5. SOIL CHEMICAL ANALYSIS

The **18 essential nutrients** for plants are classified into four groups (Brady and Weil, 1999):

1. Major non-mineral macronutrients: these are 90 - 95% of dry plant weight, and are supplied to the plant by water absorption and photosynthesis, i.e., C, H, O;
2. Primary macronutrients, i.e., N, P, K;
3. Secondary macronutrients, i.e., Ca, Mg, S; and
4. Micronutrients, i.e., B, Cl, Co, Cu, Fe, Mn, Mo, Ni, Zn.

Three major factors contributing to plant nutrition are:

1. The amount of nutrients in the soil;
2. The soil's ability to supply the nutrients to plants; and
3. Environmental factors that affect nutrient availability and their absorption.

Measurements, which involve characterization of the *soil solution and its constituents* and *of the composition of the inorganic and organic phases in soil,* are broadly termed chemical. This encompasses all nutrient elements and soil components which directly or indirectly influence such elements or components. **This section thus constitutes the core of this manual.**

The chemical procedures presented here are extensive, though by no means exhaustive. For any one element, numerous procedures or variations of procedures can be found in the literature (Walsh and Beaton, 1973; Page, 1982; Westerman, 1990). We have endeavored to select procedures which, in our experience, are appropriate for soils of the CWANA region, i.e., where a good relationship exists between the test value and crop growth. Where alternative methods are appropriate, we have presented the salient features of these methods. A bibliography of soil testing information is provided for users who may need to expand or modify their range of soil testing procedures.

We have initially presented analyses which are routinely done to characterize a soil sample or soil type in terms of background information, i.e., pH, salinity, calcium carbonate, organic matter, cation exchange capacity, and gypsum. With regard to nitrogen, the dominant fertility factor in the CWANA region soils, we have dealt with the most convenient methods for measuring different forms or fractions of N in soils. This is subsequently followed by procedures for P, soluble and exchangeable cations, soluble anions, and micronutrients. Where appropriate, we have given guidelines in the appendix **for interpreting the data** produced with the **analytical procedures** listed.

5.1 pH

The pH is defined as the negative log of the hydrogen ion activity. Since pH is logarithmic, the H-ion concentration in solution increases ten times when its pH is lowered by one unit. The pH range normally found in soils varies from 3 to 9. Various categories of soil pH may be arbitrarily described as follows: *strongly acid* (pH < 5.0), *moderately* to *slightly acid* (5.0 - 6.5), *neutral* (6.5 - 7.5), *moderately alkaline* (7.5 - 8.5), and *strongly alkaline* (> 8.5).

Significance of pH lies in its influence on availability of soil nutrients, solubility of toxic nutrient elements in the soil, physical breakdown of root cells, cation exchange capacity in soils whose colloids (clay/humus) are pH-dependent, and on biological activity. At high pH values, availability of phosphorus (P) and most micronutrients, except boron (B) and molybdenum (Mo), tends to decrease.

Acid soils are rare in semi-arid dryland areas of the world; they tend to occur in *temperate and tropical areas* where rainfall is substantial; conversely, soils of drier areas are generally alkaline, i.e., above pH 7.0, as a result of the presence of calcium carbonate ($CaCO_3$); they visibly effervesce (fizz) when 10% hydrochloric acid is added dropwise to the soil. Most soils in the Central and West Asia and North Africa region have pH values of 8.0 - 8.5. Calcareous soils with gypsum have somewhat lower pH values, while those with excess sodium (Na) have values over 8.5 (sodic soils).

Thus, soil pH is one of the most common measurements in soil laboratories. It reflects whether a soil is *acid, neutral, basic or alkaline*. Procedure for determining soil pH in a 1:1 (soil: water) suspension (McKeague, 1978; McLean, 1982) is:

Apparatus

pH meter with Combined Electrode.
Glass rod.
Glass beaker.

