



# **Fertilise Wise Product Testing Statement**

**October 2009**



South East Regional Centre for Urban Landcare

## Fertilise Wise Product Testing Statement

Granular, homogeneous fertiliser products being considered for Fertilise Wise endorsement must meet the requirements of the Fertilise Wise Endorsement Criteria. Testing procedures to demonstrate that granular homogeneous fertiliser product(s) meet the Fertilise Wise Endorsement Criteria are detailed in this Fertilise Wise Product Testing Statement, and these specifically address environmental issues associated with fertiliser products. Coated, specially formulated or liquid products may require fit for purpose treatment methods to be applied in addition to, or in place of the generic criteria provided in this document. It is desirable for consistency of testing procedures, but not essential, that testing is undertaken by contractors recommended by SERCUL. Testing is required under the following broad categories;

1. Fertiliser leaching trials<sup>1</sup>
2. Fertiliser and leached soils analysis<sup>2</sup>
3. Synthetic chemical analysis<sup>2</sup>
4. Radiological analysis<sup>3</sup>

### 1. Fertiliser leaching trials

Granular, homogeneous fertiliser products being considered for Fertilise Wise endorsement are required to undergo independent nutrient leaching trials to determine their potential to contaminate ground and surface water with nutrients, particularly Nitrogen and Phosphorus. Leaching trials are required to be undertaken using the fertiliser product according to the methods outlined in this Product Testing Statement to ensure that comparisons between products and against leaching criteria are made consistently. Soils used in the leaching trials also require chemical analysis to determine specific soil characteristics using the standards and procedures outlined.

Fertiliser products being considered for the Fertilise Wise endorsement are required to demonstrate, using the testing procedures stated, that Total Dissolved Phosphorus leaching does not exceed 1g/m<sup>2</sup>/1000mm and that this result is achieved in the absence of effects such as the accumulation of low water solubility Phosphorus in the soil sample, or the binding and retention of Phosphorus with elements such as Aluminum.

Fertiliser products being considered for the Fertilise Wise endorsement are required to demonstrate, using the testing procedures stated, that Total Dissolved Nitrogen leaching does not exceed 25g/m<sup>2</sup>/1000mm.

### Materials

Soil used in the leaching column is to be a Bassendean Sand equivalent with a low P retention and no previous fertiliser applications. The soil is to be collected from the top 10 cm of soil profile, dried at 40°C, and sieved at <2mm. Soil characteristics should approximate the analysis of the soil sample presented in Table 1.

Table 1. Approximate characteristics of the soil to be used in the leaching trial

Tested parameters	Method	Value
Electrical Conductivity (mS/m)	See soil sampling section for analysis methods	<1
pH (H <sub>2</sub> O)		5.5
pH (CaCl <sub>2</sub> )		4.4
Organic Carbon (%)		0.12
Total Nitrogen (%)		<0.005
NH <sub>4</sub> -N (mg/kg)		<1
NO <sub>3</sub> -N (mg/kg)		<1
Total P (mg/kg)		13
Bicarbonate Extractable P (mg/kg)		<2
Phosphorus Retention Index (mL/g)		0.0
Phosphorus Buffering Index (dimensionless)		0.6
Ammonium Oxalate Extractable Al (mg/kg)		<100
Ammonium Oxalate Extractable Fe (mg/kg)		<10

The fertiliser to be tested is sampled directly from the saleable packaging using composite sampling techniques and lightly ground in a mortar and pestle, with a sufficient amount retained to conduct the trial, and for chemistry analysis. Light grinding to breakup the large fertiliser granules is necessary to avoid complications with heterogeneity, and to more accurately apply fertiliser across the surface of soils in leaching columns during the trial at the application rates recommended on the packaging.

Fertiliser is to be applied according to the recommended application rates on the saleable packaging. The trial represents approximately a one year application cycle compacted into a 6 day leaching trial. For example, most product suppliers recommend fertiliser reapplication over a 6 to 10 week cycle, so an 8 week cycle is chosen as an average fertiliser application cycle. Hence each day of the leaching trial represents 8 weeks of a year (6 x 8 weeks = 48 weeks = approx 1 year). Fertilisers are therefore reapplied each day of the leaching trial according to the application rates recommended on the packaging.

