



CSBP Lab Methods

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METHODS

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METHODS

1 Soil Tests

1.1 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 from the extract through freezing and centrifugation, which is then analysed for aluminium using inductively couple 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.

1.2 Boron

Rayment and Lyons Method 12C2

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.

1.3 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 the large amount of carbon dioxide produced.

1.4 Chloride

Rayment and Lyons Method 5A2b

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.

1.5 Cobalt

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 in a closed system until completely digested. Digests are then read by inductively coupled plasma mass spectroscopy (ICP-MS). 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.

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1.6 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.

1.7 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^+ .

1.8 Exchangeable Cations in Water - Calcium, Magnesium, Sodium, Potassium

Rayment and Lyons Method 5A4 (derivative of)

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.

1.9 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 salts mask the exchangeable 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.

1.10 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 Lab 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.

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1.11 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.

1.12 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 in a closed system until completely digested. Digests are then read by inductively coupled plasma mass spectroscopy (ICP-MS). 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.

1.13 Iron and Aluminium (Reactive, Oxalate)

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

1.14 Moisture

Rayment and Lyons Method 2A1

Units of Measurement: %

Unprepared soil sample is weighed and heated in an oven overnight at 105°C. The sample is weighed a second time, post heating, to determine percentage moisture.

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1.15 Molybdenum

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 in a closed system until completely digested. Digests are then read by inductively coupled plasma mass spectroscopy (ICP-MS). 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.

1.16 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.

1.17 Organic Carbon (Walkley-Black)

Rayment and Lyons Method 6A1

Units of Measurement: %

The Walkley-Black method uses concentrated sulfuric 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

1.18 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

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1.19 Particle Size (Mid Infra-Red Method)

In-house method

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.

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

Rayment and Lyons Method 4A1 (pH water); 4B41 (pH CaCl₂); 3A1 (Conductivity)

Units of Measurement: pH; dS/m

Soils are extracted in deionised water at a ratio of 1:5, stirring for one hour. 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.

1.21 pH (Buffer - Mehlich single buffer for lime requirement)

Rayment and Lyons Method 16C1

Units of Measurement: pH units

Samples are analysed for pH with water at a 1:1 volume ratio after stirring for 30 minutes. A further equal volume of Mehlich buffer solution is added to the sample, which is then stirred for another 60 minutes and analysed for pH. This value is expressed as the Buffer pH. Aitken et al(1995) have correlated the Mehlich single buffer pH value to lime requirement as per the table below.

Mehlich Buffer pH	Lime Requirement (t/ha 10 cm)	
	To pHw 5.5	To pHw 6.5
4.5	8.6	18
5.0	5.0	11
5.5	2.2	5.7
6.0	0.5	2.3
6.5	<0.5	0.5

1.22 Phosphorus and Potassium (Colwell)

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.

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1.23 Phosphorus (Bray I)

Rayment and Lyons Method 9E2

Units of Measurement: mg/kg

Soils are extracted using ammonium fluoride and dilute hydrochloric acid, pH adjusted to 3.0 at a soil:solution ratio of 1:7 for one minute. The soil extract is then treated with ammonium molybdate/potassium antimony trichloride reagent and phosphorus concentration is measured colourimetrically. This method differs from Bray P II in that it uses a less concentrated extracting solution and will typically give lower results.

1.24 Phosphorus (Bray II)

Rayment and Lyons Method 9E2

Units of Measurement: mg/kg

Soils are extracted using ammonium fluoride and dilute hydrochloric acid, at a soil:solution ratio of 1:7 for forty seconds. The soil extract is then treated with ammonium molybdate/potassium antimony trichloride reagent and phosphorus concentration is measured colourimetrically. This method differs from Bray P I in that it uses a more concentrated extracting solution and will typically give higher results.

1.25 Phosphorus (BSES, Acid)

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 sulfuric acid and the phosphorus in the extract is determined colourimetrically.

1.26 Phosphorus (DGT)

Units of Measurement: ug/L

Soil samples are taken to water holding capacity using deionised water, before a diffuse gradient thin-film (DGT) device containing a filter and two gels makes contact with the soil surface for a minimum of six hours. The second gel at the back of the device contains ferrihydrite, which traps phosphate. The devices are then pulled apart and the ferrihydrite gel containing the phosphate is digested using hydrochloric acid and the resulting extract analysis colourimetrically.

Mason S (2012). DGT Commercial Protocol (2) – Deployment and analysis. The University of Adelaide

1.27 Phosphorus (Olsen)

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.

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1.28 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 by default, but can also offer an Olsen phosphorus adjusted or unadjusted PBI result upon request.