Reagents

A. Deionized water.
B. pH 7.0 buffer solution.
C. pH 4.0 buffer solution.

Procedure

1. Weigh 50 g air-dry soil (< 2-mm) into a 100-mL glass beaker.
2. Add 50 mL DI water using a graduated cylinder or 50-mL volumetric flask
3. Mix well with a glass rod, and allow to stand for 30 minutes.
4. Stir suspension every 10 minutes during this period.
5. After 1 hour, stir the suspension.
6. Put the Combined Electrode in suspension (about 3-cm deep). Take the reading after 30 seconds.
7. Remove the Combined Electrode from the suspension, and rinse thoroughly with DI water in a separate beaker, and carefully dry excess water with a tissue.

Note

1. Make sure that the combined electrode contains *saturated* KCl solution and some solid KCl.
2. Calibrate the pH meter using at least two buffer solutions of different pH values, usually 4.0 and 7.0. *First,* measure the temperature of the solution and adjust the *"temperature"* knob. *Second,* dip the combined electrode in pH 7.0 buffer solution, check for actual pH at measured temperature, and adjust with the *"buffer"* knob. Then, dip the combined electrode in the pH 4.0 buffer solution and adjust with *"sensitivity"* knob. Repeat until pH meter gives correct reading of both buffer solutions.
3. At ICARDA, pH is measured in a 1:1 (soil: water) suspension. For special purposes, pH can be measured in a saturated soil paste, or in more dilute suspensions. In some laboratories, pH is measured in a suspension of soil and 1 *N* KCl or 0.01 *M* $CaCl_2$. The main advantage of the measurement of soil pH in salt solution is the tendency to eliminate interference from suspension effects and from variable salt contents, such as fertilizer residues. However, this is hardly needed in alkaline-calcareous soils of CWANA.
4. Air-dry soils may be stored several months in closed containers without affecting the pH measurement.
5. If the pH meter and combined electrodes are not to be used for extended periods of time, the instructions for storage published by the instrument manufacturer should be followed.
6. For soil samples very high in organic matter, use a 1:2 or 1:5 (soil: water) ratio.

5.2 Electrical Conductivity

Soil salinity refers to the concentration of soluble inorganic salts in the soil. It is normally measured by extracting the soil sample with water (1:1 or 1:5 soil: water ratio, w/v) or in an saturated paste extract. However, soil: solution ratios of a 1:1 or wider ratio are more convenient where the soil sample is limited. Such extracts are rapid and salinity is measured by electrical conductivity (EC) using a conductivity bridge. *The total salt content of a soil can be estimated from this measurement.* A more precise method involves evaporation of the aqueous extract and weighing the residue.

Salinity is an important laboratory measurement since it reflects the extent to which the soil is suitable for growing crops. On the basis of a saturation extract, values of 0 to 2 dS/m (or mmhos/cm) are safe for all crops; yields of very sensitive crops are affected between 2 to 4 dS/m; many crops are affected between 4 and 8 dS/m; while only tolerant crops grow well above that level (Richards, 1954).

While salinity is largely a concern in irrigated areas of the CWANA region and in areas with saline soils, it is not so important in rainfed agriculture. However, with increasing use of irrigation, there will be greater emphasis on EC measurement in the future. The methodology of EC measurement is given in USDA Handbook 60 (Richards, 1954).

Apparatus

Vacuum filtration system.
Conductivity bridge.

Procedure

1. Prepare a 1:1 (soil: water) suspension, as for pH determination.
2. Filter the suspension using suction. *First,* put a round Whatman No. 42 filter paper in the Buchner funnel. *Second,* moisten the filter paper with DI water and make sure that it is tightly attached to the bottom of the funnel and that all holes are covered.
3. Start the vacuum pump.
4. Open the suction, and add suspension to Buchner funnel.
5. Continue filtration until the soil on the Buchner funnel starts cracking.
6. If the filtrate is not clear, the procedure must be repeated.
7. Transfer the clear filtrate into a 50-mL bottle, immerse the Conductivity Cell

in the solution, and take the reading.

8. Remove the conductivity cell from the filtration, rinse thoroughly with DI water, and carefully dry excess water with a tissue.

