, 1954).

ii). Sodium Adsorption Ratio (SAR)

It is calculated to indicate the sodicity or alkalinity hazard of irrigation waters.

$$SAR = \frac{Na^{+}}{\left[\frac{Ca^{2+} + Mg^{2+}}{2}\right]^{1/2}}$$

Where, the concentration of cations is in me/litre.

Based on the value of SAR, waters can be rated into different categories of sodicity as under (Richards, 1954)

Safe	< 10
Moderately Safe	10-18
Moderately unsafe	19-26
Unsafe	> 26

iii). **Residual Sodium Carbonate (RSC)**

This index is important for carbonate and bicarbonate rich irrigation waters. It indicates their tendency to precipitate calcium as $CaCO_3$. RSC is calculated as below.

RSC (me/litre) = $(CO_3^{2-} + HCO_3^-) - (Ca^{2+} + Mg^{2+})$

Concentrations of both cations and anions are in me/litre. Sodicity hazard in terms of RSC is categorized as under (Richards, 1954).

Safe	<1.25
Moderate	1.25 – 2.5
Unsafe	>2.5

The limits can vary depending upon types of soils, rainfall and climatic conditions. Higher RSC values can be considered safe for sandy soils in high rainfall area (> 600 mm/annum).

iv). **Mg/Ca ratio**

It is widely reported that calcium and magnesium do not behave identically in soil system and magnesium deteriorates soil structure particularly when waters are sodium dominated and highly saline. High level of Mg usually promotes higher development of exchangeable Na in irrigated soils. Based on ratio of Mg to Ca, waters are categorized as below.

Safe	<1.5
Moderate	1.5–3.0
Unsafe	>3.0

v).**Boron Content**

Boron, an essential micronutrient is required in very small quantity by the crops. It becomes toxic, if present beyond a particular level. In relation to boron toxicity, water quality ratings are as below.

Low hazard	<1 μg B/ml
Medium hazard	1-2 μg B/ml
High hazard	2-4 μg B/ml
Very high hazard	>4 μg B/ml

Each of the above parameters has bearing on the quality of irrigation water. However, each water source will have its specific suitability or hazardous nature depending upon the presence (and the degree) or absence of each of the constituents. Different chemical constituents interact with each other and cause a complex effect on soil properties and plant growth.

The waters with low SAR and low EC are widely suitable but when a value of any one of these parameters or both increases in its content, the waters become less and less suitable for irrigation purposes. The selection of crops for such situation becomes critical. Salt tolerant crops can be grown in such areas. Soil type is also an important consideration under such situations.

Upper permissible limits of EC, SAR, RSC and B are indicated below (Table 16) for soil having varying amounts of clay and for growing tolerant (T) and semi-tolerant (ST) crops. These limits are based on extensive trials conducted by Paliwal and Yadav (1976).

TABLE 24 Suitability of irrigation water for semi-tolerant and tolerant crops in different soil types

Textural category	Upper permissible limit							
	EC (dS/m)		SAR		RSC (me/litre)		B (μg/ml)	
	ST	T	ST	T	ST	T	ST	T
Above 30% Clay	1.5	2.0	10	15	2	3	2	3
20-30% Clay	4.0	6.0	15	20	3	4	2	3
10-20% Clay	6.0	8.0	20	25	4	5	2	3
Below 10% Clay	8.0	10.0	25	30	5	6	3	4

vi). **Trace elements**

Presence of trace elements or heavy metals cause reduction in crop growth, if their concentration increases beyond a certain level in irrigation waters and if such waters are used continuously. However, such elements are normally not a problem in common irrigation waters. They can be of concern when industrial effluent water is used for irrigation.

8.2 Methods of Water Sample Collection

A representative sample (500 ml) is collected in glass or polyethylene bottle which should be properly washed/rinsed with the same water which is being sampled. The floating debris or any other contaminant should be avoided while collecting the samples. After proper labeling, such as source of water, date of collection and the type of analysis required, the sample should be sent to the laboratory without undue delay.

Some of the anions like SO_4 and NO_3 may be quite low in irrigation waters. Hence, large volume of sample has to be first concentrated by evaporating to about 100 ml to obtain their detectable amounts.

8.3 Analytical Methods

1. pH

The pH is determined by taking about 50 ml of water sample in 100 ml clean beaker by using pH meter as described under chapter 3, soil analysis, item 6.

2. Electrical Conductivity (EC)

The conductivity meter cell is filled with water sample and the EC is determined as described under chapter 3, soil analysis, item 8a. The results are expressed as mS/cm.

3. Calcium And Magnesium

The usual method for determination of Ca+Mg is by versenate (EDTA) titration (Cheng and Bray 1951). The method for their estimation in soils is described in item 17 of the chapter on soil analysis. In case of soils, generally exchangeable calcium and magnesium are estimated. In plants total content in acid digest is determined. Once extracted, either as exchangeable, soluble or total, further estimation by EDTA method is same. The estimation of Ca and Mg can also be done by AAS which has been described under Item No.5 and 6 in Chapter 4 on Plant Analysis. Estimation of Ca + Mg in water by EDTA method is described as under:

Apparatus

- Porcelain dish
- Volumetric flasks
- Burette

Reagents

- Standard versenate solution (EDTA): An approximately 0.01N solution of ethylene diaminetetraacetic acid disodium salt (versenate) is prepared by dissolving 2.0 g in distilled water to which 0.05 g of magnesium chloride ($MgCl_2.6H_2$)) is added and diluted to one litre. This is to be standardized against 0.01N calcium chloride solution prepared by weighing 0.500 g AR grade $CaCO_3$ (oven dried) and dissolving in minimum excess of dilute HCl (AR) followed by making up the volume to one litre with distilled water.
- Ammonium chloride-ammonium hydroxide buffer (pH 10): 67.5 g pure ammonium chloride dissolved in 570 ml of concentrated ammonium hydroxide and made to one litre. pH adjusted to 10.
- Eriochrome black T indicator: 0.5 g of eriochrome black T and 4.5 g of hydroxylamine hydrochloride (AR) dissolved in 100 ml of 95% ethyl alcohol.

