## PREPARATION OF SOIL SAMPLES FOR ANALYSIS

Handling in the laboratory As soon as the samples arrive at the soil testing laboratory, they should be checked against the accompanying information list. If the laboratory personnel have collected the samples themselves, then adequate field notes should have been kept. All unidentifiable samples should be discarded. Information regarding samples should be recorded in a register, and each sample should be given a laboratory number, in addition to the sample number, to help to distinguish it where more than one source of samples is involved.

Drying of samples Samples received in the laboratory may be moist. They 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 samples 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 °C and whose relative humidity should be 30–60 percent. Oven drying a soil can cause profound changes in the sample. This step is not recommended as a preparatory procedure despite its convenience. Drying has a negligible effect on total N content, but the nitrate content in the soil changes with time and temperature. Drying at a high temperature affects the microbial population. With excessive drying, soil K may be released or fixed depending on the original level of exchangeable K. Exchangeable K will increase if its original level was less than 1 me/100 g soil (1 cmol/kg) and vice versa, but the effect depends on 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 an oven-dry basis by estimating separately the moisture content in the samples.

Post-drying care After drying, the samples are taken to the preparation room. Air-dried samples are ground with a wooden pestle and mortar so that the soil aggregate is 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 a positive bias in the sample as

the rejected part may include soil elements with differential fertility. Therefore, the entire sample should 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 until all aggregate particles are fine enough to pass the sieve and only pebbles, organic residues and concretions remain.

If the soil is to be analysed 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 has passed through the sieve, it must be mixed again 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.

ANALYTICAL METHODS The following estimations are generally carried out in a service-oriented soil testing laboratory: soil texture, soil structure, cation exchange capacity (CEC), soil moisture, water holding capacity, pH, lime requirement, electrical conductivity, gypsum requirement, organic C, total N, mineralizable N, inorganic N, available P, available K, available S, calcium, calcium plus magnesium, micronutrients – available Zn, Cu, Fe, Mn, B and Mo.

SAMPLE COLLECTION AND PREPARATION FOR ANALYSIS Representative sampling should be done of specific plant parts at the growth stage that is most closely associated with critical values as provided by research data. Sampling criteria and procedures for individual samples are similar to those of soil testing in that the sample should be representative of the field. A predetermined, representative number of plants from a homogenous sampling unit contribute to the composition of bulk sample. The composite sample should be about 200–500 g fresh weight. Factors such as the desired precision of recommendation, the nature of the crop (seasonal or perennial) and economic considerations should be taken into account. The following procedure is suggested: 1. For analysis of seasonal crop plants, pick a few representative plants at random from each plot. Remove the shoot (aerial part) with the help of a sharp stainless steel cutter for whole shoot analysis or the desired part for analysis of specific plant parts. 2. If roots are to be included, uproot the whole plant carefully from wet soil, retaining even the fine/active roots. Dip the plant roots gently in water several times to remove adhering soil. 3. Wash with water several times. 4.

Wash the samples with about 0.2 percent detergent solution to remove the waxy/greasy coating on the leaf surface. 5. Wash with 0.1M HCl followed by thorough washing with plenty of water. Give a final wash with distilled water. 6. Wash with DDW if micronutrient analysis is to be carried out. 7. Soak to dry with tissue paper. 8. Air-dry the samples on a perfectly clean surface at room temperature for at least 2–3 days in a dust-free atmosphere. 9. Put the samples in an oven, and dry at 70 °C for 48 hours. 10. Grind the samples in an electric stainless steel mill using a 0.5-mm sieve. Clean the cup and blades of the grinding mill before each sample. 11. Put the samples back in the oven, and dry again for constant weight. Store in well-stoppered plastic or glass bottles or in paper bags for analysis.

ANALYTICAL METHODS The plant sample can be brought into solution form through digestion with acids that dissolve the solid plant parts and bring the plant nutrient in liquid form for estimation. This is called wet digestion. The plant sample can also be heated at high temperatures to destroy OM, and the ash so obtained can be dissolved in acids to bring the sample into liquid form for estimation. This method is called dry ashing.

Wet digestion A mixture of $HNO_3$, $H_2SO_4$ and $HClO_4$ in the ratio of 9:4:1 is used for sample digestion. It is known as tri-acid digestion. When only two acids, viz. $HNO_3$ and $HClO_4$ (9:4), are used, it is known as di-acid digestion. Perchloric acid ($HClO_4$) is used primarily for increasing the efficiency of oxidation of the sample as $HClO_4$ dissociates into nascent chlorine and oxygen at high temperature, which increases the rate of oxidation or the digestion of the sample. At times, perchloric acid causes an explosion when it comes into direct contact with the plant sample. Therefore, pre-digestion of the sample with $HNO_3$ is considered desirable, followed by treatment with the di-acid or tri-acid mixture. Generally, 1 g of ground plant sample is taken for analysis. It is placed in a 100-ml volumetric flask, and 10 ml of acid mixture is added and the contents are mixed by swirling. The flask is placed on a hotplate in the fumehood and heated, starting at 80–90 °C and then the temperature is raised to about 150–200 °C. Heating continues until the production of red $NO_2$ fumes ceases. The contents are further heated until the volume is reduced to 3–4 ml and becomes colourless, but it should not be dried. After cooling the contents, the volume is made up with the distilled water and filtered through No. 1 filter paper. This solution is used for nutrient estimation. Di-acid digestion is used for determination of most of the elements (P, K, Ca, Mg, S, Fe, Mn, Zn and Cu). However, tri-acid digestion is preferred for P and K estimations. It cannot be

used for S estimation owing to the presence of $H_2SO_4$. Sulphuric acid also contains many trace elements as contaminants. Therefore, micronutrients should preferably be estimated through di-acid digestion or by using a dry-ash sample solution. Wet digestion can also be accomplished by using $H_2O_2$ to destroy OM followed by digestion with $H_2SO_4$ to dissolve the sample. In such digestion, N estimation can be carried out as per the Kjeldahl method of total N estimation as described in Chapter 3.

Dry ashing High-temperature oxidation destroys the OM. The plant sample is ashed at 500–600 °C by placing a suitable weight (0.5–1.0 g) of the sample in a silica crucible and heating it in a muffle furnace for 4–6 hours. The ash residue is dissolved in dilute $HNO_3$ or HCl, filtered through acid-washed filter paper in a 50/100-ml volumetric flask, and the volume is made up to the mark. The estimation of K, Ca, Mg and micronutrients (including B and Mo) is carried out in the dry-ashed sample solution. Dry ashing is a preferred method for the analysis of P, K, Ca, Mg and trace elements, especially B and Mo. It is a relatively simple method and requires very little operational attention. It does not involve the use of perchloric acid. It also avoids the use of boiling acids. However, at times, incomplete recovery of some elements may be caused by: Volatilization of elements such as S (also Se and halogens). To avoid loss of S, $Mg(NO_3)_2$ should be mixed with plant samples while dry ashing. Retention of elements such as Cu on the walls of silica crucibles. Hence, platinum crucible should be used. Formation of compounds that are not completely soluble in the acid used for digestion. A blank should always be carried out to account for any contamination through the acids used in the digestion.