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

1.29 Phosphorus Retention Index (PRI)

Allen and Jeffrey method (1990)

Units of Measurement: PRI

Phosphorus 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. This value is then used to calculate the phosphorus retention index.

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

1.30 Phosphorus Total (Kjeldahl)

Rayment and Lyons Method 9A3b

Units of Measurement: mg/kg

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

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

1.31 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.

1.32 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.

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1.33 Potentially Mineralisable Nitrogen (PMN)

Rayment and Lyons Method 7D2

Units of Measurement: (mg/kg)

Soil samples are sieved moist and a sub-sample is analysed immediately for 2M KCl extractable ammonium, while another sub-sample is incubated at 40°C with 10ml deionised water for seven days. Following incubation, concentrated KCl solution is added to the wet sample to bring to the equivalent of 2M KCl and extractable ammonium is measured from the incubated sub-sample. The difference in ammonium concentration between the sample before and after incubation is reported as Potentially Mineralisable Nitrogen. This is expressed on an oven-dry basis, so the Moisture test is included with this test.

1.34 Saturated Paste - Electrical Conductivity

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.

1.35 Saturated Paste - Electrical Conductivity and Exchangeable Cations

Rayment and Lyons Method 14B1, 14H1

Units of Measurement: dS/m; meq/L

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.

1.36 Saturated Paste - Electrical Conductivity and Exchangeable Cations and Carbonates

Rayment and Lyons Method 14B1, 14H1, 19A1

Units of Measurement: dS/m; meq/L

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 against hydrochloric acid to determine the concentration of carbonates.

1.37 Selenium

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 in a closed system until completely digested. Digests are then read by inductively coupled plasma mass spectroscopy (ICP-MS). 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.

1.38 Silicon (CaCl₂)

Units of Measurement: mg/kg

Plant available silicon is extracted from the soil by adding 0.01M calcium chloride and tumbling samples end on end overnight. The extract is then centrifuged and analysed colourimetrically at 660nm.

Page et al., Methods of soil analysis 2nd edition, part 2 p265-271

METHODS

1.39 Sulfur (KCl 40)

Rayment and Lyons Method 10D1

Units of Measurement: mg/kg

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

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

1.40 Sulfur (MCP)

Rayment and Lyons Method 10B3

Units of Measurement: mg/kg

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

1.41 Texture (in-house method)

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.

1.42 Texture – Hand Bolus

Texture - Hand Bolus is a measure of the behaviour of a small handful of soil when moist and kneaded into a ball and then pressed out between thumb and forefinger. The texture reflects the proportion of sand (2 – 0.02mm), silt (0.02 – 0.002 mm) and clay (<0.002mm) in soil.

Texture Grades: Sand; Loamy Sand; Clayey Sand; Sandy Loam; Loam; Silty Loam; Sandy Clay Loam; Clay Loam; Silty Clay Loam; Sandy Clay; Light Clay; Medium Clay; Heavy Clay.

McDonald, R.C, Isbell, R.F, Speight, J.G, Walker, J and Hopkins, M.S. (1998). Australian Soil and Land Survey Field Handbook (2nd ed.), Department of Primary Industries and Energy and CSIRO Australia.

1.43 Total Carbon

Rayment and Lyons Method 6B2

Units of Measurement: %

Soil samples analysed for total carbon using the Dumas high temperature combustion method (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.

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1.44 Total Nitrogen

Rayment and Lyons Method 7A5

Units of Measurement: %

Soil samples analysed for total nitrogen using the Dumas high temperature combustion method (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.

1.45 Total Organic Carbon

Rayment and Lyons Method 6B1

Units of Measurement: %

Concentrated sulfuric 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.

Heanes, D.L (1984) Determination of Total Organic-C in Soils by and Improved Chromic Acid Digestion and Spectrophotometric Procedure. Communications in Soil Science and Plant Analysis 15, 1191-1213.

1.46 Total Organic Carbon (acid wash)

Rayment and Lyons Method 6B3

Units of Measurement: %

Samples are washed with sulfurous acid to remove carbonates. The washed samples are then analysed for total carbon using the Dumas high temperature combustion method (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.

1.47 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.

1.48 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.

1.49 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.

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1.50 Water Repellency (MED Droplet Method)

MED Droplet Test

Units of Measurement: Index (0-5)

A few droplets of deionised water are applied to dried, sieved soil and observed. If the water does not penetrate the surface of the soil within 10 seconds, there is a level of water repellence. Higher concentrations of ethanol up to 4M are then applied and observed until the solution penetrates the soil surface within 5 seconds. A water repellency rating is given based on the concentration of ethanol required to penetrate the soil surface from 0-5, where a score of 0 means no ethanol was required and a score of 5 means that the 4M ethanol solution could not penetrate the soil within 5 seconds. The higher the rating, the more water repellent the soil.