Procedure

1. Take 5 ml of the water sample in a porcelain dish (8 cm diameter).
2. Dilute to about 25 ml with distilled water.
3. Add 1 ml. of ammonium chloride-hydroxide buffer and 3 to 4 drops of eriochrome black T indicator.
4. Titrated with the standard versenate solution. The colour change is from wine red to bright blue or bluish green. At the end point no tinge of the red colour should remain.

Calculation

From the volume of 0.01N EDTA (standardized against 0.01N $CaCl_2$) solution required for titration, the concentration of Ca+Mg is directly obtained in me/litre as follows:

$$\text{Ca} + \text{Mg (me/litre)} = \frac{\text{ml versenate (EDTA) used x normality of EDTA x 1000}}{\text{ml aliquot taken}}$$

$$\text{Ca} + \text{Mg (g/litre)} = \frac{\text{Ca} + \text{Mg in me/litre x equivalent wt.}}{1000}$$

$$= \frac{\text{Ca} + \text{Mg in me/litre x 32.196}}{1000}$$

OR

$$\text{Ca} + \text{Mg(g/litre)} = \frac{\text{ml versenate(EDTA)used x normality of EDTA x 1000 x equivalent wt.}}{\text{ml aliquot taken x 1000}}$$

$$= \frac{\text{ml versenate (EDTA) used x normality of EDTA x 32.196}}{\text{ml aliquot taken}}$$

4. **Sodium**

Small amount of sodium is generally present even in the best quality of irrigation water. The concentration of sodium may be quite high in saline water with EC greater than 1 mS/cm and containing relatively less amount of Ca and Mg. Obviously, its estimation is of interest when the water sample tests saline (i.e. having EC above 1.0 mS/cm at 25°C). The determination of Na is carried out directly with the help of flame photometer using appropriate filters and standard curves prepared by taking known concentration of Na.

Apparatus

- Flamephotometer
- Volumetric flasks
- Beakers

Reagents

- **NaCl (AR grade)**

Procedure

1. Preparation of standard curve:

   - Take 2.5413 g of NaCl (AR), dissolve in water to make to the volume to 1 litre and this will give a solution of 1000 µg Na/ml. From this solution take 100 ml and dilute to 1 litre to obtain 100 µg Na/ml as stock solution.
   - For preparing working standards, take 5, 10, 15 and 20 ml of stock solution in 100 ml volumetric flask and make up the volume. It would give 5, 10, 15 and 20 µg Na/ml.
   - Feed the standards on the flamephotometer one by one to obtain a standard curve taking absorbance on Y-axis and respective concentrations of Na on X-axis.
2. Water samples are fed on the flamephotometer and absorbance is recorded for each sample.
3. Concentration of Na is observed against each absorbance which is in µg Na/ml.

Calculation

$$\text{Content of Na in mg/litre of water} = \frac{A \times 1000}{1000} = A$$

where,
A = absorbance reading (µg/ml) from the standard curve

Note:

- If a water sample is diluted for estimation, the quantity of sodium as observed on standard curve is multiplied by the dilution factor.
- If the water sample is concentrated before estimation, the quantity noted from the standard curve is divided by the concentration factor.
- Normally, no dilution and conc. is required.

5. **Carbonates and Bi-Carboantes (Richards, 1954)**

The estimation is based on simple acidimetric titration using different indicators which work in alkaline pH range (above 8.2) or in acidic pH range (below 6.0).

Apparatus

- Porcelain dish
- Burette

Reagents

- Phenolphthalein indicator: 0.25% solution in 60% ethyl alcohol
- Methyl orange indicator: 0.5% solution in 95% alcohol
- Standard sulphuric acid (0.01M).

Procedure

1. Take 5 ml of the water sample (containing not more than one milliequivalent of carbonate plus bicarbonate) in a porcelain dish.
2. Dilute with distilled water to about 25 ml.
3. A pink colour produced with a few (2 to 3) drops of phenolphthalein indicates presence of carbonate and it is titrated with 0.01M sulphuric acid until the colour just disappears (phenolphthalein end point) because of alkali carbonate having been converted to bicarbonate. This is called half neutralization stage. This burette reading (volume used) is designated as Y.
4. To the colourless solution from this titration (or to the original sample of water if there was no colour with phenolphthalein) add 1 to 2 drops of methyl orange indicator and continue titration with brisk stirring to the methyl orange end point (yellow) and the final reading (volume used) is designated as Z.

Calculation

$$\text{Carbonates (me/litre)}$$

$$= 2(\text{Volume of } H_2SO_4) \text{ x Molarity of } H_2SO_4 \text{ x } \frac{1000}{\text{ml of aliquot}}$$

$$= 2Y \text{ x } 0.01 \text{ x } \frac{1000}{5}$$

$$= 2Y \text{ x } 2 = 4Y$$

$$\text{Carbonates (g/litre)} = \frac{2(\text{Vol. of } H_2SO_4) \text{ x Molarity x } 1000 \, x \text{ Eq. wt. of } CO_3 (30)}{\text{ml of sample x } 1000}$$

$$= \frac{2Y \text{ x } 0.01 \text{ x } 30}{5} = 0.12Y$$

Note:
The volume of acid used for half-neutralization of carbonate is Y, hence for full neutralization it has been assumed as 2Y.

$$\text{Bicarbonates (me/litre)} = (Z - 2Y) \text{ x molarity of } H_2SO_4 \text{ x } \frac{1000}{\text{ml of aliquot}}$$

$$= \frac{(Z - 2Y) \text{ x } 0.01 \text{ x } 1000}{5}$$

$$= (Z - 2Y) \times 2$$

Where carbonate is absent: Z x 2

6. **Residual Sodium Carbonate (RSC)**

This is an important character for assessing the suitability of irrigation water in consideration of likely sodium hazard. It is calculated from the analysis data for carbonates, bicarbonates and calcium plus magnesium in the following manner:

$$RSC(me/litre) = (CO_3^{2-} + HCO_3^-) - (Ca^{2+} + Mg^{2+})$$

Note: all expressed in me/litre.

