

CSBP Laboratory Methods

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Soil Testing Methods

Soil Sample Preparation

Soil samples are dried at 50 °C and ground and sieved to 2 mm.

Acid Phosphorus (BSES)

Rayment and Lyons Method 9G2

Units of Measurement: mg/kg

Acid extractable phosphorus can be used to measure labile phosphorus within the sample. Soils are extracted using 0.005M sulphuric acid and the phosphorus in the extract is determined colourimetrically.

Aluminium (CaCl₂)

Bromfield method (1987)

Units of Measurement: mg/kg

Soils are extracted using a 0.01M Calcium chloride solution in a ratio of 1:5. Colloidal material is separated and removed from the extract and which is then analysed for aluminium using inductively coupled plasma (ICP) spectroscopy.

Bromfield, S.M. (1987). Simple tests for the assessment of aluminium and manganese levels in acid soils. *Australian Journal Agriculture* 27, 399-404.

Boron

Rayment and Lyons Method 12C1

Units of Measurement: mg/kg

Soils are extracted using 0.01M calcium chloride, at a ratio of 1:4. The mixture is heated to 90 °C and the extract is read for boron using inductively coupled plasma (ICP) spectroscopy.

Bray Phosphorus I and Bray Phosphorus II

Rayment and Lyons Method 9E2

Units of Measurement: mg/kg

Soils are extracted using a dilute hydrochloric acid solution, containing ammonium fluoride, at a ratio of 1:7. The soil extract is then treated with ammonium molybdate/potassium antimony trichloride reagent and phosphorus concentration is measured colourimetrically.

Calcium Carbonate Percentage

Rayment and Lyons Method 19B2

Units of Measurement: %

The carbonates in soil samples are neutralised using dilute hydrochloric acid. This reaction produces carbon dioxide and is performed in a closed vessel. The pressure increase within the vessel is thus proportional to the amount of carbonate in the closed system. This test cannot be performed on soil samples with high carbonate content due to large amount of carbon dioxide produced within the test, imposing safety risks to the technicians performing the test.

Chloride

Rayment and Lyons Method 5A1

Units of Measurement: mg/kg

Water soluble chloride in soil is determined using a 1:5 soil: water extraction. Chloride concentration in the resulting extract is determined colourimetrically.

Colwell Phosphorus and Colwell Potassium

Rayment and Lyons Method 9B and 18A1

Units of Measurement: mg/kg

Measures plant available phosphorus and potassium. Using a soil to solution ratio of 1:100, soils are extracted with 0.5M sodium bicarbonate solution adjusted to pH 8.5 for 16 hours. The extract is then acidified and measured colourimetrically for Phosphorus. Potassium is determined using atomic absorption spectroscopy.

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

Dispersion

Emerson method

Units of Measurement: Dispersion index 0-16

Soil samples are immersed in deionised water and dispersion is rated after 2 and 20 hours. Dispersion is a subjective physical characterisation of the soil. .

Allen, DG and Jeffery, RC (1990) 'Methods for Analysis in Western Australian Soils', Chemistry Centre of WA.

Loveday & Pyle (1973). The Emerson dispersion test and its relation to hydraulic conductivity. CSIRO Aust. Div. Soil. Tech. Paper No. 15.

Electrical Conductivity - Saturated Paste

Rayment and Lyons Method 14B1

Units of Measurement: dS/m

Soil samples are mixed with deionised water until they reach the saturation point and incubated. The supernatant from these samples are then taken and read for electrical conductivity using conductivity electrodes.

Electrical Conductivity and Exchangeable Cations - Saturated Paste

Rayment and Lyons Method 14B1, 14H1

Units of Measurement: dS/m; meq/100g

Soil samples are mixed with deionised water until they reach the saturation point and incubated. The supernatant from these samples are then taken and read for electrical conductivity using conductivity electrodes. Exchangeable cations are measured using inductively coupled plasma (ICP) spectroscopy.

Electrical Conductivity and Exchangeable Cations and Carbonates - Saturated Paste

Rayment and Lyons Method 14B1, 14H1

Units of Measurement: dS/m; meq/100g

Soil samples are mixed with deionised water until they reach the saturation point and incubated. The supernatant from these samples are then taken and read for electrical conductivity using conductivity electrodes. Exchangeable cations are measured using inductively coupled plasma (ICP) spectroscopy. The supernatant is then titrated with hydrochloric acid to determine the concentration of carbonates.

