Proposed Australian Standard: Biochar for Soils – ANZBI Draft for Comment

APPENDICIES - CONTENTS

REV C – September 2019

APPENDIX A: *PAH, PCDD/F AND PCB COMPOUNDS TO BE TESTED*

PAHs, PCDD/Fs, and PCBs are each suites of related chemical compounds (congeners), sometimes numbering in the hundreds. The US EPA maintains a list of 126 Priority Pollutants as part of the (US) Clean Water Act that have been determined to have detrimental human and environmental health impacts; these compounds must be reported under requirements of the Clean Water Act. Contained therein are the primary PAHs and PCBs of concern. For PCDD/Fs, the World Health Organization (WHO) maintains a list of the primary PCDD/Fs of concern as well as the toxic equivalency factor (TEF) of each PCDD/F (Van den Berg et al, 2005).

For the purposes of biochar testing for PAHs, PCDD/Fs, & PCBs, testing labs shall test for the following priority compounds as determined by the US EPA & WHO. The 16 PAH priority compounds to be tested are:

PAH		**CAS number**
1	Acenaphthene	83-32-9
2	Acenaphthylene	208-96-8
3	Anthracene	120-12-7
4	Benz(a)anthracene	56-55-3
5	Benzo(a)pyrene	50-32-8
6	Benzo(b)fluoranthene	205-99-2
7	Benzo(k)fluoranthene	207-08-9
8	Benzo(ghi)perylene	191-24-2
9	Chrysene	218-01-9
10	Dibenz(a,h)anthracene	53-70-3
11	Fluoranthene	206-44-0
12	Fluorene	86-73-7
13	Indeno(1,2,3-cd)pyrene	193-39-5
14	Naphthalene	91-20-3
15	Phenanthrene	85-01-8
16	Pyrene	129-00-0

APPENDIX B: *THE USE OF H:CORG TO INDICATE C STABILITY*

(From IBI *Standardized Product Definition and Product Testing Guidelines for Biochar That Is Used in Soil* (aka *IBI Biochar Standards*), Version 2.1)

The molar H:Corg ratio is recommended to distinguish biochar from other thermochemically altered organic matter for several reasons:

1) H:C ratios change substantially with thermochemical treatment (Keiluweit et al., 2010);
2) O:C ratios have been shown to correlate well with stability of biochars (Spokas, 2010);
3) H:C and O:C ratios are closely related (for low-ash biochars <50% ash and <80% volatiles (ash-free basis));
4) H is determined directly in most laboratories, whereas O is calculated by subtraction.

The modification of using the organic C values rather than total C for this ratio is motivated by the presence of inorganic carbonates in some high-ash biochars. These inorganic carbonates do not form aromatic groups distinctive of biochar materials.

The molar H:Corg ratio is a material property that is correlated with the degree of thermochemical alteration that produces fused aromatic ring structures in the material. The presence of these structures is an intrinsic measure of the stability of the material.

The upper H:Corg limit of 0.7 is used to distinguish biochar from biomass that has not been thermochemically altered and from other materials that have been only partially thermochemically altered. We use the term "thermochemically converted" to refer to thermochemically altered materials that have an H:Corg below 0.7. These materials have a greater proportion of fused aromatic ring structures. Other thermochemically processed materials that have an H:Corg value greater than 0.7 may be thermochemically "altered" but they are not considered to be thermochemically "converted".

Figure A7.1 below shows relationships between processing temperature and H:Corg molar ratio for a number of thermochemically altered materials, as compared to unprocessed biomass.