### Leaching column setup

Soils are packed into 10 cm deep, 9.3cm diameter leaching columns, with the lightly ground fertiliser applied to the surface of a leaching column, and one column with no fertiliser applied as a control. Three replicates are required. Columns are leached using a peristaltic pump under unsaturated flow conditions.

### Leaching and sampling procedure

Leaching rates are to approximate annual rainfall plus recommended supplementary irrigation of lawns. For the urban areas in and around Perth, this rate is equivalent to 8.4 mm rainfall per hour using de-ionised (DI) water, and equivalent to 1200 mm rainfall and irrigation over the 144 hour duration of a complete leaching cycle occurring over 6 days. Leaching was performed through the use of a peristaltic pump, so that each column is leached at the same rate. Each column receives approximately 57mL of DI water per hour to achieve the required volumes of rainfall plus irrigation. Leachate volumes accumulated in collection vessels are recorded as the first and last activity of the working day. The collected leachate is agitated, and a subsample (30-50 mL) is filtered (<0.45um), the remainder of the leachate discarded, and leaching continued. The filtered samples are stored in a cool room at 4°C. To reduce chemical analysis requirements, leachate can be collected once per day, and then samples composited according to the leachate volume collected each day, or the entire volume of leachate over the 6 day cycle can be collected and subsampled. Care needs to be taken using this latter approach to ensure collected leachate does not degrade in quality as it accumulates. Up to twelve water samples per treatment and replicate can be collected for analysis if the leachate is subsampled twice per day over the 6 day leaching cycle.

### Analyses

Table 2. Summary of methods for leachate analysis

Analyte	Method <sup>1</sup>	Reference <sup>1</sup>
Total Dissolved P	Persulfate method for simultaneous determination of Total Nitrogen and Total Phosphorus	EH2
Total Dissolved N	Persulfate method for simultaneous determination of Total Nitrogen and Total Phosphorus	EH2
Filterable Reactive P	Antimony-phospho-molybdate complex	EH3
NO3	Quantitative reduction of nitrate to nitrite, and the subsequent formation of an azo dye	EH4
NH4	Ammonium nitrogen is measured colourimetrically at 420nm using the indo-phenol blue reaction	EH5
pH		APHA(2005)
EC		APHA(2005)

<sup>1</sup> Analysis techniques and references recommended, and provided by, the Centre for Ecohydrology, Department of Agriculture and Food, Albany, WA.

At the completion of a six day leaching cycle soil is removed from the columns, homogenised, dried, packaged, and labeled in preparation for analysis.

## 2. Soil analysis

Table 3. Summary of methods for soil analysis

Analyte	Method <sup>2</sup>	Reference <sup>2</sup>
Aluminium (Al)	(AmOx), extracted in Ammonium Oxalate	S29
Cation Exchange Capacity (CEC)	(NH <sub>4</sub> Cl), 1M NH <sub>4</sub> Cl pH 7.0	S22.0
Electrical conductivity EC (1:5)	(1:5) at 25 deg C	S02
Iron (Fe)	(AmOx), Fe extracted in Ammonium Oxalate	S29
Total Nitrogen (Total N)	N by Kjeldahl digestion	S10
Ammonium nitrogen N (NH <sub>4</sub> -N NO <sub>3</sub> -N)	Extracted in 1M KCl	S11.0
Nitrate nitrogen N (NO <sub>3</sub> -N NO <sub>3</sub> )	Extracted in 1M KCl	S11.0
Organic Carbon (Org C)	Walkley and Black method	S09
Bicarbonate Extractable Phosphorus (P)	P in 0.5M NaHCO <sub>3</sub> (1:100) by method	S12.0
Total Phosphorus (Total P)	Colourimetry on Kjeldahl digest	S14
Phosphorus Retention Index (PRI)	Allen and Jeffery method (1990)	S15
Phosphorus Buffering Index (PBI)	Burkitt et al (2002) method	PBI1
pH (CaCl <sub>2</sub> )	pH (1:5) in 0.01M CaCl <sub>2</sub>	S03
pH (H <sub>2</sub> O)	pH (1:5) in deionised water	S01

<sup>2</sup> Analysis techniques and references recommended, and provided by, the ChemCentre, Bentley, WA - NATA certified, AS/NZS ISO 9001:2000 (NSCI 6712) and ASPAC accredited laboratory.