7. **Boron**

The method for Boron estimation is same as described for soils in chapter 3, item 18. The determination is carried out by azomethione-H colorimetric method. It can also be estimation on AAS. Suitable quantities of the sample may be taken depending upon the Boron content in the waters.

8. **Chlorides**

Mohr's titration method is most commonly used for chloride estimation. It depends upon the formation of a sparingly soluble brick-red silver chromate ($AgCrO_4$) precipitate at the end point when the sample is titrated against standard silver nitrate ($AgNO_3$) solution in the presence of potassium chromate (K_2CrO_4) as indicator. Initially the Cl ions are precipitated as AgCl and dark brick-red precipitate of Ag_2CrO_4 starts just after the precipitation of AgCl is over.

Apparatus

- Beakers/porcelain dish
- Burette

Reagents

- Potassium chromate (K_2CrO_4) indicator (5%) solution: Dissolve 5 g of K_2CrO_4 in about 75 ml distilled water and add drop by drop saturated solution of $AgNO_3$ until a slight permanent red precipitate is formed. Filter and dilute to 100 ml. With high purity analytical reagent, the indicator solution can be prepared directly.
- Standard silver nitrate solution (0.05M): Dissolve 8.494 g of silver nitrate ($AgNO_3$) in distilled water and make the volume to one litre. Standardize it against standard NaCl solution and keep in amber coloured bottle away from light.

Procedure

1. Take 5 ml of the sample in a 100 ml beaker or a porcelain dish and diluted to about 25 ml with distilled water.
2. Add 5-6 drops of K_2CrO_4 indicator (making it dark yellow), and titrate against the standard $AgNO_3$ solution with continuous stirring till the first brick-red tinge appears.
3. Run a blank to avoid error due to any impurity in chemicals.

Calculation

$$\text{Cl mg/litre of water} = X \times 1.775 \times \frac{1000}{\text{ml of sample}}$$

Where,
ml of water sample taken = 5
X = ml of 0.05M $AgNO_3$ consumed in titration
1.775 = factor representing mg of Cl in aliquot/sample as calculated below:
1 ml of 1M $AgNO_3$ = 1 me of Cl
1 ml of 0.05M $AgNO_3$ = 0.05 me of Cl = 35.5 x 0.05
= 1.775 mg of Cl (in aliquot).

8. Sulphate

While traces of sulphate occur universally in all types of waters, its content may be appreciably high in several saline waters showing EC greater than 1 dS/m at 25°C. Sulphate can be determined gravimetrically, colorimetrically, turbidimetrically or titrimetrically. Here, the turbidimetric method is described:

Sulphate content is determined by the extent of turbidity created by precipitated colloidal barium sulphate suspension. Barium chloride solid crystals are added to ensure fine and stable suspension of $BaSO_4$ at a pH of about 4.8. It also eliminates the interference from phosphate and silicate. Fine suspension of $BaSO_4$ is stabilized by gum acacia and the degree of turbidity measured by turbidity meter or estimated by spectrophotometrically at 440 nm.

Apparatus

- Spectrophotometer
- Beakers
- Volumetric flasks

Reagents

- Sodium acetate-acetic acid buffer: Dissolve 100 g of pure sodium acetate in 200 ml of distilled water. Add 31 ml of glacial acetic acid and make the volume to one litre. Adjust pH at 4.8.

- Gum acacia: Dissolve 2.5 g of gum acacia in one litre of distilled water. Keep overnight and filter.
- Barium chloride: Pure $BaCl_2$ crystals ground to pass through 0.5 mm sieve but retained on a 0.25 mm sieve.
- Potassium sulphate solution: To make a stock solution of 10 me S/litre, weigh 1.74 g of pure K_2SO_4 salt and dissolve in one litre water.

Procedure

1. Take 5 ml of the water sample (having <1 me S/litre) in 25 ml of volumetric flask. If the EC of water is >1 dS/m, dilute it with distilled water to bring EC below 1 dS/m.
2. Add 10 ml of sodium acetate-acetic acid buffer to maintain the pH around 4.8.
3. Add 1 ml of gum acacia and 1 g of $BaCl_2$ crystals and shake well.
4. Make the volume to 25 ml with distilled water.
5. Invert the flask several times and measure the turbidity with a spectrophotometer at 440 nm using blue filter.
6. Preparation of the standard curve: For 0, 1, 2, 3, 4 and 5 me S/litre, pipette 2.5, 5, 7.5, 10 and 12.5 ml from stock solution containing 10 me S/litre into 25 ml volumetric flasks. Then develop the turbidity and measure its intensity as in case of samples. Draw a curve showing sulphur concentration on x-axis and absorbance on y-axis.

Calculation

Calculate the S content of samples using the standard curve taking in to the dilution factor of 5 (5 ml made to 25 ml) expressed as me S/litre of water.

9. Nitrate Nitrogen (NO_3-N)

This method depends upon the reduction of nitrate to ammonia by adding Devarda's alloy and alkali. The nitrites (NO_2^-) if present in the sample are also reduced and determined along with NO_3^--N.

Apparatus

- Kjeldahl distillation assembly
- Electric muffle furnace
- Desiccator

Reagents

- Magnesium oxide (MgO): Heat the MgO at 65°C for 2 hours in an electric muffle furnace to remove traces of $MgCO_3$ which may be present. Cool in a desiccator over solid KCl and store in tightly stoppered bottle.

- Boric acid with mixed indicator: Weigh 20 g of boric acid and add approximately 900 ml of hot distilled water. Cool and add 20 ml of mixed indicator and make up the volume to 1 litre.
- Mixed indicator: Dissolve 0.066 g of methyl red and 0.099 g of bromocresol green in 100 ml of alcohol.
- Standard sulphuric acid (0.02M).
- Devarda's alloy: Mix Cu:Al:Zn in the ratio of 50:45:5 and grind to pass through 0.15 mm sieve.

Procedure

1. Take 50 ml of water sample in the distillation flask.
2. Add 0.5 g of MgO and 0.2 g of Devarda's alloy.
3. Put the heaters on and collect the NH_4 (NO_3 coverted in NH_4 by reducing agent – Devarda's alloy) into boric acid (20 ml) having mixed indicator into conical flask, which is connected with distillation apparatus.
4. Continue distillation to collect about 35-40 ml.
5. Remove the distillate first and then switch off the heating system.
6. Titrate the distillate against 0.02M H_2SO_4 till the pink colour appears.
7. Carry out a blank simultaneously.