Note

1. Readings are recorded in milli-mhos per centimeter (mmhos/cm) or deci-Siemens per meter (dS/m). The use of the unit deci-Siemens is preferred over the unit milli-mhos. Both units are equal, that is, **1 dS/m = 1 mmho/cm.**
2. Reading are usually taken and reported at a standard temperature of 25°C.
3. Check accuracy of the EC meter using a **0.01 *N* KCl** solution, which should give a reading of **1.413 dS/m at 25°C.**

5.3 Calcium Carbonate

Inorganic carbonate, either as calcium (calcite) or magnesium (dolomite) carbonate or mixtures of both, occurs in soils as a result of weathering, or is inherited from the parent material. *Most soils of arid and semi-arid regions are calcareous.* In fact, soils of the CWANA region may contain up to 50% $CaCO_3$-equivalent or even more.

As with alkaline pH, soils with free $CaCO_3$ tend to have lower availability of P and of some micronutrient cations. Consequently, $CaCO_3$ equivalent is normally determined in most laboratories of the CWANA region.

While some laboratories also determine "active" $CaCO_3$, it is less common than "total" $CaCO_3$, being mainly in areas of French influence since it was developed by Drouineau (1942) in France. It basically reflects surface area or reactivity of $CaCO_3$ particles, mainly the clay-size fraction. Measurement is based on reaction with excess ammonium oxalate followed by titration with permanganate in an acid medium.

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

Principle

A given weight of soil is reacted with an excess of acid. In this reaction, CO_2 gas is released and the acid not used in the dissolution of carbonates is back-titrated with sodium hydroxide solution (FAO, 1974). Some methods of carbonate determination in soils are based on the collection of CO_2 gas, and the measurement of CO_2 pressure which develops if acid is added to a calcareous soil in a closed flask. In the titrimetric method, two equivalents of acid are assumed to react with one mole of $CaCO_3$. Hence, one equivalent of acid is assumed to be equivalent to one-half mole of $CaCO_3$.

Apparatus

Hot plate.
Burette.
Erlenmeyer flask.
Volumetric pipette.

Reagents

A. Hydrochloric Acid Solution (HCl), 1 *N*

Dilute 82.8 mL *concentrated hydrochloric acid* (37%, sp. gr. 1.19) in DI water, mix well, let it cool, and bring to 1-L volume with DI water.

B. Sodium Hydroxide Solution (NaOH), 1 *N*

Dissolve 40 g *sodium hydroxide* in DI water, and transfer to a 1-L volume, let it cool, and bring to volume with DI water.

C. Phenolphthalein Indicator [$C_6H_4COOC\ (C_6H_4\text{-}4\text{-}OH)_2$]

Dissolve 0.5 g *phenolphthalein* indicator in 100-mL ethanol (ethyl alcohol).

D. Methyl-Orange Indicator [$4\text{-}NaOSO_2C_6H_4N\text{: }NC_6H_4\ /\text{-}4\text{-}N\ (CH_3)_2$]

Dissolve 0.1 g *methyl-orange* indicator in 100-mL DI water.

E. Ethanol (C_2H_5OH), 95%

F. Sodium Carbonate Solution (Na_2CO_3), 1 *N*

Dissolve 53 g *anhydrous sodium carbonate* in DI water, and bring to 1-L volume with DI water.

Procedure

1. Weigh 1 g air-dry soil (0.15-mm) into a 250-mL Erlenmeyer flask.
2. Add 10 mL 1 *N* **hydrochloric acid** solution to the flask with a volumetric pipette.
3. Swirl and leave the flask overnight, or heat to 50 - 60°C, and let the flask cool.
4. Add 50 - 100 mL DI water using a graduated cylinder, and add 2 - 3 drops **phenolphthalein** indicator.
5. Titrate with 1 *N* **sodium hydroxide** solution while swirling the flask. Continue the titration until a faint pink color develops, and take the reading, **R.**

CALCULATION

Percentage **Calcium Carbonate** in soil:

$$\% CaCO_3 = [(10 \times N_{HCl}) - (R \times N_{NaOH})] \times 0.05 \times \frac{100}{Wt} \quad \text{..... (13)}$$

Where:
N_{HCl} = Normality of HCl solution.
R = Volume of NaOH solution used (mL)
N_{NaOH} = Normality of NaOH solution.
Wt = Weight of air-dry soil (g)

Standardization of Solutions

1. Hydrochloric Acid (HCl), 1 *N*

Pipette 10 mL 1 *N Sodium Carbonate* solution into a 100-mL Erlenmeyer flask, add 2 drops *methyl-orange* indicator, and titrate this solution against 1 *N hydrochloric acid* (in the burette). The solution color changes from light to dark orange.