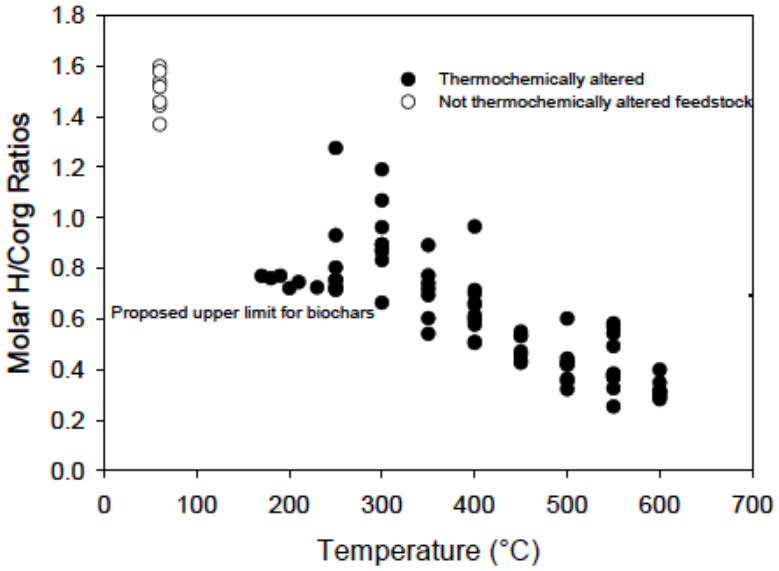

Figure A7.1. Relationship between molar H:C_{org} ratios and temperature of thermochemically altered organic matter in comparison to untreated biomass. The dashed line is the upper limit of 0.7. Data points below the 0.7 line are thermochemically altered materials that are considered to be thermochemically "converted" (data from Sevilla and Fuertes, 2009; Calvelo Pereira et al, 2011; Enders et al., 2012).

References

1. Enders A., Hanley K., Whitman T., Joseph S. and Lehmann J. *Characterization of biochars to evaluate recalcitrance and agronomic performance*. Bioresource Technology 114, 644-653.
2. Keiluweit M., Nico P.S., Johnson M.G. and Kleber M. (2010*) Dynamic molecular structure of plant-derived black carbon (biochar). Environmental Science and Technology* 44:1247- 1253.
3. Sevilla M. and Fuertes A.B. (2009a) *Chemical and structural properties of carbonaceous products obtained by hydrothermal carbonization of saccharides.* Chemistry - A European Journal 15:4195-4203.
4. Sevilla M. and Fuertes A.B. (2009b) *The production of carbon materials by hydrothermal carbonization of cellulose*. Carbon 47:2281–2289.
5. Spokas K.A. (2010) *Review of the stability of biochar in soils: predictability of O:C molar ratios*. Carbon Management 1:289-303.
6. Calvelo Pereira, R., Kaal, J., Camps Arbestain, M., Pardo Lorenzo, R., Aitkenhead, W., Hedley, M., Macías, F., Hindmarsh, J., Maciá-Agulló, J.A. (2011*) Contribution to characterisation of biochar to estimate the labile fraction of carbon. Organic Geochemistry* 42:1331–1342.

APPENDIX C: *TOXICITY AND GERMINATION INHIBITION TESTS*

Toxicity testing of biochar-amended soils is conducted using the Organisation for Economic Cooperation and Development (OECD) earthworm avoidance method (OECD 1984), using the prescribed test species *Eisenia fetida*. Transparent plastic containers (170x120x70 mm) are divided in half with a plastic divider. Amended soil (200 g at field capacity) was placed on one side and unamended soil, of the same soil type, was placed on the other side. The divider was then removed and the container tapped lightly to settle the soil.

Ten clitellate (mature) worms are placed on the soil surface in the centre of each container. Once the worms are observed to enter the soil, the container was covered with a perforated lid. The containers are placed in a controlled-environment chamber at 22°C (±2°C) under constant illumination. After 48 h, the containers are gently removed from the chamber, and the control and test soils separated along the mid-line with a spatula and the plastic divider replaced. Worms found in contact with the centre-line are removed; these individuals are recorded as mid-line. The soils from each side of the container are decanted into separate containers and the worms present in each soil counted.

Germination inhibition study

Germination trays with individual wells are used to test rates of germination of the three plant species for each soil type, with and without biochar. The effect of nutrient addition is not tested in this study. The wells had a small non-absorbent cotton wool plug placed in the bottom, and 15g soil mix added (dry weight equivalent). Soils in the wells are incubated in the glasshouse for 22d prior to addition of the seed. All trays are housed in a single chamber in the controlled environment glass house set at 25/15°C day/ night temperatures. Watering was via a mist irrigation system set to operate for 5s every 45 min. Each well is sown with one seed. In total, there are 110 seeds per plant species/ soil and biochar combination.