Calculation

$$NO_3^- - N \text{ (mg/litre)} = \frac{(X - Y) \text{ x } 0.28}{50 \text{ (ml of sample)}} \text{ x } 1000 = X - Y \text{ x } 0.56$$

Where,
X = volume (ml) of 0.02M H_2SO_4 consumed in sample titration.
Y = volume (ml) of 0.02M H_2SO_4 consumed in blank titration.
0.28 = Factor (1 lit 1M H_2SO_4 = 14 g N. Therefore,

$$1 \text{ ml } 0.02\text{M } H_2SO_4 = \frac{14 \text{ x } 0.02 \text{ x } 1000}{1000} \text{ mg N} = 0.28 \text{ mg N}$$

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Annexure 1

List of equipment, provided under National Project, for setting up of Soil Testing Laboratory with an analyzing capacity of 10,000 samples per annum (For analyzing NPK, secondary and micronutrients in soil and 200 samples of irrigation waters)

Sl.	Items	Cost (Rs.in lakh)
1	Equipment*	18.00
2	Chemicals & glasswares	10.00
3.	Contingencies	6.00
4.	Standby Generator/Electricity source	6.00
5.	Assistance for outsourcing technical support	20.00
	Total	**60.00**

Note: *Subsidy is provided @ 50% of project cost limited to maximum of Rs.30 lakh as one time subsidy.*

* **Equipment-wise estimates approved**

S.No.	Name of Equipment	No	Cost (Rs. in lakh)
1	Atomic Absorption Spectrophotometer (AAS) #	1	10.00
2	Spectrophotometer #	1	1.00
3	Flame Photometer #	1	0.70
4	Conductivity Meter	2	0.30
5	pH Meter	2	0.30
6	Shaking Apparatus	2	0.30
7	Electronic Balance	1	1.00
8	Analytical Balance / Top Loading balance	2	0.70
9	Drying Oven	1	0.20
10	Computer with appropriate software	1	1.50
11	Table Top Centrifuge	1	0.25
12	Misc. laboratory articles	-	1.25
	Total		**18.00**

Note: # or Inductively Coupled Plasma Spectrometer (ICP) in lieu of equipment mentioned at Sl. No. 1, 2, and 3.

The specifications of the major equipment are given in **Annexure-6A.**

The details of the equipment, cost estimates and subsidy pattern to set up mobile soil testing lab are given in **Annexure – 1A.**

Annexure 1-A

List of equipment for setting up of Mobile Soil Testing Laboratory with an analyzing capacity of 5,000 samples per annum in the field and 5,000 samples when the van is attached with the stationary lab (for analyzing NPK, secondary and micronutrients in soil and 200 samples of irrigation waters)

Sl.	Items	Cost (Rs.in lakh)
1	Equipment	18.00
2	Chemicals & glasswares	1.50
3.	Contingencies	1.00
4.	Generator	1.00
5.	Cost of van	15.00
6.	Assistance for manpower	3.50
	Total	**40.00**

Note: *Subsidy is provided @ 75% of project cost limited to maximum of Rs.30 lakh as one time subsidy.*

Details of Equipment approved

S.No.	Name of Equipment	No	Cost (Rs. in lakh)
1	Atomic Absorption Spectrophotometer (AAS)	1	10.00
2	Spectrophotometer	1	1.00
3	Flame Photometer	1	0.70
4	Conductivity Meter	2	0.30
5	pH Meter	2	0.30
6	Shaking Apparatus	2	0.30
7	Electronic Balance	1	1.00
8	Analytical Balance	2	0.70
9	Drying Oven	1	0.20
10	Computer with appropriate software	1	1.50
11	Table Top Centrifuge	1	0.25
12	GPS System with mobile phones	1	0.25
12	Misc. laboratory articles	-	1.50
	Total		**18.00**

Annexure-2

Staff Requirement for a Central / Nodal Soil Testing Laboratory having 10,000 soil and 200 water samples annual analysing capacity and with the responsibility of preparing soil fertility maps and arranging training for I/C Soil Testing Labs

S.No.	Name of Post	Number	Qualifications
1	Senior Soil Scientist/ Joint Director(Agri.)	01	M.Sc(Soil.Sci. / Agri.Chemistry) Or B.Sc.Ag. with 08 years experience in soil testing / soil survey /fertilizer testing
2	Agronomist /Dy.Director(Agronomy)	01	M.Sc(Agronomy) with experience as per Dy. Director in the State
3	Asstt. Soil Chemist	02	M.Sc(Soil.Sci. / Agri.Chemistry) Or B.Sc.Ag./Chemistry with 05 years experience in soil testing / soil survey /fertilizer testing
4	Analytical Asstt.	02	B.Sc.Ag./ Chemistry with 03 years experience in soil testing / soil survey /fertilizer testing
5	Lab Attendant	03	High School Science
6	LDC /Steno	01	High School, Typing /stenographer experience
7	Computer Operator	01	Relevant experience
8	Peon	01	As per Govt. Rules
	Total	**12**	

Staff Requirement of a Soil Testing Laboratory having 10,000 soil and 200 water samples annual analysing capacity

S.No	Name of Post	Number	Qualifications
1	Asstt. Soil Chemist	01	M.Sc(Soil.Sci. / Agri.Chemistry) Or B.Sc.Ag./Chemistry with 05 years experience in soil testing / soil survey /fertilizer testing
2	Analytical Asstt.	03	B.Sc.Ag./ Chemistry with 03 years experience in soil testing / soil survey /fertilizer testing
3	Lab Attendant	03	High School Science
4	LDC	01	High School, Typing experience
	Total	**08**	

Annexure-3

State wise Number of Soil Testing Laboratories, Annual Analyzing Capacity and its Utilization During the year 2008-09