## Fertiliser analysis

Fertiliser products being considered for Fertilise Wise endorsement are required to undergo independent chemical analysis to verify stated elemental analysis and heavy metal content using the following standards.

Table 4. Summary of methods for fertiliser analysis

Analyte	Method <sup>2</sup>	Reference <sup>2</sup>
Aluminium (Al)	ICP-AES method	F02
Boron (B)	acid soluble by ICP-AES.	F02
Calcium (Ca)	acid soluble by ICP-AES	F02
Copper (Cu)	acid soluble by ICP-AES.	F02
Iron (Fe)	acid soluble by ICP-AES.	F02
Potassium (K)	acid soluble by ICP-AES.	F02
Magnesium (Mg)	acid soluble by ICP-AES.	F02
Manganese (Mn)	acid soluble by ICP-AES.	F02
Molybdenum (Mo)	acid soluble by ICP-AES.	F02
Nitrogen (N)	combustion method	P3.1
Sodium (Na)	acid soluble by ICP-AES.	F02
Phosphorus (P)	acid soluble by ICP-AES.	F02
Sulphur (S)	acid soluble by ICP-AES.	F02
Zinc (Zn)	acid soluble by ICP-AES.	F02
Water Soluble Phosphorus (WSP)	AOAC Method 960.01	WSP1
Total Phosphorus (Total P)		S14.0
pH (CaCl <sub>2</sub> )	pH meter in 0.01 M CaCl <sub>2</sub>	S03.1
pH (H <sub>2</sub> O)	pH meter in deionised water	S01.1

<sup>2</sup> Analysis techniques and references recommended, and provided by, the ChemCentre, Bentley, WA - NATA certified, AS/NZS ISO 9001:2000 (NSCI 6712) and ASPAC accredited laboratory.

## Heavy metal analysis

Heavy metal content of the assessed product is not to exceed the standards stated in Table 5: Guidelines for maximum levels of heavy metals allowed in end products for sale as manufactured inputs (9.2. Agricultural Inputs – Selection Criteria) of the Australian Organic Standards (March 2006) pp 87.

Table 5: Guidelines for maximum levels of heavy metals allowed in end products for sale as manufactured inputs. Note all other statutory regulations and requirements (eg EPA) may override (ie be more restrictive than) these guidelines (Australian Organic Standards, 2006).

Element (in manures)	mg/kg
Arsenic (As)	20
Cadmium (Cd)	5
Chromium (Cr)	250
Copper (Cu)	375
Lead (Pb)	150
Mercury (Hg)	4
Nickel (Ni)	125
Zinc (Zn)	700

Table 6. Summary of methods for heavy metal analysis in fertilisers

Analyte	Method <sup>2</sup>	Reference <sup>2</sup>
Aluminium (Al)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Arsenic (As)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Boron (B)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Barium (Ba)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Cadmium (Cd)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Cobalt (Co)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Chromium (Cr)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Copper (Cu)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Iron (Fe)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Manganese (Mn)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Molybdenum (Mo)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Nickel (Ni)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Lead (Pb)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Silicon (Si)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Vanadium (V)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP
Zinc (Zn)	Based on USEPA 3051A method (mg/kg)	iMET1SAICP

<sup>2</sup>Analysis techniques and references recommended and provided by the ChemCentre, Bentley, WA - NATA certified, AS/NZS ISO 9001:2000 (NSCI 6712) and ASPAC accredited laboratory.

### 3. Synthetic chemicals

Where fertilisers contain animal or vegetable products, (manure, blood, bone, feathers, straw etc.) manufacturers/distributors of the product being considered for Fertilise Wise endorsement must demonstrate that bagged products are free from Persistent Organic Pollutants (POPs), and other synthetic chemicals such as herbicides, pesticides, fungicides, miticides, wormicides and antibiotics and growth regulators. Testing for synthetic chemicals in the fertiliser product being considered for Fertilise Wise endorsement may be required by SERCUL.