Exchangeable Acidity/Aluminium

Rayment and Lyons Method 15G1

Units of Measurement: meq/100g

Soils are extracted using a 1M potassium chloride solution at a ratio of 1:10. The exchangeable Aluminium (Al^{3+}) and the exchangeable acidity are measured by titration using sodium hydroxide and hydrochloric acid. A calculation is then used to determine the concentration of H^+ .

Exchangeable Cations in Water (Calcium, Magnesium, Sodium, Potassium)

Rayment and Lyons Method 5A4

Units of Measurement: meq/100g

Water soluble exchangeable cations are determined using a 1:5 soil: water extraction. Exchangeable cations in the resulting extracts are determined using inductively couple plasma (ICP) spectroscopy.

Exchangeable Cations with pre-wash (Calcium, Magnesium, Sodium, Potassium)

Rayment and Lyons Method 15A2

Units of Measurement: meq/100g

In soils with high ionic strength (high salinity), soluble salt hinders extraction of cations. In this method, soil is leached with both an alcohol and glycerol solution (pre-wash) to remove soluble salts from the soil prior to extraction using 1M ammonium chloride. Exchangeable cation concentrations of the resulting extracted are determined using inductively couple plasma (ICP) spectroscopy.

Exchangeable Cations without pre-wash (Calcium, Magnesium, Sodium, Potassium, Aluminium)

Rayment and Lyons Method 15E1

Units of Measurement: meq/100g

Soils are extracted using a mixture of 0.1M ammonium chloride and barium chloride, at a ratio of 1:10. Exchangeable cation concentrations of the resulting extracts are determined using inductively couple plasma (ICP) spectroscopy.

Note: CSBP laboratory is able to perform 15A1 as well; which is extraction using 1M ammonium chloride, at 1:20 ratio. Please ask our customer service for more information.

Fizz

Rayment and Lyons Method 19D1

Units of Measurement: qualitative only

Fizz result	Reaction to 1M HCl
NL	No effervescence
SL	Slight effervescence
MD	Moderate effervescence
HI	Strong effervescence
VH	Violent effervescence

Carbonates in soil can be identified by effervescence when introduced to the presence of hydrochloric acid (HCl), which dissolves carbonates present and releases CO₂ gas. The extent of the CO₂ gas is characterised to give the Fizz result. This test is purely qualitative and is a subjective physical characterisation of the soil.

Heavy Metals (Cadmium, Molybdenum, Cobalt, Selenium, Lead, Chromium, Arsenic)

Rayment and Lyons Method 17B2

Units of Measurement: µg/kg

Hydrochloric and nitric acid mixture (Aqua Regia) is added to dry soil sample and heated until completely digested. Digests are then read by inductively coupled plasma (ICP) spectroscopy. This method determines total trace elements present in soil.

McQuaker, NR, Brown, DF and Kluckner, PD (1979) Anal. Chem. 51, 1082. AOAC Official Methods of Analysis 15 Ed. 975.03.

Moisture

Rayment and Lyons Method 6G1

Units of Measurement: %

Unprepared soil sample is weighed and heated in an oven overnight. The sample is weighed a second time, post heating, to determine percentage moisture.

Nitrate Nitrogen and Ammonium Nitrogen

Rayment and Lyons Method 7C2b

Units of Measurement: mg/kg

Soil nitrate nitrogen and ammonium nitrogen are extracted using 2M potassium chloride solution. After dilution of the resulting soil solution, ammonium nitrogen is measured colourimetrically. Nitrate nitrogen is reduced to nitrite through a copperised cadmium column and measured colourimetrically.

Olsen Phosphorus

Rayment and Lyons Method 9C2

Units of Measurement: mg/kg

Soils are extracted using sodium bicarbonate (pH 8.5) at a ratio of 1:20. The orthophosphate ion created reacts with ammonium molybdate and antimony trichloride under acidic conditions to form a phosphomolybdic acid complex, which is reduced to ascorbic acid, which is then measured colourimetrically.