SN	Name of the State	No. of Dist	No. of Soil Testing Laboratories							Annual Anlyzing Capacity In'00000'	Sample Analyzed In'00000'	Cap. Utili. %
			State Govt.		Fert. Industry		Total					
			Static	Mobile	Static	Mobile	Static	Mobile	Total			
1	Andhra Pr.	23	80	4	2	0	82	4	86	4.38	4.55	103.9
2	Karnataka	29	20	3	1	1	21	4	25	2.64	1.60	60.6
3	Kerala	14	14	9	1	0	15	9	24	3.67	2.36	64.3
4	Tamil Nadu	30	19	16	1	1	20	17	37	8.34	7.20	86.3
5	Pondicherry	4	2	0	0	0	2	0	2	0.04	0.06	150.0
6	A&N Island	3	1	1	0	0	1	1	2	0.12	0.07	58.3
7	Lakshdweep	1	NA	NA	NA	NA	NA	NA	NA	NA	NA	-
	TOTAL	104	**136**	**33**	**5**	**2**	**141**	**35**	**176**	**19.19**	**15.84**	**82.5**
8	Gujarat	25	22	1	3	1	25	2	27	2.40	3.12	130.0
9	M.P.	48	27	6	0	3	27	9	36	3.56	2.00	56.2
10.	Maharashtra	35	29	0	6	4	35	4	39	2.25	2.62	116.4
11	Rajasthan	32	21	12	1	0	22	12	34	3.75	3.29	87.7
12.	Chhattisgarh	16	5	4	0	0	5	4	9	0.65	0.41	63.1
13.	Goa	02	1	1	0	0	1	1	2	0.25	0.18	72.0
	TOTAL	**158**	**105**	**24**	**10**	**8**	**115**	**32**	**147**	**12.86**	**11.62**	**90.4**
14.	Haryana	19	30	0	0	0	30	0	30	3.08	2.06	66.9
15.	Punjab	17	54	3	0	1	54	4	58	5.56	3.82	68.7
16.	H.P	12	11	2	0	0	11	2	13	1.25	1.22	97.6

17.	Uttar Prades	70	73	18	4	4	77	22	99	21.39	21.61	101.0
18.	(J&K) - Jammu	14	6	0	0	0	6	0	6	0.45	0.33	72.3
	Shrinagar		6	4	0	0	6	4	10	0.29	0.07	24.1
	J & K Total		12	4	0	0	12	4	16	0.74	0.40	54.1
19.	Uttaranchal	13	13	2	0	0	13	2	15	0.85	0.61	71.8
20.	Delhi	9	1	0	0	0	1	0	1	0.01	0.01	100.0
	TOTAL	**154**	**194**	**29**	**4**	**5**	**198**	**34**	**232**	**32.88**	**29.73**	**90.4**
21.	Bihar	38	39	0	0	0	39	0	39	2.00	1.03	51.5
22.	Orissa	30	11	0	0	0	11	0	11	1.20	1.15	95.8
23.	West Bengal	19	10	8	0	0	10	8	18	1.26	0.41	32.5
24.	Jharkhand	22	7	0	0	0	7	0	7	0.39	0.12	30.8
	TOTAL	**109**	**67**	**8**	**0**	**0**	**67**	**8**	**75**	**4.85**	**2.71**	**55.9**
25.	Assam	23	8	4	0	0	8	4	12	1.06	0.59	55.6
26.	Tripura	04	2	4	0	0	2	4	6	0.21	0.12	57.1
27.	Manipur	09	3	1	0	0	3	1	4	0.20	0.01	0.1
28.	Nagaland	08	3	0	0	0	3	0	3	0.45	0.12	26.7
29.	Arunachal Pr	16	1	1	0	0	1	1	2	0.05	0.04	80.0
30.	Meghalaya	07	1	1	0	0	1	1	2	0.10	0.10	100.0
31.	Sikkim	04	1	0	0	0	1	0	1	0.08	0.08	100.0
32.	Mizoram	08	1	0	0	0	1	0	1	0.08	0.07	87.5
33.	Daman & Diu	02	-	-	-	-	-	-	-	-	-	-
34.	Dadra Nagar	02	-	-	-	-	-	-	-	-	-	-
	TOTAL	**83**	**20**	**11**	**0**	**0**	**20**	**11**	**31**	**2.23**	**1.13**	**50.7**
GRAND TOTAL		**608**	**522**	**105**	**19**	**15**	**541**	**120**	**661**	**72.01**	**60.83**	**84.5**

Annexure -4

Plant tissue sampling guideline for different crops

Crop (Reference)	**Index Tissue**	**Growth stage/time**
Food Crops		
Rice	3rd leaf from apex	Tillering
Wheat	Flag leaf	Before head emergence
Sorghum	3rd leaf below inflorescence	Bloom
Maize	Ear leaf	Before tasseling
Barley	Flag leaf	At head emergence
Pulses	Recently matured leaf	Bloom initiation
Potato	Most recent fully developed leaf	Half grown
Oil crops		
Groundnut	Recently matured leaflets	Maximum tillering
Sunflower	Youngest matured leaf blade	Initiation of flowering
Mustard	Recently matured leaf	Bloom initiation
Soybean	3rd leaf from top	2 months after planting
Fibre crops		
Cotton	Petiole, 4th leaf from apex	Initiation of flowering
Jute	Recently mature leaf	60 days age
Other field crops		
Sugarcane	3rd leaf from top	3-5 months after planting
Sugarbeet	Petiole of youngest matured leaf	50-80 days old
Tobacco	3rd leaf from top	45-60 days old
Fruit crops		
Apple	Leaves from middle of terminal shoot growth	8-12 weeks after full bloom 2 to 4 weeks after formation of terminal buds in bearing tree
Apricot	Fully expanded leaves midshoot current growth	Early June to mid July
Blackberry	Latest matured leaf from non-tipped canes	4-6 weeks after peak bloom
Cherry	Fully expanded leaves, midshoot current growth	July-August
Peach	Midshoot leaves. Fruiting or non-fruiting spurs	Mid-summer leaves. Fruiting
Banana	Petiole of 3rd open leaf from apex	Bud differentiation 4 months after planting
Cocoa	3rd leaf from apex	Bloom initiation
Cashew	4th leaf from tip of matured branches	At beginning of flowering

