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by

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The Soil Fertility and Analytical Services Section of the KwaZulu-Natal Department of Agriculture and Rural Development performs a variety of analyses on soil, plant tissue, compost, manure and water samples. This document gives brief descriptions of the analyses most commonly performed.

### **Soil analysis**

The Soil Fertility Laboratory routinely performs the following analyses as part of the Department's Fertilizer Advisory Service, using the rapid procedures described by Hunter (1975) and Farina (1981): Ambic-2-extractable P, K, Cu, Mn and Zn, KCl-extractable Ca, Mg and acidity, and pH (KCl); NIRS-estimates of organic carbon and clay content are also done routinely. Other soil analyses done by the Section are: EC, Ca, Mg, K, and Na in the saturation extract, ammonium-acetate extractable Na, pH (water), total carbon, nitrogen, and sulphur, Walkley-Black organic carbon, and particle size distribution. These methods are briefly described below.

#### ***Sample preparation: Soil Fertility Laboratory***

Soil samples are air dried at room temperature; they are spread out in drying trays and air is forced over them. When dry, the samples are crushed between rubber belts on a soil crusher and passed through a 1-mm sieve. Material coarser than 1 mm that cannot be crushed (such as stones, gravel and concretions), is discarded.

#### ***Batch handling: Soil Fertility Laboratory***

Samples are scooped into trays which each contain 11 PVC cups (capacity 70 mL); a tray is used for nine unknown samples, one standard soil sample (for quality control) and one blank. For operations such as dispensing and stirring and for quality control, batches of three trays (27 samples, three unknowns, and three blanks) are used. Multiple dispensers and diluter/dispensers are used to dispense aliquots of extractant or reagent to three samples at a time.

#### ***Sample density***

Soil samples are analysed on a volume rather than a mass basis. To enable the conversion of the results to a mass basis, the mass of a 10-mL scoop of a dried and milled sample is measured and the calculated sample density is reported.

#### ***pH (KCl)***

10 mL of soil is scooped into sample cups. 25 mL of 1 M KCl solution is added and the suspension is stirred at 400 r.p.m. for 5 min using a multiple stirrer. The suspension is allowed to stand for about 30 minutes, and the pH is measured using a gel-filled combination glass electrode while stirring. De-ionised water is substituted for the 1 M KCl solution if pH (water) is required.

### ***Extractable (1 M KCl) calcium, magnesium and acidity***

2.5 mL of soil is scooped into sample cups. 25 mL of 1 M KCl solution is added and the suspension is stirred at 400 r.p.m. for 10 min using a multiple stirrer. The extracts are filtered using Whatman No.1 paper. 5 mL of the filtrate is diluted with 20 mL of 0.0356 M SrCl<sub>2</sub>, and Ca and Mg determined by atomic absorption. To determine extractable acidity, 10 mL of the filtrate is diluted with 10 mL of de-ionised water containing 2-4 drops of phenolphthalein, and titrated with 0.005 M NaOH.

### ***Extractable (Ambic-2) phosphorus, potassium, zinc, copper and manganese***

The Ambic-2 extracting solution consists of 0.25 M NH<sub>4</sub>CO<sub>3</sub> + 0.01 M Na<sub>2</sub>EDTA + 0.01 M NH<sub>4</sub>F + 0.05 g L<sup>-1</sup> Superfloc (N100), adjusted to pH 8 with a concentrated ammonia solution. 25 mL of this solution is added to 2.5 mL soil, and the suspension is stirred at 400 r.p.m. for 10 min using a multiple stirrer. The extracts are filtered using Whatman No.1 paper. Phosphorus is determined on a 2 mL aliquot of filtrate using a modification of the Murphy and Riley (1962) molybdenum blue procedure (Hunter, 1974). Potassium is determined by atomic absorption on a 5 mL aliquot of the filtrate after dilution with 20 mL de-ionised water. Zinc, Cu and Mn are determined by atomic absorption on the remaining undiluted filtrate.

### ***Effective CEC (ECEC) and Acid saturation***

Effective CEC is calculated as the sum of KCl-extractable Ca, Mg, and acidity and Ambic-2 extractable K. Percent acid saturation of the ECEC is calculated as "extractable acidity" x 100 / (Ca + Mg + K + "extractable acidity").

### ***Estimate of organic carbon by near-infrared spectroscopy***

Organic carbon is estimated for all soil samples routinely analyzed in the Soil Fertility Laboratory, by near-infrared reflectance, using the air-dry, milled soil samples.

### ***Estimate of clay content by near-infrared spectroscopy***

Clay content is estimated for all soil samples routinely analyzed in the Soil Fertility Laboratory, using a combination of near-infrared reflectance using the air-dry, milled soil samples, and the measured sample density.

### ***Total carbon and nitrogen in soil***

Total C, N and S are analyzed by the Automated Dumas dry combustion method using a LECO CNS 2000 (Leco Corporation, Michigan, USA; Matejovic, 1996). Briefly, this method involves weighing samples into a ceramic crucible to which 0.5g of vanadium pentoxide is added as a combustion catalyst. The vanadium pentoxide also improves the recovery of sulphur. The crucible is introduced into a horizontal furnace, where the sample is burned in a stream of oxygen at 1350°C. The gases produced are passed through two infra-red cells where the sulphur (as SO<sub>2</sub>) and carbon (as CO<sub>2</sub>) are determined. Nitrogen is determined (as N<sub>2</sub>) in a thermal conductivity cell.

