CSBP SOIL ANALYSIS METHOD SUMMARIES

Revised by Stephen Loss and others June 2012

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SOIL METHODS

Unless specified otherwise, all soils are dried at 40°C for 24 hours or until dry, and then crushed and sieved to less than 2mm.

Most of the following methods are based on those described in "Soil Chemical Methods - Australasia" (2011) by Rayment, GE and Lyons, D. CSIRO Publishing. Melbourne, Australia. The codes used by this reference (eg. 10D1) are included below.

1. COLWELL PHOSPHORUS & POTASSIUM (mg/kg) - 9B & 18A1

Plant available P and K are typically measured using the Colwell method. Using a soil to solution ratio of 1:100, soils are extracted with 0.5 M sodium bicarbonate solution adjusted to pH 8.5 for 16 hours. The acidified extract is treated with ammonium molybdate/antimony trichloride reagent and the phosphorus is measured colorimetrically at 880nm using a discrete analyser. The potassium in the extract is determined using a flame atomic absorption spectrophotometer at 766.5nm.

Colwell, J.D. (1965). An automatic procedure for the determination of Phosphorus in sodium hydrogen carbonate extracts of soils. *Chemistry Industry*. pp. 893-895.

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. pp. 162 & 385.

2. NITRATE & AMMONIUM (mg/kg) - 7C2b

Soil nitrate nitrogen and ammonium nitrogen are extracted with a 2M potassium chloride solution for 1 hour at 25°C. After dilution the resulting soil solution is measured on a Lachat Flow Injection Analyzer. Ammonium is measured colorimetrically at 630nm using the indo-phenol blue reaction. Nitrate is reduced to nitrite through a copperized-cadmium column and the nitrite is also measured colorimetrically at 520nm.

Searle, P.L. (1984). The Bertholet or indophenol reaction and its use in the analytical chemistry of Nitrogen. *Analyst* 109, 549-568.

Lachat Instruments, Methods Manual for the 'Quikchem' Auto Ion Analyser. Quikchem Method No 12-107-04-1-B.

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 130.

3. SULPHUR (mg/kg) - 10D1

Plant available sulphur in soils is determined by extraction with a 0.25M potassium chloride solution for 3 hours at 40°C. The sulphur content of extracts are analysed by Inductively Coupled Plasma Spectrometry (ICP). This method is known as the KCI-40 or Blair/Lefroy Extractable Sulphur method.

Blair, G., Chinoim, N., Lefroy, R., Anderson, G. & Crocker, G. (1991). *Aust J Soil Res* <u>29</u>: 619-626. Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 223.

4. ORGANIC CARBON (%) - 6A1

In the Walkley Black (1934) method concentrated sulfuric acid is added to soil wetted with dichromate solution. The chromic ions produced are proportional to oxidized organic carbon and are measured colorimetrically at 600nm on a plate reader. Unlike the method for total organic carbon where external heat is applied (see non-routine tests), the heat of the acid-based reaction is used to induce oxidation of soil organic matter.

 Walkley, A. & Black, I.A. (1934). An Examination of the Degtjareff Method for Determining Soil Organic Matter, A Proposed Modification of the Chromic Acid Titration Method. Soil Science. 37(1):29-38
Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 68.

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5. SOIL pH & CONDUCTIVITY (ds/m) - 4A1, 4B3, 3A1

Using a soil to solution ratio of 1:5, soils are extracted in deionised water for 1 hour. The water pH and electrical conductivity of the extract are measured using a combination pH and conductivity electrode. After the water pH and EC have been measured, calcium chloride solution is added to the soil solution to the equivalent of 0.1M and after thorough mixing for 10 minutes the calcium chloride pH is also measured. All measurements are recorded while the solution is stirred.

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. pp.38 & 20.

7. PHYSICAL OBSERVATIONS

Unprepared soil samples are laid on a cardboard tray and soil texture, colour and gravel content are estimated by a quick physical observation. These procedures were devised to give customers an indication whether the soil type changed between samples, not as an accurate measurement of the soil characteristics. Our classification systems do not correspond to any other systems eg. Northcote classification.

7.1 Texture

Texture is assessed by wetting the soil and "feeling" the wet soil between the forefinger and thumb. Six texture categories are used: sand (1.0), loamy sand (1.5), loam (2.0), clay loam (2.5), Clay (3.0) and heavy clay (3.5).

7.2 Colour

Soils are classified into the following colours: white, grey, yellow, brown, orange, red, pink and black. More than one colour may be included (eg, brown yellow) and "light" and "dark" may also be assigned.

7.3 Gravel Content

The gravel content is estimated visually and by running the fingers through the soil. Approximate figures are reported eg. 10-15%.

11. TRACE ELEMENTS - Cu, Fe, Mn, Zn (mg/kg)

<u>11.1 DTPA - 12A1</u>

Soils are extracted at a ratio of 1:2 with diethylene-triamine-penta-acetic acid (DTPA) solution (which has a high affinity for metal ions) for 2 hours and the concentration of copper, zinc, manganese and iron measured by Atomic Absorption Spectroscopy. This is the most common method for determining plant available trace elements.

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 240.