Testing procedures for synthetic chemicals, suitable to assess the product(s) being considered, will be advised by SERCUL where required. General guidelines for what SERCUL considers acceptable materials in a Fertilise Wise endorsed fertiliser product are given in the Australian Organic Standards (March 2006). (9.2. Agricultural Inputs – Selection Criteria).

#### 4. Radiological testing

Fertiliser products being considered for Fertilise Wise endorsement are required to undergo independent radiological testing using the following techniques (with regulatory certifications) and achieve the stated standards;

1. Radionuclide content using Inductively Coupled Plasma Mass Spectrometry (ICPMS) testing for Uranium (<80ppm)<sup>3</sup> and Thorium (<245ppm)<sup>3</sup>
2. Surface gamma radiation dose rate on sample no less than 10kg;  
Gamma levels (<0.5µSv/hr)<sup>3</sup>

<sup>3</sup> Analysis techniques and maximum levels recommended and provided by Radiation Professionals, Maddington, WA.

#### Reference summary of chemical analysis methods

##### APHA (2005)

American Public Health Association (2005) Standard Methods for the examination of Water and Wastewater

<http://www.standardmethods.org/>

##### **EH1: Total Phosphorus and Nitrogen (TP & TN)**

Determined after the samples were digested to convert all phosphorus forms to phosphate and all nitrogen forms to nitrate. Digestion is achieved after autoclaving 5 mL samples at 120°C for 30 minutes in a 1:1 mix of sample and 4% alkaline persulphate solution (Ebina et al. 1983).

All analyses of phosphorus and nitrogen are determined by standard colorimetric methods (APHA 1995) on a segmented flow auto-analyser (Alpkem, Wilsonville, OR, USA). Results are reported as mg units of element per litre.

APHA (1995). "Standard methods for the examination of water and wastewater, 19th edition." Greenberg A.E. (Ed.). American Public Health Association, American Water Works Association and Water Environment Federation. Washington D.C. USA

Ebina, J., T. Tsutsui, et al. (1983). "Simultaneous determination of total nitrogen and total phosphorus in water using peroxodisulphate oxidation." Water Research 17: 1721-1726.

##### **EH2: Total Dissolved Phosphorus and Nitrogen (TDP & TDN)**

Samples are filtered to pass through a 0.45µm nitrocellulose membrane prior to digestion and analysis as for the total phosphorus and nitrogen analysis.

All analyses of phosphorus and nitrogen are determined by standard colorimetric methods (APHA 1995) on a segmented flow auto-analyser (Alpkem, Wilsonville, OR, USA). Results are reported as mg units of element per litre.

APHA (1995). "Standard methods for the examination of water and wastewater, 19th edition." Greenberg A.E. (Ed.). American Public Health Association, American Water Works Association and Water Environment Federation. Washington D.C. USA

### **EH3: Filterable Reactive Phosphate (FRP) H2b**

Samples are filtered to pass a 0.45µm nitrocellulose membrane prior to reaction with acidic molybdate in the presence of antimony. This forms an antimony-phospho-molybdate complex which is reduced by ascorbic acid to an intensely blue complex: phosphomolybdenum blue which is measured spectrophotometrically at 880 nm (Rayment and Higginson 1992).

Rayment, G.E. and Higginson, F.R. (1992). 'Australian Laboratory Handbook of Soil and Water Chemical Methods'. (Inkata Press: Melbourne.).

### **EH4: Nitrate + Nitrite (NOx) 7C1b**

Based on the quantitative reduction of nitrate to nitrite, and the subsequent formation of an azo dye. The reduction is achieved by passing the sample through a copperised cadmium reactor. The pink azo dye is analysed at 540 nm. To determine nitrate only, the reactor is removed so that nitrite can be determined and subtracted from the original NOx determination.