### ***Organic carbon by the Walkley-Black method***

This method is based on the Walkley-Black procedure (Allison, 1965) and measures the readily oxidizable organic carbon. The organic matter is oxidized by potassium dichromate in a sulphuric acid medium. The excess dichromate is determined by titration with standard ferrous sulphate solution.

### ***Particle size distribution of soils and soil texture***

Suspended clay and fine silt are determined after dispersion and sedimentation; sand fractions are determined by sieving (Day, 1965). A 20 g soil sample (<2 mm) is treated with hydrogen peroxide to oxidise the organic matter. The sample is made up to 400 ml with de-ionized water and left overnight. The clear supernatant is siphoned off and the sample puddled. A further addition of de-ionized water is added, the sample stirred and left overnight. The clear supernatant is again siphoned off. Dispersing agents (NaOH and sodium hexametaphosphate) are added and the sample stirred on Hamilton Beach stirrers. The suspension is made up to 1 litre in a measuring cylinder and the clay (<0.002 mm) and fine silt (0.002-0.02 mm) fractions measured with a pipette after sedimentation. Fine silt plus clay is measured after 4-5 min (exact time depends on temperature) at 100 mm, and clay after 5-6 h at a depth of 75 mm. Sand fractions include very fine sand (0.05 - 0.10 mm), fine sand (0.10 - 0.25 mm), medium sand (0.25 - 0.50 mm) and coarse sand (0.50 - 2.0 mm) which are determined by sieving. Coarse silt (0.02-0.05 mm) is estimated by difference.

### ***Determination of textural class by means of a textural triangle***

Once the particle size distributions of the two soils are known, their textural class is determined from a diagram (textural triangle) defining particle size limits of the various textural classes (Soil Classification Working Group, 1991).

### ***Salinity and sodicity***

Saturation extracts of soils are analyzed for electrical conductivity (EC) using an EC meter and Ca, Mg, Na and K are determined by atomic absorption. Sodium adsorption ratio (SAR) is determined by calculation.  $SAR = [Na] / \{([Ca] + [Mg]) / 2\}^{0.5}$ , where [Na], [Ca] and [Mg] are concentrations of the elements expressed in mmol/L.

### ***Exchangeable Na***

Exchangeable Na is determined by atomic absorption after extraction of a 2.5 mL soil sample with 25 mL of 1 M ammonium acetate (Soil Classification Working Group, 1991).

### ***Ammonium - N (NH<sub>4</sub><sup>+</sup>) and nitrate - N (NO<sub>3</sub><sup>-</sup>)***

Ammonium - N (NH<sub>4</sub><sup>+</sup>) and nitrate - N (NO<sub>3</sub><sup>-</sup>) in filtered extracts are measured by segmented flow analysis with a Perstorp Flow Solution III analyzer using the sodiumsalicylate-sodium nitropusside-hypochlorite method for NH<sub>4</sub><sup>+</sup> - N (Perstorp Analytical, 1993) and the sulphanimide-naphthyl-ethylenediamine method for NO<sub>3</sub><sup>-</sup> - N plus NO<sub>2</sub> - N after having reduced nitrate to nitrite with copperized cadmium wire (Willis & Gentry, 1987).

## Plant tissue, manure and compost analysis

Plant material, manures and compost samples are analyzed using the following procedures. The batch-handling procedures are similar to those used for the soil analyses described above. Samples are dried at 75°C, and milled to pass through a 0.84 mm sieve. Subsamples are then dry ashed at 450°C overnight and taken up in 1 M HCl. The P concentration is determined colorimetrically by the same method used for soil extracts, and K, Na, Ca, Mg, Cu, Mn, and Zn are determined by atomic absorption. New development:- P, K, Al, Ca, Mg, Na, Cu, Mn and Zn are determined on ICP. Samples for B analysis are ashed separately, and B is determined photometrically by the azomethine H method (Gaines & Mitchell, 1979).

Nitrogen is determined using one of the following methods:

- before ashing, using near-infrared reflectance (Eckard *et al.*, 1988);
- after macro-Kjeldahl digestion, by bubble-segmented flow analysis using the colorimetric phenol-hypochlorite method (de Figueredo & Thurtell, 1998); or
- by the Automated Dumas dry combustion method using a LECO CNS 2000 (Leco Corporation, Michigan, USA; Matejovic, 1996), described above (**Total C and N in soil**).

## Water analysis

Water samples are analyzed to determine their suitability for irrigation, hydroponics and for use with herbicide and pesticide sprays. Electrical conductivity (EC) is measured using an EC meter, and Ca, Mg, Na, K, Fe and Mn are determined by atomic absorption. Sodium adsorption ratio (SAR) is determined by calculation.  $SAR = [Na] / \{([Ca] + [Mg]) / 2\}^{0.5}$ , where [Na], [Ca] and [Mg] are concentrations of the elements expressed in mmol/L.

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