Organic Carbon (Walkley-Black)

Rayment and Lyons Method 6A1

Units of Measurement: %

The Walkley-Black method uses concentrated sulphuric acid and dichromate solution, which are added to soil samples. The chromic ions produced are proportional to the oxidised organic carbon and measured colourimetrically. The heat of the acid based reaction is used to induce oxidation of 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

Particle Size (Wet Chemistry Method)

“Pipette” method

Units of Measurement:

Particle Size Fraction	Physical Size
Clay	<2 µm
Silt	2-20 µm
Fine Sand	20-200 µm
Coarse Sand	200-2000 µm

Prepared soil samples (<2 mm) are treated with hydrogen peroxide to remove organic matter and shaken with a 1:1 Calgon and sodium hydroxide solution to disperse soil particles. Using known particle sedimentation times, aliquots of solution are removed at set times and the remaining sample is sieved by size. Solution from aliquots taken are evaporated and weighed to determine the percentage of soil in the coarse sand, fine sand, silt and clay fractions.

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

Particle Size (Mid Infra-Red Method)

Rayment and Lyons Method 6B4b (derivative of)

Units of Measurement:

Particle Size Fraction	Physical Size
Clay	<2 µm
Silt	2-20 µm
Sand	20-2000 µm

Light from the mid infra-red (MIR) range is focused onto the surface of air-dried soil samples. Some light is absorbed by the soil and the remainder is reflected back to the detector. The wavelengths reflected are correlated to a calibration set of spectra within the laboratory, determined using wet chemistry particle size methods. Particle size

determination using this method is highly dependent on the homogeneity of the soil sampled, due to the small sample size used in this method.

Phosphorus Buffering Index (PBI)

Rayment and Lyons Method 9I2c

Units of Measurement: Phosphorus buffering index

Phosphorus buffering index is measured by the amount of phosphorus sorbed by the soil when the solution concentration of phosphorus is increased by 100 mg/mL. Soil is extracted using a calcium chloride and sodium dihydrogen phosphate solution and the phosphorus sorption is measured colourimetrically using an ammonium molybdate/ammonium metavanadate reagent. Phosphorus buffering index is then calculated using the phosphorus sorption measurement and measurement of Colwell Phosphorus or Olsen. CSBP laboratory offers calculation of phosphorus buffering index using Colwell phosphorus measurement only.

Allen, D.G. and Jeffrey, R.C. (1990). Methods for analysis of phosphorus. *Western Australian Soil Report of Investigation No.37* Chemistry Centre WA, p. 37

Phosphate Retention Index (PRI)

Allen and Jeffrey method (1990)

Units of Measurement: PRI

Phosphate retention index is the ratio of adsorbed phosphorus to the equilibrium concentration. Phosphorus is extracted using a 0.02M solution of potassium chloride, at a ratio of 1:20 and the concentration of phosphorus in the resulting solution is determined colourimetrically.

Allen, D.G. and Jeffrey, R.C. (1990). Methods for analysis of phosphorus. *Western Australian Soil Report of Investigation No.37* Chemistry Centre WA, p. 37

pH (Water), pH (CaCl₂), Electrical Conductivity

Rayment and Lyons Method 4A1 (pH water); AB1 (pH CaCl₂); 3A1 (Conductivity)

Units of Measurement: pH; dS/m

Soils are extracted in deionised water at a ratio of 1:5. Water pH and electrical conductivity of the extract are measured using a pH and conductivity electrode. Calcium chloride is added to the mixture to the equivalent of 0.1M and the calcium chloride pH is measured.

Potassium (Nitric)

Rayment and Lyons Method 18C1

Units of Measurement: mg/kg

Soils are extracted using boiling 1 M Nitric acid and the extract is read for potassium using atomic absorption spectroscopy. This test measures non-exchangeable, available potassium as well as soluble, exchangeable potassium and provides a close estimation to the total concentration of potassium present within the sample. Potassium measured using this method provides a repeatable, consistent result compared to the previous Kjeldahl (Total Potassium) method.

Potassium (Skene)

Rayment and Lyons Method 18B1

Units of Measurement: mg/kg

Skene potassium determines available potassium in the soil and values from skene potassium are typically lower than Colwell Potassium values when soils are alkaline. Soils are extracted using 0.05M hydrochloric acid and the extract is read for potassium using atomic absorption spectroscopy.