King, P.M. (1981) Comparison of Methods for Measuring Severity of Water Repellence of Sandy Soils and Assessment of some Factors that Affect its Measurement. Australian Journal Soil Research 19, 275-285.

1.51 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 atomic absorption spectroscopy. (AAS)

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

2 Soil Calculations

2.1 Calcium:Magnesium Ratio (Ca:Mg ratio)

Rayment and Lyons Method 15M1

The ratio is calculated using exchangeable calcium and magnesium results from Rayment and Lyons Method (15E1).

2.2 Effective Cation Exchange Capacity (ECEC)

Unit of Measurement: meq/100g

The sum of exchangeable cations; calcium, magnesium, potassium, sodium and aluminium from Rayment and Lyons Method (15E1).

2.3 Exchangeable Sodium Percentage (ESP)

Rayment and Lyons Method: 15N1

Unit of Measurement: %

Exchangeable sodium percentage is calculated as follows; $ESP (\%) = [100 \times \text{exchangeable sodium}] / \text{ECEC}$.

The individual cation; calcium, magnesium, potassium, sodium and aluminium can be expressed as a percentage of the CEC.

2.4 Electrochemical Stability Index (ESI)

The ESI is determined by calculating the ratio of the electrical conductivity (EC1:5 dS/m) and the exchangeable sodium percentage (ESP).

METHODS

3 Plant Tests

3.1 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.

3.2 Heavy Metals - Cadmium, Molybdenum, Cobalt, Selenium, Lead, Chromium, Arsenic, Nickel

McQuaker 1979

Units of Measurement: µg/kg

Hydrogen peroxide and nitric acid mixture is added to dry plant sample and heated until completely digested. Digests are then read by inductively coupled plasma mass spectroscopy (ICP-MS). 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.

3.3 ICP Test - Phosphorus, Potassium, Sulfur, Copper, Zinc, Manganese, Calcium, Magnesium, Sodium, Iron, Boron

McQuaker 1979

Units of Measurement: mg/kg for B, Cu, Fe, Mn, Zn. % for P, K, S, Ca, Na.

Hydrogen peroxide and nitric acid mixture is added to dry plant sample and heated until completely digested. Digests are then 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.

3.4 Total Carbon

Rayment and Lyons Method 9G2

Units of Measurement: %

Plant samples are analysed for total carbon using the Dumas high temperature combustion method (Leco analyser). Samples are loaded into a combustion tube at 950°C and flushed with oxygen. Gases generated from this process are measured using an infra-red detector for carbon.

3.5 Total Nitrogen

Rayment and Lyons Method 9G2

Units of Measurement: %

Plant samples are analysed for total nitrogen using the Dumas high temperature combustion method (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.

METHODS

4 Compost, Mulch and Soil Conditioner Tests

4.1 Moisture

Rayment and Lyons Method 2A1

AS4454-2012 Appendix I

Units of Measurement: %

Unprepared sample is weighed and heated in an oven overnight at 105°C. The sample is weighed a second time, post heating, to determine percentage moisture.

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

Rayment and Lyons Method 4A1 (pH water); 4B41 (pH CaCl₂); 3A1 (Conductivity)

Units of Measurement: pH; dS/m

Samples are extracted in deionised water at a ratio of 1:5, stirring for one hour. 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.

4.3 Saturated Paste - Available Nutrients (Phosphorus, Potassium, Sulfur, Copper, Zinc, Manganese, Calcium, Magnesium, Sodium, Iron, Boron), and pH and Electrical Conductivity

Rayment and Lyons Method 14A4,14F1, 14B1, 14C1,

Units of Measurement: mg/kg for Ammonium N, Nitrate N, B, Ca, Cu, Fe, K, Mg, Mn, Na, P, S and Zn. pH units for pH and dS/m for EC.

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 Boron, Calcium, Copper, Iron, Potassium, Magnesium, Manganese, Sodium, Phosphorus, Sulfur and Zinc using inductively coupled plasma (ICP) spectroscopy. The same supernatant is also used to measure EC and pH by probe and measured colourimetrically for ammonium N. Nitrate nitrogen is reduced to nitrite through a copperised cadmium column and also measured colourimetrically.

4.4 Soluble Nutrients (NH₄-N, NO₃-N, PO₄), pH and Electrical Conductivity – AS4454

AS4454-2012 Appendix B

Units of Measurement: dS/m for EC, pH units, mg/kg for Ammonium N, Nitrate N and Orthophosphate P

Samples are extracted in deionised water at a ratio of 1:5 and tumbled end on end for 90 minutes. The pH and electrical conductivity of the extract are measured using a pH and conductivity electrode. The sample extract is then taken and measured colourimetrically for ammonium N and orthophosphate. Nitrate nitrogen is reduced to nitrite through a copperised cadmium column and measured colourimetrically.