HCl normality is:

$$N_{HCl} = \frac{10 \times N_{Na_2CO_3}}{V_{HCl}} \quad \text{..... (14)}$$

Where:
N_{HCl} = Normality of HCl solution.
V_{HCl} = Volume of HCl solution used (mL)
$N_{Na_2CO_3}$ = Normality of Na_2CO_3 solution.

2. Sodium Hydroxide (NaOH), 1 *N*

Pipette 10 mL standardized 1 *N hydrochloric acid* solution into a 100-mL Erlenmeyer flask, add 2 drops *phenolphthalein* indicator, and titrate against 1 *N sodium hydroxide* solution. The solution color changes from colorless to pink.

NaOH normality is:

$$N_{NaOH} = \frac{10 \times N_{HCl}}{V_{NaOH}} \quad \text{..... (15)}$$

Where: N_{NaOH} = Normality of NaOH solution.
V_{NaOH} = Volume of NaOH solution used (mL)
N_{HCl} = Normality of HCl solution.

Note

1. It requires some experience to accurately determine **color change of the suspension from colorless to pink.**
2. 10 mL 1 *N* **HCl** would dissolve up to 0.5 g $CaCO_3$. That is, if a soil contains 50% $CaCO_3$ or more, 10 mL 1 *N* **HCl** would not be sufficient. In that case, 15 or 20 mL would have to be added.
3. When a soil is reacted with acid to dissolve carbonates, other soil components may also consume acid. Most of the latter reactions are assumed to be reversible, i.e., if the suspension is back-titrated, the acid is released again. For this reason it is *not recommended* to filter the suspension and titrate the clear filtrate. The color change is easier to determine in a clear solution, but the titration value may overestimate the actual $CaCO_3$ content of the soil.
4. Not all reactions involving acid and soil components are completely reversible, and therefore the acid titration of the soil suspension may also slightly over-estimate the actual soil carbonate content. The acid titration method may be calibrated against the **Calcimeter,** if available; however, it is rarely used nowadays.

5.4 Organic Matter

Soil organic matter represents the remains of roots, plant material, and soil organisms in various stages of decomposition and synthesis, and is variable in composition. Though occurring in relatively small amounts in soils, *organic matter (OM) has a major* influence on *soil aggregation*, *nutrient reserve* and its *availability, moisture retention,* and *biological activity*.

Organic carbon (OC) ranges from being the dominant constituent of peat or muck soils in colder regions of the world to being virtually absent in some desert soils. Cultivated, temperate-region soils normally have more than 3 - 4 % OM, while soils of semi-arid rainfed areas, such as in the CWANA region, have normally less than 1% OM.

Most laboratories in the region perform analysis for soil organic matter. The most common procedure involves reduction of potassium dichromate ($K_2Cr_2O_7$) by OC compounds and subsequent determination of the unreduced dichromate by oxidation-reduction titration with ferrous ammonium sulfate (Walkley, 1947; FAO, 1974). While the actual measurement is of oxidizable organic carbon, the data are normally converted to percentage organic matter using a constant factor, assuming that **OM contains 58% organic carbon**. However, as this proportion is not in fact constant, we prefer to report results as **oxidizable organic carbon**, or **multiplied by 1.334 as organic carbon.**

Apparatus

Magnetic stirrer and teflon-coated magnetic stirring bar.
Glassware and pipettes for dispensing and preparing reagents.
Titration apparatus (burette).

Reagents

A. Potassium Dichromate Solution ($K_2Cr_2O_7$), 1*N*

- Dry *potassium dichromate* in an oven at 105°C for 2 hours, cool in a desiccator (silica gel), and store in a tightly stoppered bottle.
- Dissolve 49.04 g *potassium dichromate* in DI water, and bring to 1-L volume with DI water.