Haysom, M.B. (1971). The estimation of potassium availability in Mackay soils. Qld Soc Sugar Cane Technol, 38th Conf p 113-119.

Reactive Iron and Aluminium

Rayment and Lyons Method 13A1

Units of Measurement: mg/kg

Soils are extracted using Tamms reagent (oxalic acid/ammonium oxalate). The concentration of iron is determined using atomic absorption spectroscopy and aluminium is measured using inductively coupled plasma (ICP) spectroscopy.

Tamm, O. (1922) Medd. Skogforsoksanst, 19,1-20

Sulphur (KCI 40)

Rayment and Lyons Method 10D1

Units of Measurement: mg/kg

Plant available sulphur in soil is determined by extracting soil using a 0.25M potassium chloride solution. The sulphur content of extracts are then analysed by inductively coupled plasma spectroscopy. Also known as the Blair/Lefroy Extractable Sulphur method.

Blair, G., Chinoim, N., Lefroy, R., Anderson, G. & Crocker, G. (1991). *Aust J Soil Res* 29: 619-626.

Sulphur MCP

Rayment and Lyons Method 10B3

Units of Measurement: mg/kg

Sulphate Sulphur is extract from soil using a solution of mono calcium phosphate (MCP) with sufficient phosphate ions to displace adsorbed sulphur. Soil are extracted in MCP solution overnight and is measured for adsorbed sulphur and water soluble sulphur using inductively coupled plasma (ICP) spectroscopy

Texture

Units of measurement:

Texture code	Texture category
1.0	Sand
1.5	Sand/Loam
2.0	Loam

2.5	Loam/clay
3.0	Clay
3.5	Very heavy clay

Texture is assessed by wetting soil samples with deionised water. The texture category of the soil is determined by the technician based on the amount of stretch of the soil when rubbed against the fingers. Texture is a subjective physical characterisation of the soil.

Total Carbon

Rayment and Lyons Method 6B2

Units of Measurement: %

Soil samples analysed for total nitrogen using the Dumas high temperature combustion (Leco analyser). Samples are loaded into a combustion tube at 1350 °C and flushed with oxygen. Gases generated from this process are measured using an infra-red detector for carbon.

Total Nitrogen

Rayment and Lyons Method 6B1

Units of Measurement: %

Soil samples analysed for total nitrogen using the Dumas high temperature combustion (Leco analyser). Samples are loaded into a combustion tube at 950 °C and flushed with oxygen. Gases generated from this process are measured using a Thermal conductivity cell for nitrogen.

Total Organic Carbon

Rayment and Lyons Method 6B1

Units of Measurement: %

Concentrated sulphuric acid is added to soil wetted with dichromate solution and heated. The samples are subsequently diluted, inducing oxidation of soil organic matter. The amount of chromic ions produced is proportional to the organic carbon oxidised and is measured colourimetrically.

Walkley, A (1947). A critical examination of a rapid method for determining organic carbon in soils – effect of variations in digestion conditions and of inorganic soil constituents. *Soil Science*. 63, 251-64.

Total Organic Matter (Loss on Ignition)

Rayment and Lyons Method 6G1

Units of Measurement: %

Unprepared soil sample is weighed and heated in an oven overnight. The sample is weighed a second time, post heating, to determine percentage moisture. Soils are then heated to 400 °C to determine loss of sample on ignition. Loss on ignition measurements are an estimate of organic matter of soil.

Total Phosphorus

Rayment and Lyons Method 9A3b

Units of Measurement: mg/kg

Soils are digested in sulphuric acid in the presence of a BDH Kjeldahl catalyst tablet. The total phosphorus concentration is measured colorimetrically after incubation with colouring reagent, ammonium molybdate/potassium antimony tartrate in an acid medium.

Allen, D.G. and Jeffery, R.C. (1990). *Report of the Investigation No: 37 'Methods for Analysis of Phosphorus in Western Australian Soils'*, p. 7.