<u>11.2 EDTA - 12B1</u>

Soils are extracted at a ratio of 1:5 with ethylenediamine tetra-acetic acid di-sodium salt (EDTA - a chelating agent widely used to sequester metal ions) for 1 hour and the concentration of copper, zinc, manganese and iron in the resulting extractants are determined by Inductively Coupled Plasma (ICP) Spectroscopy.

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 240.

<u>11.3 HCL - 12D1</u>

Soils are extracted with 0.1M hydrochloric acid in a ratio of 1:10 for one hour and read for Fe, Mn, Cu and Zn by Inductively Coupled Plasma (ICP) Spectroscopy.

Nelson, J.L., Boawn, L.C., and Viets, F.G (1959) A method for assessing Zn status of soils using acidextractable Zn and titratable alkalinity values. Soil Science, vol 88, 275-283 Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 245.

11.4 Mehlich - 18F1

Soils are extracted in a ratio of 1:7 with the Mehlich solution (ammonium nitrate, ammonium fluoride, EDTA, acetic acid and nitric) for 5 minutes and read for Fe, Mn, Cu and Zn by Inductively Coupled Plasma (ICP) Spectroscopy.

Mehlich A (1984) Mehlich 3 soil test extractant: A modification of Mehlich 2. Communications of Soil Science and Plant Analysis 15, 1409-1416

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 398.

11.5 Aqua Regia - 17B2

Soil samples are digested in a hot hydrochloric acid and nitric acid mix. Digests are diluted with deionised water to dissolve any precipitates and the clear solutions are read by Inductively Coupled Plasma Spectrometry (ICP). This method determines the total trace elements present in the soil.

 Chen, M & L.Q. Ma. (2001). Lena Q. Ma. Comparison of Three Aqua Regia Digestion Methods for Twenty Florida Soils Soil Science Society of America Journal 65:491-499
Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 379.

12. EXCHANGEABLE CATIONS - Al, Ca, Mg, K, Na (meq/100g)

<u>12.1 NH₄Cl₂/ BaCl₂ - 15E1</u>

Soils are extracted at a ratio of 1:10 with 0.1M $NH_4Cl/0.1M$ $BaCl_2$ for 2 hours. Exchangeable cation concentrations of the resulting extracts are determined by Inductively Coupled Plasma (ICP) Spectroscopy.

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 322.

<u>12.2 NH₄Cl - 15A1</u>

Soils are extracted at a ratio of 1:20 with 1 M NH₄Cl for 1 hour. Exchangeable cation concentrations of the resulting extracts are determined by Inductively Coupled Plasma (ICP) Spectroscopy.

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 293.

12.3 Prewash - 15A2

In soils with high ionic strength (i.e. saline soils) the soluble salts hinders the extraction of cations. Leaching of the soil with an alcohol solution removes the soluble salts from the soil, thus allowing for accurate measurement. Following an ethanol prewash, soils are extracted with 0.1 M NH_4Cl for 1 hour. Exchangeable cation concentrations of the resulting extracts are determined by Inductively Coupled Plasma (ICP) Spectroscopy.

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 302.

12.4 Saturation Paste - 14H1

Samples are mixed with distilled water until they reach saturation point and are left overnight before centrifuging. Saturation paste extracts are collected and read by Inductively Coupled Plasma (ICP) Spectroscopy for cations. The extract is also read for conductivity.

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 287.

12.4 Water Soluble Cations & Chloride - 5A4

Water soluble soil chloride and exchangeable cations are determined using a 1:5 soil:water extraction for 1 hour. Exchangeable cation and chloride concentrations in the resulting extracts are determined by Inductively Coupled Plasma (ICP) Spectroscopy. This test is used in combination with

the NH_4Cl_2 / $BaCl_2$ extractable test, where the value for water soluble exchangeable cations are subtracted from the value for NH_4Cl_2 / $BaCl_2$ extractable cations.

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 62.

20. TOTAL CARBON & NITROGEN (LECO) (%) - 7A5

Total carbon and total nitrogen are determined by Dumas high temperature combustion (LECO analyzer), where soil samples are loaded into a combustion tube at 950°C and flushed with oxygen. All gases generated are collected and measured on an infrared detector for carbon and a thermal conductivity cell for nitrogen.

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 110

23. CARBONATE ANALYSIS (%) - 19B2

Carbonate minerals play an important role in soil management. In this method carbonates are neutralized with dilute hydrochloric acid in a reaction that produces carbon dioxide. This reaction is performed in a closed vessel the pressure increase in the vessel is proportional to the amount of carbonate in the closed system.

Rayment, GE & Lyons, D. (2011) Soil Chemical Methods - Australasia. CSIRO Publishing. p. 420.

25. PARTICLE SIZE

Prepared soil samples are treated with hydrogen peroxide to remove the organic matter, and then shaken with a 1:1 Calgon - Sodium Hydroxide mixture to disperse the soil particles. Using a table of particle sedimentation times, 25ml aliquots of the solution are removed at the set times and the remaining sample is sieved. The aliquots are evaporated in an oven and weighed to determine the coarse and fine sand, silt and clay contents.

S.J. Indorante, L.R. Follmer, R.D. Hammer and P.G. Koenig 1990. Particle-Size Analysis by a modified Pipette procedure. Soil Sci. Soc. Am. J., Vol 54