4.5 Total Carbon

Rayment and Lyons Method 6B2

Units of Measurement: %

Samples are analysed for total carbon using the Dumas high temperature combustion method (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.

METHODS

4.6 Total Nitrogen

Rayment and Lyons Method 7A5

Units of Measurement: %

Samples are analysed for total nitrogen using the Dumas high temperature combustion method (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.

4.7 Total Nutrients (Phosphorus, Potassium, Sulfur, Copper, Zinc, Manganese, Calcium, Magnesium, Sodium, Iron, Boron)

AS4454-2012 Appendix D; US EPA method 3051A.

Units of Measurement: mg/kg for B, Cu, Fe, Mn, Zn. % for P, K, S, Ca, Na.

Nitric acid and hydrogen peroxide is added to dried sample and microwave heated until completely digested. Digests are the read by inductively coupled plasma (ICP) spectroscopy. This method determines total trace elements present within the sample.

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

4.8 Total Organic Carbon (acid wash)

Rayment and Lyons Method 6B3

Units of Measurement: %

Samples are washed with sulfurous acid to remove carbonates. The washed samples are then analysed for total carbon using the Dumas high temperature combustion method (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.

METHODS

5 Gypsum and Lime Tests

5.1 Gypsum Purity

Acid Decomposition method- VGB Powertech 2008

Units of measurement: %

Gypsum samples are dried at 40°C, then ground to pass through a 0.6mm sieve. Gypsum samples are heated to boiling with concentrated nitric acid and hydrochloric acid. Samples are then analysed using Inductively Coupled Plasma (ICP) Spectroscopy for total Sulfur and Calcium content. Gypsum purity % is then calculated based on the presence of Sulfur and Calcium compared to pure gypsum.

VGB Powertech eV (2008) Analysis of FGD Gypsum 2nd edition.

5.2 Gypsum Chloride Content

Rayment and Lyons Method 5A2b

Units of Measurement: mg/kg

Water soluble chloride in gypsum is determined using a 1:5 sample:water extraction. Chloride concentration in the resulting extract is determined colourimetrically. The soluble chloride content is a good estimator of salinity within Gypsum, as most chloride would be present as sodium chloride in a gypsum sample.

5.3 Lime Neutralising Value (Bulk)

AOAC Official Method 955.01

Units of measurement: %

Lime samples are dried, ground and then digested in boiling hydrochloric acid. Total Calcium, Magnesium and Sodium are then determined by Inductively Coupled Plasma (ICP) Spectroscopy using this digest. The Neutralising Value is determined by titrating the acidified extract against sodium hydroxide until the solution reaches pH 7 to determine carbonate concentration. The result is expressed as Neutralising Value % as compared to pure calcium carbonate.

Association of Official Analytical Chemists (AOAC) Method 955.01, Neutralising Value for liming material.

5.4 Lime Neutralising Value (Weighted average)

AOAC Official Method 955.01

Units of measurement: %

This test is identical to the Lime Neutralising Value (bulk) test, except a pre-sieving stage is implemented and each particle size range is analysed and a weighted average calculation is taken. The Neutralising Value (NV) of the following particle size ranges: 0-0.125mm, 0.125-0.25mm, 0.25-0.5mm, 0.5-1mm and >1mm is reported. This sieving is performed dry. The Effective Neutralising Value is also provided based on values from Cregan et al. (1989), which takes into consideration the reduced capacity of larger particles to alter pH in the short term. In this calculation, the NV of particles under 0.5mm are not discounted, the NV of particles 0.5-1mm are discounted by 50% and the NV of particles greater than 1mm are discounted by 80%.

Association of Official Analytical Chemists (AOAC) Method 955.01, Neutralising Value for liming material.

Cregan PD, Hirth JR, Conyers MK (1989). Amelioration of soil acidity by liming and other amendments. In: Soil Acidity and Plant Growth. AD Robson (Ed.) Academic Press: Sydney, pp. 206-264.

METHODS

6 Water Tests

6.1 Carbonate/bicarbonate

Units of Measurement: mg/L

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

6.2 Chloride

Units of Measurement: mg/L

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

6.3 ICP/Trace Elements - Phosphorus, Potassium, Sulfur, 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.

6.4 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.

6.5 pH and Electrical Conductivity

Units of Measurement: pH units for pH, dS/m for conductivity

The pH and electrical conductivity of water samples are measured directly using a pH probe and conductivity probe.



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CSBP Lab. Extract Value.