Citrus fruits	3 to 5 months old leaves from new flush. 1st leaf of the shoot	June
Guava	3rd pair of recently matured leaves	Bloom stage (Aug. to Dec.)
Mango	Leaves + Petiole	4-7 months old leaves from middle of shoot
Papaya	6th petiole from apex	6 months after planting
Pineapple	Middle one third portion of white basal portion of 4th leaf from apex	4-6 months
Plantation Crops		
Coconut	Pinnal leaf from each side of 4th leaf	
Coffee	3rd or 4th pair of leaves from apex of lateral shoots	Bloom
Oil Palm	Middle 1/3rd minus midrib of 3 upper and 3 lower leaflets from 17 fronds of mature trees and 3 fronds of young trees.	
Tea	Third leaf from tip of young shoots	
Clove	10th to 12th leaves from tip of non fruiting shoot	End of blooming period
Source : Bhargava and Raghupati – Methods of Analysis Edited by Tandon (2005)		

Plant analysis is the subject of rather extensive research programmes among plant nutritionists. A great deal remains to be discovered about this diagnostic tool and research is constantly uncovering new facts and establishing standards. It can be a valuable addition to available diagnostic tools. At present, the plant analysis is not done in the soil testing labs. However, it is an important parameter to study the nutrient need of growing crops and may become a facility in future in the soil testing labs also.

Tissue testing: Tissue testing is the determination of the amount of a plant nutrient in the sap of the plant, a semi-quantitative measurement of the unassimilated, soluble content. A large amount of an un assimilated nutrient in the plant sap indicates that the plant is getting enough of the nutrient being tested for good growth. If the amount is low, there is a good chance that the nutrient is either deficient in the soil or is not being absorbed by the plant because of lack of soil moisture or some other factors. Tissue tests can be run easily and rapidly in the field. Green plant tissue can be tested for several nutrients, NO_3-N, P, K and sometimes Mg, Mn and Fe. However, it takes a lot of practice and experience to interpret

the results, especially those for the micronutrients. Tissue tests are used to identify one nutrient (N, P or K) that may be limiting crop yields. If one nutrient is very low, others might accumulate in the sap because plant growth has been restricted, resulting in an improper interpretation. If the crop grows vigorously after the deficiency has been corrected, one might find that other nutrients are not present in required amounts produce high yields. What is identified, or tested for, is the most limiting nutrient at a particular growth stage. The on-the-spot tissue tests can be very helpful. Right in the field, N deficiencies can be detected and corrective measures suggested. As with total analysis of plants, it pays to compare healthy plants with poor ones wherever possible. In India, tissue testing is used in a limited way for giving fertilizer recommendations for plantation crops in the southern states. However, in the western countries, kits containing instructions and supplies for running tissue tests are available. When properly used, tissue tests work well with soil tests and plant analysis as another good diagnostic tool.

Annexure-5

Format of registers for maintaining record of equipment, glasswares, chemicals and miscellaneous items in a Laboratory

Register for Equipment

Item	Description	Qty	Reference	Model	Price/ Unit (US$)	Total Price (US$)
1	Spectr ophoto meter	1	BDH331/10 42/04	6035	3620	3620
2	Flame photo meter					
3						
4						

Register for Glasswares

Item	Description	Qty	Referen ce	Model	Price/ unit (US$)	Total Price (US$)
1	Beaker 5 ml	30	BDH/209/031 0/01	Pyrex	5	150
2	Flask					
3						
4						

Register for chemicals

Item	Description	Qty	Reference	Model	Price/ piece (US$)	Total / Price (US$)
1	Hydrochloric Acid	2 litres	BDH-10125-5Y	AR	16	3 2
2	Sulphuri c Acid					
3						
4						

Register for Miscellaneous items

Item	Description	Qty	Reference	Model	Price/ unit (US$)	Total Price (US$)
1	Funnel stand	2 litres	ABC	Wooden	5	10
2						
3						

Annexure-6

Equipment, Chemicals and Glasswares required for a Laboratory having a capacity to analyse 10,000 Soil and 200 Water samples annually

Equipments

S.No.	Name/Specification/Description	Nos.
1.	Analytical / semi-micro balance Capacity = 300 g Resolution = 0.1 mg Pan size = 100 mm	2 (1 each type)
2.	Two pan balance Capacity = 500 g Resolution = 0.2%	1
3.	pH meter: Range 0-14 pH with accuracy of ±0.05 pH, complete with combination electrode, mains operated, to work on 220V, 50 CY.	2
4.	Conductivity Bridge: Single range 0-15 mS/cm directly calibrated with temperature compensation and cell constant adjuster, complete with pipette type conductivity cell having platinum electrodes duly coated with platinum black and having a cell constant of 1.00±0.01%; mains operated with electronic "Eye" null indicator, to work on 220V 50CY.	1
5.	Photoelectric colorimeter: Having dual barrier type matched photocells with sensitive galvanometer for null adjustment and logarithmically calibrated potentiometer to read directly optical density on dial, complete with optical glass filters for maximum transmission at 420, 540 and 660 nm wavelengths; mains operated to work on 220V 50 CY. Or Spectronic-20 Spectrophotometer	1
6(i).	Shaking machine: Reciprocating type, variable speed of 70 to 300 strokes per minute, with box type platform carrier (size 79 cm L x 43 cm W x 8 cm H) fitted with heavy duty electric motor for continuous operation and built-in 0-60 minutes timeswitch; to work on 220 V, 50 CY.	1
6(ii).	Water Bath Shaker with 50 cm x 38 cm water bath size mounted on the shaking machine as 6(i).	1