Trace Elements (DTPA: Copper, Zinc, Manganese, Iron)

Rayment and Lyons Method 12A1

Units of Measurement: mg/kg

Soils are extracted with diethylene-triamine-penta-acetic acid (DTPA) solution (ratio of 1:2) for 2 hours and the concentration of copper, zinc, manganese and iron is measured using atomic absorption spectroscopy.

Trace Elements (EDTA: Copper, Zinc, Manganese, Iron)

Rayment and Lyons Method 12B1

Units of Measurement: mg/kg

Soils are extracted using ethylenediamine tetra-acetic acid di-sodium salt (EDTA) solution (ratio of 1:5) for 1 hour and the concentration of copper, zinc, manganese and iron is measured using atomic absorption spectroscopy.

Zinc (HCl)

Rayment and Lyons Method 12D1

Units of Measurement: mg/kg

Soils are extracted using 0.1M hydrochloric acid (HCl) (ratio of 1:10) for 1 hour and read using 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 acid-extractable Zn and titratable alkalinity values. *Soil Science*, vol 88, 275-283

Plant Testing Methods

Plant Sample Preparation

Plant samples are dried at 40 °C and ground and sieved to 2 mm.

Chloride and Nitrate

Units of Measurement: mg/kg

Plant Nitrate and Chloride are extracted using deionised water. The extracted nitrate is reduced to nitrite using a copperised cadmium column and measured colourimetrically. The chloride measurement is performed using a reaction which liberates thiocyanate ions from mercuric thiocyanate by formation of soluble mercuric chloride. In the presence of ferric ion free thiocyanate ion forms ferric thiocyanate which is read colourimetrically.

Heavy Metals (Cadmium, Molybdenum, Cobalt, Selenium, Lead, Chromium, Arsenic, Nickel)

McQuaker 1979

Units of Measurement: mg/kg

Hydrogen peroxide and nitric acid mixture (Aqua Regia) is added to dry plant sample and heated until completely digested. Digests are the read by inductively coupled plasma (ICP) spectroscopy. This method determines total heavy metal elements present within the plant.

McQuaker, NR, Brown, DF and Kluckner, PD (1979) Anal. Chem. 51, 1082. AOAC Official Methods of Analysis 15 Ed. 975.03.

ICP Test (Phosphorus, Potassium, Sulphur, Copper, Zinc, Manganese, Calcium, Magnesium, Sodium, Iron, Boron)

McQuaker 1979

Units of Measurement: mg/kg

Hydrogen peroxide and nitric acid mixture is added to dry plant sample and heated until completely digested. Digests are the read by inductively coupled plasma (ICP) spectroscopy. This method determines total trace elements present within the plant.

McQuaker, NR, Brown, DF and Kluckner, PD (1979) Anal. Chem. 51, 1082. AOAC Official Methods of Analysis 15 Ed. 975.03.

Total Carbon

Rayment and Lyons Method 9G2

Units of Measurement: %

Plant samples analysed for total nitrogen using the Dumas high temperature combustion (Leco analyser). Samples are loaded into a combustion tube at 950 °C and flushed with oxygen. Gases generated from this process are measured using a Thermal conductivity cell for nitrogen.

Total Nitrogen

Rayment and Lyons Method 9G2

Units of Measurement: %

Plant samples analysed for total nitrogen using the Dumas high temperature combustion (Leco analyser). Samples are loaded into a combustion tube at 950 °C and flushed with oxygen. Gases generated from this process are measured using a Thermal conductivity cell for nitrogen.

Water Testing Methods

Carbonate/bicarbonate

Units of Measurement: mg/L

The water sample is titrated with hydrochloric acid to determine the concentration of carbonates.

Chloride

Units of Measurement: mg/L

Chloride concentration in the water sample is determined using inductively couple plasma (ICP) spectroscopy.

ICP/Trace Elements Test (Phosphorus, Potassium, Sulphur, Copper, Zinc, Manganese, Calcium, Magnesium, Sodium, Iron, Boron)

Units of Measurement: mg/L

Trace elements within water samples are measured using inductively coupled plasma (ICP) spectroscopy.

Nitrate Nitrogen and Ammonium Nitrogen

Units of Measurement: mg/L

The ammonium nitrogen within the water sample is measured colourimetrically. Nitrate nitrogen is reduced to nitrite through a copperised cadmium column and measured colourimetrically.