B. Sulfuric Acid (H_2SO_4), concentrated (98 %, sp. gr. 1.84)

C. Orthophosphoric Acid (H_3PO_4), concentrated

D. Ferrous Ammonium Sulfate Solution [$(NH_4)_2SO_4.FeSO_4.6H_2O$], 0.5 *M*

Dissolve 196 g *ferrous ammonium sulfate* in DI water, and transfer to a 1-L volume, add 5 mL *concentrated sulfuric acid,* mix well, and bring to volume with DI water.

E. Diphenylamine Indicator $(C_6H_5)_2NH$

Dissolve 1 g diphenylamine indicator in 100 mL concentrated sulfuric acid.

Procedure

1. Weigh 1 g air-dry soil (0.15 mm) into a 500-mL beaker.
2. Add 10 mL 1 *N* **potassium dichromate** solution using a pipette, add 20 mL **concentrated sulfuric acid** using a dispenser, and swirl the beaker to mix the suspension.
3. Allow to stand for 30 minutes.
4. Add about 200 mL **DI water,** then add 10 mL **concentrated orthophosphoric acid** using a dispenser, and allow the mixture to cool.
5. Add 10 - 15 drops **diphenylamine** indicator, add a teflon-coated magnetic stirring bar, and place the beaker on a magnetic stirrer.
6. Titrate with 0.5*M* **ferrous ammonium sulfate** solution, until the color changes from violet-blue to green.
7. Prepare two blanks, containing all reagents but no soil, and treat them in exactly the same way as the soil suspensions.

CALCULATIONS

Percentage **Organic Matter** in soil:

$$M = \frac{10}{V_{blank}} \quad \text{..... (16)}$$

$$\% \text{ Oxidizable Organic Carbon (w/w)} = \frac{[V_{blank} - V_{sample}] \times 0.3 \times M}{Wt} \quad .. (17)$$

$$\% \text{ Total Organic Carbon(w/w)} = 1.334 \times \% \text{ Oxidizable Organic Carbon} \quad .... (18)$$

$$\% \text{ Organic Matter (w/w)} = 1.724 \times \% \text{ Total Organic Carbon} \quad ..... (19)$$

Where:

M = Molarity of ferrous ammonium sulfate solution (approx. 0.5 M)

V_{blank} = Volume of ferrous ammonium sulfate solution required to titrate the blank (mL)

V_{sample} = Volume of ferrous ammonium sulfate ferrous ammonium sulfate solution required to titrate the sample (mL)

Wt = Weight of air-dry soil (g)

0.3 = $3 \times 10^{-3} \times 100$, where 3 is the equivalent weight of C.

Note

1. For soils high in organic matter (1% Oxidizable Organic Carbon or more), more than 10 mL **potassium dichromate** is needed.
2. The factors 1.334 and 1.724 used to calculate ***TOC*** and ***OM*** are approximate; they may vary with soil depth and between soils.
3. Soils containing large quantities of chloride (Cl^-), manganese (Mn^{--}) and ferrous (Fe^{++}) ions will give higher results. The chloride interference can be eliminated by adding **silver sulfate** (Ag_2SO_4) to the oxidizing reagent. No known procedure is available to compensate for the other interferences.
4. The presence of $CaCO_3$ up to 50% causes no interferences.

5.5 Cation Exchange Capacity

Many minerals in soils are negatively charged and, as a consequence, can attract and retain cations such as potassium (K^+), sodium (Na^+), calcium (Ca^{++}), magnesium (Mg^{++}), ammonium (NH_4^+), etc. Cation exchange is a reversible process. Thus, elements or nutrients can be held in the soil and not lost through leaching, and can subsequently be released for crop uptake.

Certain organic compounds also contribute to cation exchange capacity (CEC). Additionally, CEC is influenced by soil pH. A certain portion of the total negative charge is permanent, while a variable portion is pH-*dependent*.

Several methods are available for CEC determination (Rhoades, 1982). Most involve saturation of the soil with an index cation (NH_4^+), removal by washing of excess cation, and subsequent replacement of the adsorbed index cation by another cation (Na^+) and measurement of the index cation in the final extract (Richards, 1954). Modified procedures have been introduced because of high calcium solubility in calcareous and gypsiferous soils (FAO, 1990; Rhoades and Polemio, 1977).