7.	Centrifuge, clinical type with head to take 12 tubes of 15 ml capacity, complete with metal shields, rubber cushions and 15 ml centrifuge polythene tubes; to work on 220V, 50CY.	1
8.	Voltage stabilizers (Constant voltage transformers): Input 170 to 250V, AC output 220V Capacity: 1, 2, 5 KW	1 each
9.	Voltage stabilizers (Constant voltage transformers): Input 170 to 250V AC, Output 220V$\pm$5V	2
10.	Pressure Vacuum Pump: To deliver 1.5 cuft. (0.04 m^3 /minute) air at a maximum pressure of 15lb per sq.inch and create vacuum of 28^0 mercury column, complete with pressure gauge and ballast, to work on 220V, 50 CY.	2
11.	Automatic pipetting machine: Brewer type, adjustable volume and frequency of cycling, to deliver upto 50 ml solution; syringe, plunger cylinder, valves and intake and delivery tubes made of neutral hard glass or resistant plastic material; to work on 220V 50CY.	2
12.	Demineralizer Plant: For obtaining deionised water, regenerating type, separate or mixed bed resin columns treated water to have pH 6.8 to 7.0, portable model, capacity – 75 litres of deionised silica free water per hour.	1
13.	Oven: Laboratory model, made of stainless steel inside and outside, maximum temperature 180^0C, thermostatically controlled, $\pm 1.0^0$C accuracy, inside chamber – 30 cm x 30 cm x30 cm.	1
14.	Trolleys: (Push carts) made of tubular frame mounted on rubber casters, with mild steel top size 60 cm x 75 cm and height 90 cm.	4
15.	Flask stand: To hold eleven 100 ml conical flasks, with adjustable base and collar – total length of stand 75 cm, width 8 cm, overall height 12 cm, distance between flasks 6.5 cm to fit multiple dispensing equipment (wood).	10

16.	Funnel Stand: To hold eleven 5 to 7 cm diameter glass funnels to correspond to flask stand	10
17(i).	Beaker Stand: To hold eleven 50 ml beakers – to fit multiple dispensing equipment (wood).	10
17(ii).	Burette Stand with Clamps.	4
18.	Test Tube Stand: To hold eleven test tubes of 25 mm x 150mm, distance between test tubes 6.5 cm, overall height 16 cm (wood).	10
19.	Multiple dispensing equipment: With eleven units each 50 ml, to fit 75 cm tray, distance between flasks from center to center 6.5 cm, width of tray 8 cm, distance from center of outer flasks and end of tray 4.5 cm, overall height of tray 12 cm.	2
20.	Multiple dispensing equipment: With eleven units each 25 ml, to fit 75 cm tray, distance between flasks from center to center 6.5 cm, width of tray 8 cm, distance from centre of outer flasks and end of tray 4.5 cm, overall height of tray 12 cm.	2
21.	Multiple dispensing equipment: With eleven units each 20 ml, to fit 75 cm tray, distance between flasks from center to center 6.5 cm, width of tray 8 cm, distance from center of outer flasks and end of tray 4.5 cm overall, height of tray 12 cm.	2
22.	Washing assembly: For washing glassware used with multiple dispensing equipment, pipe and jet made of PVC, combined unit for both tap water washing and distilled water rinsing, complete with brackets for mounting in sink.	1
23.	Scoops: For soil sampling, made of brass with wooden handle. A set of five scoops to measure 1 g, 2.5 g, 5.0 g, 10 g and 12.5 g of soil.	2
24.	Mortar and pestle: Heavy cast iron mortar or porcelain mortar glazed outside only, size 160 mm dia with wooden rubber tipped pestle.	2
25(i).	Sieves: 20 cm diameter, 5 cm height having 2 mm round holes, preferably made of stainless steel, complete with coverlid and	3

	receiver pan.	
25(ii).	1 mm with similar specifications	1
26.	Trays: For drying soil samples, made of 18 gauge aluminium sheet, 22 cm x 22 cm x 8 cm.	100
27.	Hot Plate: Rectangular 45 cm x 60 cm with three position: Low, Medium and High, heavy duty rotary switch, 1 KW to work on 220V, 50CY.	2
28.	Kjeldahl assembly, both as digestion and distillation set having a capacity to hold 6 round bottom flask of 300 ml capacity each complete with condensor and connecting tubes. Heating capacity of 500 watt of each heater.	1
29.	Fume hood – digestion chamber to hold Kjeldahl assembly of 6 sets.	1
30.	Atomic Absorption Spectrophotometer (AAS) Double beam with spare hollow cathode lamps for zinc, copper, manganese and iron.	1
31.	Muffle furnace with temperature 1 000^{0}C $\pm$ 5^0, size of furnace 10 cm x 15 cm.	1

Chemicals

S.No.	Chemical	Estimated annual requirement
1.	Acetic Acid (glacial)	10 litres
2.	Activated Charcoal – Darco G-60	10 kg.
3.	Ammonium Ferrous Sulphate (CP)	100 kg
4.	Ammonia Solution (CP)	10 litres
5.	Ammonium Acetate (CP)	40 kg
6.	Ammonium Bi-carbamate (AR)	1 kg
7.	Ammonium Metavanadate	1 kg
8.	Ammonium Molybdate (AR)	3 kg

9.	Ammonium Vanadate	500 g
10.	Ascorbic acid	100 g
11.	Barium Chloride - CP and AR	1 kg each
12.	Boric Acid (CP)	10 kg
13.	Bromocresol Green	500 g
14.	Buffer Solutions (pH 4.0, 7.0, 9.2)	2 bottles each
15.	Calcium Acetate (CP)	500 g
16.	Calcium Carbonate (CP)	0.5 kg
17.	Calcium Chloride (CP)	0.5 kg
18.	Calcium Sulphate (CP)	500 g
19.	Copper Sulphate (AR)	500 g
20.	Copper Sulphate (CP)	1 kg
21.	Di-phenyl amine indicator	100 g
22.	Di-ethylene Triamine Pentaacetic Acid	100 g
23.	Digestion mixture containing Potassium Sulphate and Copper Sulphate	10 kg
24.	Di-phenyl Amine Indicator	100 g
25.	DTPA (AR)	2 kg
26.	EDTA – disodium salt	1 kg
27.	Eriochrome Black – T	50 g
28.	Ethyl Alcohol	5 litres
29.	Ferrous Sulphate (AR)	500 g
30.	Ferrous Sulphate (CP)	500 g
31.	Filter paper, Whatman No.1 (460 mm x 570 mm)	2500 sheets
32.	Gum Acacia	0.5 kg
33.	Hydrochloric Acid (CP)	100 litres
34.	Hydrogen Peroxide	5 litres.
35.	Hydroxyl amine	1 kg