### **EH5: Ammonium (NH4) 7C1a**

The concentration of ammonium nitrogen is measured colourimetrically at 420nm using the indo-phenol blue reaction.

### **F02: Fertilisers and Feeds**

The procedure destroys organic matter and digests mineral components by heating with nitric and perchloric acids. During the procedure nitric acid is evaporated and the solution is made to volume by the addition of water. Quantification of the resulting solutions is by ICP-AES against known standards.

McQuaker, N R, Brown, D F and Kluckner, P D (1979). Anal. Chem. 51,1082-4.

### **ICP-AES**

ICP-AES is an emission spectrophotometric technique, exploiting the fact that excited electrons emit energy at a given wavelength as they return to ground state.

<http://www-odp.tamu.edu/publications/tnotes/tn29/technot2.htm>

### **iMET1SAICP Digestion of Soils, sludges, (including fertilisers)**

Nitric/Hydrochloric digestion by microwave, elemental determination by ICP\_AES  
Based on USEPA 3051A method.

### **P3.1 Total Nitrogen Plants Leco Tru Spec N Analyser Method**

Combustion of the sample in pure oxygen at 850°. Conversion of nitrogen oxides to nitrogen. Removal of water, oxygen and carbon dioxide and measurement of nitrogen by thermal conductivity.

### **PBI1: Phosphorus Buffering Index**

5g of sample extracted with 100P/L on 0.01 CaCl<sub>2</sub> for 17 hours then read on a UV-Vis.

Burkitt LL, Moody PW, Gourley CJP, Hannah MC (2002) A Simple phosphorus buffering index of Australian soils. Australian Journal of Soil Research 40, 497-513.

[www.soilquality.org.au](http://www.soilquality.org.au)

### **S01: pH (H<sub>2</sub>O)**

Measured by pH meter using a glass electrode on a 1:5 extract of soil and deionised water.

Rayment, G.E. and Higginson, F.R. (1992) Soil pH. In "Australian Laboratory Handbook of Soil and Water Chemical Methods". (Inkata Press, Melbourne) 17-18. (Method 4A1).

#### **S01.1: pH(H<sub>2</sub>O)**

Measured by pH meter using a glass electrode, 1% solution (ie 1g in 100mL) for water soluble fertilizers and a 1:20 extract for organic type fertilizers, and deionised water.

**S02: EC (1:5)** Measured by conductivity meter at 25°C on a 1:5 extract of soil and deionised water.

Rayment, G.E. and Higginson, F.R. (1992) Electrical Conductivity. In "Australian Laboratory Handbook of Soil and Water Chemical Methods". (Inkata Press, Melbourne) 15-16. (Method 3A1).

### **S03: pH (CaCl<sub>2</sub>)**

Measured by pH meter using a glass electrode on a 1:5 extract of soil and 0.01 M CaCl<sub>2</sub>.

Rayment, G.E. and Higginson, F.R. (1992) Soil pH. In "Australian Laboratory Handbook of Soil and Water Chemical Methods". (Inkata Press, Melbourne) 19. (Method 4B1).

#### **S03.1: pH (CaCl<sub>2</sub>)**

Measured by pH meter using a glass electrode , 1% solution (ie 1g in 100mL) for water soluble fertilizers and a 1:20 extract for organic type fertilizers, and 0.01 M CaCl<sub>2</sub>.

### **S06: Particle Sizing**

Determined by a modified "plummet" procedure. Soil is dispersed with a solution of "Calgon" - sodium hydroxide, then silt (0.002 - 0.020 mm) and clay (<0.002 mm) are measured by density measurements using a plummet after standard settling times.

Based on:

Loveday, J. (ed) (1974) "Methods for Analysis of Irrigated Soils" Comm. Bureau of Soils., Tech. Communication No 54.

Australian Standard AS 1289.C6.3

### **S09: Organic Carbon**

Organic Carbon is determined, on soil ground to less than 0.15 mm, by Metson's colorimetric modification of the Walkley & Black method. The procedure is based on oxidation of soil organic matter by dichromate in the presence of sulphuric acid. The heat for the reaction is supplied by the heat of dilution of the sulphuric acid with the aqueous dichromate.