Cation exchange capacity is reported as milliequivalents per 100 g soil or more recently as cmol (+)/*kg soil* **(S.I. unit)**; the actual numbers being the same **(1 meq/100 g = 1 cmol (+)/kg)**. Values of CEC are in the range of 1.0 to 100 meq/100g, least for sandy soils and most for clay soils. Similarly, higher CEC values reflect the dominance of 2:1 clay minerals, and lower values reflect the presence of 1:1 clay minerals.

Apparatus

Flame photometer.
Mechanical shaker, reciprocating.
Centrifuge, capable of 3000 rmp.
Conical centrifuge tubes (50 mL)

Reagents

A. Sodium Acetate Solution (NaOAc), 1 *N*

- Dissolve 136 g *sodium acetate trihydrate* ($CH_3COONa.3H_2O$) in about 950 mL DI water, mix well, and let the mixture cool.
- Adjust pH to 8.2 by adding more *acetic acid* or *sodium hydroxide*, and bring to 1-L volume with DI water.

B. Ethanol (C_2H_5OH), 95%

C. Ammonium Acetate Solution (NH_4OAc), 1*N*

- Add 57 mL *concentrated acetic acid* (CH_3COOH) to 800 mL DI water, then add 68 mL *concentrated ammonium hydroxide* (NH_4OH), mix well, and let the mixture cool.
- Adjust to pH 7.0 by adding more *acetic acid* or *ammonium hydroxide,* and bring to 1-L volume with DI water.

D. Standard Stock Solution

- Dry about 5 g *sodium chloride* (NaCl) in an oven at 105°C for 3 hours, cool in a desiccator, and store in a tightly stoppered bottle.
- Dissolve 2.5418 g dried *sodium chloride* in DI water, and bring to 1-L volume with DI water. This solution contains 1000 ppm Na (*Stock Solution*).
- Prepare a series of Standard Solutions from the *Stock Solution* as follows: Dilute 2, 4, 6, 8, 10, 15, and 20 mL *Stock Solutions* to 100 mL final volume by adding 1 *N* ammonium acetate solution, and 25 mL LiCl *(Diluted Stock Solution)*. These solutions contain 20, 40, 60, 80, 100, 150, and 200 ppm Na, with each containing the same concentration of LiCl (25 ppm).

Procedure

1. Weigh 4 g (for medium to fine textured) or 6 g (for coarse textured) air-dry soil into a 40-mL centrifuge tube, and add 33 mL 1 *N* **sodium acetate trihydrate** solution, stopper tube, and shake for 5 minutes.
2. Remove stopper from tube and centrifuge at 3000 rpm until supernatant liquid is clear. Decant the supernatant as completely as possible and discard.
3. Repeat with 33-mL portions 1 *N* **sodium acetate trihydrate** solution, a total of four times, discarding the supernatant liquid each time. Then add 33-mL 95% **ethanol,** stopper tube, and shake for 5 minutes, unstopper tube, and centrifuge until the supernatant is clear and decant.
4. Wash the sample with 33 mL portions 95% **ethanol,** a total of three times, discarding the supernatant liquid each time. The electrical conductivity (EC) of the supernatant liquid from the third washing should be less than 400 µS/cm.

5. Replace the adsorbed sodium (Na^+) from the sample by extraction with three 33-mL portions 1 *N* **ammonium acetate** solution. Each time shake for 5 minutes, and centrifuge until supernatant liquid is clear.
6. Decant the three supernatant liquids as completely as possible into a 100-mL volumetric flask, bring to volume with 1 *N ammonium acetate* solution, and mix well.
7. Run a series of suitable Na standards, and draw a calibration curve.
8. Measure the samples (soil extract) and take the emission readings by a **Flame Photometer** at 767 nm wavelength.
9. Calculate sodium (Na) concentration according to the calibration curve.