	hydrochloride	
36.	Magnesium Chloride (AR)	500 g
37.	Manganese Sulphate (AR)	500 g
38.	Manganese Chloride (CP)	1 kg
39.	Methyl Orange	25 g
40.	Methyl Red	25 g
41.	Methylene Blue	50 g
42.	Mono-calcium Phosphate (AR)	500 g
43.	Mureoxide Indicator	100 g
44.	Nitric Acid (CP)	10 Lit.
45.	Nitric acid AR	10 litres
46.	Nitrophenol (CP)	500 g
47.	Para-nitrophenol (CP)	1 kg
48.	Perchloric Acid (CP)	10 litres
49.	pH Indicator Papers (full pH range)	10 books
50.	Phenolphthalein indicator	100 g
51.	Phosphoric Acid (LR)	5 litres
52.	Potassium Hydrogen Phosphate (KH_2PO_4) (AR)	500 g
53.	Potassium Chloride (AR)	2 kg
54.	Potassium Chromate (CP)	500 g
55.	Potassium Dichromate (AR)	5 kg
56.	Potassium Dihydrogen Orthophosphate	500 g
57.	Potassium Hydrogen Pthalate (AR)	500 g
58.	Potassium Permanganate (CP)	2 kg
59.	Potassium Sulphate (AR)	1 kg
60.	Salicylic Acid (CP)	1 kg

61.	Silver Sulphate	1 kg
62.	Sodium bi-carbonate (LR)	30 kg
63.	Sodium Carbonate (AR)	500 g
64.	Sodium Carbonate (CP)	1 kg.
65.	Sodium Cyanide	100 g
66.	Sodium diethyl dithiocarbamate	500 g
67.	Sodium Fluoride (CP)	500 g
68.	Sodium Hydroxide (CP)	50 kg
69.	Sodium Thiosulphate (CP)	500 g
70.	Stannous Chloride (AR)	500 g
71.	Sucrose (AR)	500 g
72.	Sucrose (CP)	1 kg
73.	Sulphuric Acid (conc/CP)	500 litres
74.	Toluene	500 ml
75.	Tri-ethanol Amine Granule(CP)	500 g
76.	Tri-ethanol Amine	500 ml
77.	Universal Indicator	100 ml
78.	Whatman No.42; 110 mm	2 packets
79.	Whatman No.44; 110 mm	2 packets
80.	Zinc Sulphate (AR)	500 g

Glassware

S.No.	Item	Size/Specification	Quantity (Nos.)
1.	Bottle (Polyethylene)	20 litres.	5
2.	Bottle (Polyethylene)	10 litres	5
3.	Bottle (Glass) for reagents with glass stoppers	Glass stoppered 125 ml 250 ml 500 ml 1 000 ml 2 000 ml	 10 10 20 5 5

4.	Bottle (Glass), Amber	250 ml 500 ml	5 5
5.	Bottle (Polyethylene) –Wash bottle	250 ml 500 ml	6 6
6. (i)	Burettes fitted with screw thread stopcocks Graduation Interval (ml) 0.05 0.05 0.1 0.1	 10 ml 25 ml 10 ml 25 ml	 1 1 1 1
(ii)	Burette (automatic) (Mounted on Reservoir) Graduation interval (ml) 0.11 0.1	 25 ml 50 ml	 2 2
7.	Cylinder (Glass) graduated with an interval of: 0.5 ml 1 ml 2 ml 2 ml 5 ml	 10 ml 25 ml 50 ml 100 ml 500 ml	 2 2 2 2 2
8.	Crucible (Silica)	30 ml	10
9.	Desiccator with Appx. I.D. of ground flange as 200 mm		2
10.	Dishes, evaporating flat bottom with pour out, having outer diameter as 150 mm and height as 80 mm		20
11.	Water distilling unit, mounted with borosilicate condenser, having a capacity (output) to distill 2.5 litre/hour		2
12.	Flask		

	distilling/kjeldahl, round bottom, long neck Capacity : O.D. x height (mm) 100 ml 64 x 210 250 ml 85 x 226	 12 12
13.	Flask (Conical) (i) 100 ml cap., 64x105 mm (O.D. x height) with approx. neck O.D. as 25 mm. (ii) 250 ml cap., 85x140 mm (O.D. x height) with app. neck O.D. as 34 mm. (iii) 500 ml cap., 104x180 mm (O.D. x height) with app. neck O.D. as 34 mm. (iv) 1000 ml cap., 131x225 mm (O.D.x height) with app. neck O.D. as 34 mm.	 50 50 10 10
14.	Flask (Volumetric) Capacity (ml) Tolerance (± ml) 25 0.04 50 0.06 100 0.10 250 0.15 500 0.25 1000 0.40	 50 50 100 25 25 10
15.	Funnel, Plain, 60° angle Diameter 50 mm 65 mm 75 mm 100 mm	 20 20 20 20

16.	Pipettes (Measuring) Capacity (ml) Graduation Interval (ml) Tolerance (± ml) 1.0 0.1 0.006 2.0 0.1 0.01 5.0 0.1 0.05 10.0 0.1 0.05 25.0 0.2 0.1 50.0 0.5 0.1	 2 2 2 5 5 2
17.	Porcelain Dish - 100, 150 ml	6 each
18.	Test Tube App. O.D. x Height (mm) 12 x 10 15 x 125 18 x 150	 60 60 60
19.	Watch Glass App. Diameter (mm) 100 120	 60 60
20.	Rubber Stopper 15, 18, 20, 25, 30 mm diameter	12 each
21.	Spatula (stainless) with wooden handle, blade length 100 mm	12
22.	Wire gauge with asbestos center 150 x 15 0mm	20

Note: 20% glassware may need annual replacement due to breakage or changed requirement.
O.D. = outer diameter
I.D. = internal diameter

- When Boron is required to be estimated, the boron free glass wares may be used. Commonly required glass wares include beakers, flasks, pipettes, funnels and water distilling sets. Generally, marketed glass wares are made of borosilicate which contains traces of boron, hence not suitable for boron estimation.