Metson A.J. (1956) NZ Soil Bureau Bulletin, 12.

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 constituents. *Soil Sci.* 63, 251-64.

### **S10.0: Total Nitrogen**

Measured by Kjeldahl digestion of soil (Copper sulphate-potassium sulphate catalyst). Total nitrogen is measured as ammonium-N by automated colorimetry by the nitroprusside.dichloro-S-triazine modification (Blakemore *et al.* 1987) of the Berthelot indophenol reaction reviewed by Searle (1984).

Rayment, G.E. and Higginson, F.R. (1992) Nitrogen. In "Australian Laboratory Handbook of Soil and Water Chemical Methods". (Inkata Press, Melbourne) 41-43. (Method 7A2).

Blakemore, L.C., Searle, P.L. and Daly B.K. (1987). Methods for chemical analysis of soils. N.Z. Soil Bur. Sci. Rep. 80.

Searle, P.L. (1984). The Berthelot or indophenol reaction and its use in the analytical chemistry of nitrogen. A review. *Analyst* 109, 549-68.

### **S11: Ammonium and Nitrate**

Extraction from soil by 1M KCl solution and measured by automated colorimetry : ammonium by salicylate - chlorine (Reardon et al 1966) and nitrate by reduction, diazotisation and coupling with N-1-naphthylethylenediamine dihydrochloride (Best 1976). [Reardon J. Foreman J.A. and Searcy R.L. (1966) New reactants for the determination of ammonia. *Clinica Chimica Acta* 14 403-5.]

[Best E.K. 1976. An automated method for the determination of nitrate nitrogen in soil extracts. *Qld. J. Agr. & An. Sc.*, 33 161-166]

### **S12: BIC-P (Plant Available)**

Samples of soil are extracted in 0.5 M sodium bicarbonate solution (pH 8.5) for 16 hours at 23°C using an end-over-end shaking technique. Inorganic phosphorus in the centrifuged extract is measured using automated colorimetry. Orthophosphate in the extract reacts with a reagent containing ammonium molybdate, potassium antimony tartrate, ascorbic acid as reductant and sulphuric acid to form a blue complex ion (Murphy and Riley, 1962).

Bolland, M.D.A., Baker, M.J., Allen, D.G., and Gilkes, R.J. (1987), *W.A. Department of Agriculture, Technical Bulletin No. 10.*

Olsen, S.R., *et. al*, (1954), USDA Circular 939.

Colwell, J.D., (1963), *Aust. J. Exp. Agric. Anim. Husb.*, 3, 190-197.

Murphy, J. and Riley, J.P., (1962), *Anal. Chim. Acta* 27, 31-36. 10.5 Agricultural Chemistry Laboratory, file 7.6.24

Rayment, G.E. and Higginson, F.R. (1992) "Australian Laboratory Handbook of Soil and Water Chemical Methods". (Inkata Press, Melbourne) 66.

### **S14.0: Total Phosphorus**

Measured by colorimetry on the Kjeldahl digest for total N using a modification of the Murphy and Riley (1962) molybdenum blue procedure.

Murphy, J. and Riley, J.P., (1962), *Anal. Chim. Acta*, 27: 31-36.

### **S15: Phosphorus Retention Index (PRI)**

PRI is determined by the method of Allen and Jeffery (1990) and published in Allen et al (2001)

Allen, D.G. and Jeffery, R.C. (1990) Methods for Analysis of Phosphorus in Western Australian Soils. Chemistry Centre (WA) Report of Investigation No 37.

Allen D.G., Barrow, N.J. and Bolland, M.D.A. (2001). Comparing simple methods for measuring phosphate sorption by soils. *Aust. J. Soil Res.*, 39, 1433-1442.

### **S17.1: BIC-K (Plant Available)**

Potassium displaced from soil by dilute salt or acid solutions is considered to be a measure of plant available potassium. In this procedure 0.5 M sodium bicarbonate (pH 8.5) is used as the extracting solution (soil : solution ratio 5:100, 16 hour extraction, 23°C). This procedure is a modification of the standard test for bicarbonate-extractable potassium (soil to solution ratio 1:100). The greater soil to

solution ratio used in this procedure provides improved accuracy and precision for sandy soils containing relatively low concentrations of extractable potassium (<100 mg K/g soil).