CALCULATION

For **Cation Exchange Capacity** in soil:

$$CEC\ (meq/100\ g) = meq/L\ Na\ (from\ calibration\ curve) \times \frac{A}{Wt} \times \frac{100}{1000} \quad (20)$$

Where: A = Total volume of the extract (mL)
Wt = Weight of the air-dry soil (g)

Note

Though quite laborious, the method of Rhoades and Polemio (1977) is more appropriate for soils containing carbonates, gypsum, and zeolite.

5.6 Gypsum

Soils with variable contents of gypsum ($CaSO_4.2H_2O$) are common in many countries of the CWANA region, including Syria and Iraq. Gypsum is primarily a concern in irrigated areas and less so in rainfed agriculture. Thus, its determination is of importance to some laboratories in the region.

The standard method for gypsum determination described here is that of Richards (1954) which involves precipitation with acetone. Modifications of that method and other procedures (Sayegh *et al*., 1978) are found in the FAO bulletin on gypsiferous soils (FAO, 1990).

Apparatus

Centrifuge, capable of 4000 rmp.
Conical centrifuge tubes (50 mL)
Conductivity cell and Wheatstone bridge.
Mechanical shaker.

Reagent

Acetone

Procedure (Quantitative)

1. Weigh 10 to 20 g air-dry soil (medium to fine textured) into a 250-mL bottle, and add a measured volume of **DI water** sufficient to dissolve the gypsum present.
2. Stopper the bottle and shake by hand six times at 15-minute intervals or agitate for 15 minutes in a mechanical shaker.
3. Filter the extract through filter paper of medium porosity, and transfer a 20-mL aliquot of filtered extract into a 50-mL conical centrifuge tube.
4. Add 20 mL **acetone,** mix well, and let stand until precipitate is flocculated. This usually requires 5 to 10 minutes.
5. Centrifuge at 4000 rpm for 3 minutes, decant supernatant liquid, invert tube, and drain on filter paper for 5 minutes.
6. Disperse precipitate and rinse tube wall with a stream of 10 mL blown from a pipette.

7. Again, centrifuge for 3 minutes, decant supernatant liquid, invert tube, and drain on filter paper for 5 minutes.
8. Add exactly 40 mL **DI water** to tube, stopper, and shake until the precipitate is completely dissolved. Measure electrical conductivity of solution, and correct conductivity reading to 25°C.
9. Determine gypsum concentration in the solution by reference to a graph showing the relationship between the concentration and EC constructed by means of the following data from the International Critical Tables (Richards, 1954, **Fig. 2**).

Gypsum Concentration	**Electrical Conductivity (25°C)**
----- meq L^{-1} ------	---------- dS m^{-1} ----------
1.0	0.121
2.0	0.226
5.0	0.500
10.0	0.900
20.0	1.584
30.5	2.205

CALCULATIONS

For **Gypsum** in soil:

$$CaSO_4.2H_2O \text{ in aliquot (meq)} = CaSO_4.2H_2O \text{ from conductivity reading (meq/L)} \times \text{water used to dissolve precipitate (mL)}/1000 \quad (21)$$

$$\text{Gypsum (meq/100 g)} = 100 \times CaSO_4.2H_2O \text{ in aliquot (meq)}/\text{(soil water) ratio} \times \text{(soil - water) extract used (mL)} \quad (22)$$

Note

1. Sodium and potassium sulfates, when present in sufficiently high concentrations, are also precipitated by acetone. The maximum concentrations of sodium sulfate and of potassium that may be tolerated are 50 and 10 meq/L, respectively.
2. At a 1:5 (soil: water) ratio, water will dissolve approximately 15 meq gypsum per 100 g soil. If it is found that the gypsum content of the soil approaches 15 meq/100 g using a 1:5 (soil: water) extract, the determination should be repeated, using a diluted extract.
3. In some soils from the Euphrates Basin, gypsum may be well over 25%, in which case dilution's of 1:500 or 1:1000 (soil: water) ratio have to be used.
4. **Qualitative test** for gypsum should be made on all soils as a routine in order to save time later when analyzing for gypsum. Pipette 5 mL of the soil extract into a small centrifuge tube and add 5 mL acetone. Mix well, and allow to stand for 10 minutes if a flocculate white precipitate forms, the soil contains gypsum; if no precipitate forms, the soil is considered to have no gypsum.