Jeffery, R.C., 1982 "Measurement of potassium in 0.5 M NaHCO<sub>3</sub> extracts of soil by flame AAS", Annual Technical Report No. 2, Agricultural Chemistry Laboratory, Government Chemical Laboratories, Perth WA. NATURAL

### **S18.0: Al (CaCl<sub>2</sub>)**

Aluminium is extracted from soil with a 0.01 M CaCl<sub>2</sub> solution. After clarification of the extract, aluminium is measured by reaction with catechol violet. Interference from iron is controlled using 1,10-phenanthroline while interferences from fluoride and phosphate are minimal. The catechol violet method described here is also used in the 0.005 M KCl extractable aluminium method and similarly can be used to measure exchangeable aluminium extracted by 0.01 M BaCl<sub>2</sub>.

Dougan, W.K., and Wilson, A.L., (1974). *Analyst*, 99, 413.

### **S21 – S22: Exchangeable Cations**

Measured by 3 procedures:

a) 1M NH<sub>4</sub>Cl at pH 7.0. - Used for neutral soils (pH (H<sub>2</sub>O) between 6.5 and 8)

Rayment, G.E. and Higginson, F.R. (1992) Ion-exchange Properties. In "Australian Laboratory Handbook of Soil and Water Chemical Methods". (Inkata Press, Melbourne) 138-145. (Method 15A1, 15A2).

Cations (Ca, Mg, Na and K) are measured by ICP-AES

Soluble salts are removed from soils with EC(1:5) >20 mS/m by washing with glycol-ethanol.

b) 0.1 M BaCl<sub>2</sub> (unbuffered) - used for acidic soils only (pH <6.5).

Unpublished Agricultural Chemistry Laboratory procedure based on modifications of :

Gillman, G.P., Skjemstad, J.O. and Bruce, R.C.. (1984) A comparison of methods used in Queensland for determining cation exchange properties. CSIRO Australia, Division of Soils, Technical Paper No 44.

Cations (Ca, Mg, Na, K, Al and Mg) are measured by ICP-AES

Soluble salts are removed from soils with EC(1:5) >20 mS/m by washing with glycol-ethanol.

c) 1 M NH<sub>4</sub>Cl, pH 8.5 used for calcareous soils.

Modification of method of Rayment, G.E. and Higginson, F.R. (1992) Ion-exchange Properties. In "Australian Laboratory Handbook of Soil and Water Chemical Methods". (Inkata Press, Melbourne) 148-154. (Method 15C1).

Cations (Ca, Mg, Na and K) are measured by flame AAS (atomic absorption spectrophotometry).

### **S29: Al & Fe Extracted in Ammonium Oxalate**

Shake, for 2 hours at 23C, 3 g of soil with 100 mL of ammonium oxalate solution. Remove floating organic matter from the extract and centrifuge at 3,500 rpm for 10 minutes. Measure the concentrations of Fe, Al and Mn by either flame AAS or ICP-AES against the standards in ammonium oxalate solution.

Blakemore L.C. Searle P.L. and Daly B.K. (1981) N.Z. Soil Bur. Sci. Rep No 10A.

Rayment, G.E. and Higginson, F.R. (1992) In "Australian Laboratory Handbook of Soil and Water Chemical Methods" (Inkata Press, Melbourne) 121.

#### **S42: Mehlich-3 Extractable Elements**

Soil is extracted with the Mehlich 3 extraction (2.5 g + 25 mL) for 5 minutes, then analysed for extractable P, K, Ca, Mg, Na, S, B, Cd, Co, Cu, Fe, Mn, Mo, Ni and Zn by ICP-AES (Varian Vista axial spectrometer).

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

#### **WSP1 Water Soluble Phosphorus**

AOAC Method 960.01

Association of Official Agricultural Chemists (2009) *Official Methods of Analysis*, 18th edition, <http://www.eoma.aoac.org/>