Observations of germination are conducted daily between days 2 and 9 following sowing of seed. The proportion of seeds germinated under each soil amendment was calculated and then compared by constructing a logistic regression model for the probability of germination as a function of amendment. The model enabled a statistical test of the hypothesis that germination is not affected by amendment, and calculation of the standard error about each proportion.

APPENDIX D: TCLP TEST

What is TCLP?

Toxicity Characteristic Leaching Procedure (TCLP) is a laboratory analysis process designed to determine the potential mobility of both organic and inorganic compounds present in liquid, solid as well as multiphasic samples. TCLP (USEPA method 1311) was originally developed for assessing solid materials proposed for **landfill** disposal (particularly acidic environments), and is commonly used as a **"worst case" indicator** of the **potential** maximum leachability of solids **particularly in acidic environments**.The two types of leaching reagent solutions used in TCLP methods are typically pH<3 (2.88) or pH<5 (4.93) depending on the character of the original test material. The mobilization of various metals typically increases with increasingly acidic solute conditions (particularly below pH <5.5-5.0), and in some cases (specific metals) also in very alkaline conditions.

TCLP provides an appropriate regulatory framework for conservative (worst case) compliance, however it is noted that **TCLP is NOT** intended as an indicator of **actual expected leachability in all receiving environments (non-landfill), including the application of biochar in most agricultural soils**. Accordingly, it is important that biochar producers understand (and communicate to users) the context of TCLP results, and consider providing additional supplementary information when appropriate which is applicable to the biochar for the intended application to demonstrate it is "fit for purpose". In contrast to TCLP, various biochars may indeed present the opposite effect to leaching, exhibiting **'binding'** capability to soluble metals (organic compounds are well known for their capacity to bind metal ions). Additional complementary testing to demonstrate binding capability and/or TCLP in additional pH buffer solutions (eg at neutral and alkaline pH), or other leaching tests (consult your state EPA for alternatives such as ASLP or LEAF) can also be considered in addition to the minimum required TCLP per USEAPA method 1311.

TCLP Process

Biochar producers should consult with relevant state(s) EPA for leachability testing requirements, including appropriate Limits of Reading (LOR) for laboratory analyses. TCLP analysis as per USEPA Method 1311 begins by determining the amount of solids in the sample. If the sample is a liquid waste that contains ≤0.5% dry solid material, the sample itself is defined as TCLP extract. On the other hand, if the sample contains ≥0.5% dry solid, the liquid portion of the sample is separated from the solid phase and stored for later analysis. The remaining solid phase sample will undergo treatment in order to make sure every particle is <10mm. After that, an appropriate extraction fluid, which is determined by pH testing procedures stated in US EPA method 1311 is used for TCLP extraction. The sample is then extracted with an amount of TCLP extraction fluid equal to 20 times the weight of the solid phase for 16-20 hours on an agitation tumbler.

After extraction, the TCLP extract is separated from the solid phase by filtering through a 0.6 micron filter. Finally, the TCLP extract is combined with any liquid from the initial separation and the sample is ready for analysis of TCLP metals by using ICPMS and FIMS (for Hg only).

***APPENDIX E**: METHOD FOR SAMPLING, SAMPLING HANDLING AND PREPARATION PRIOR TO ANALYSIS*

(Taken from EU guidelines (EBC))

The biochar samples have to be taken following the procedure described here. The accredited controlling inspector is entitled to take samples and sent to the accredited laboratory. To obtain a biochar sample as representative as possible (in terms of accuracy and precision) of a total lot (batch), it must be taken in a proper way. For this, the following general guidelines have to be followed:

- A biochar lot (batch) subject to sampling must consist of at least the amount of one day of production.
- Before sampling, the whole lot has to be thoroughly mixed 3 times by turning and piling it upside-down by means of physical replacement with a front loader or comparable technical device.
- 15 subsamples of 1.5 liter each have than to be arbitrarily gathered from different spots of the homogenized biochar lot (ISO (2006) or Bunge & Bunge (1999)).
- For small scale production of less than 200 liters per day the subsample size may be reduced to 0.5 liters.
- The 15 subsamples have to be united and milled or crushed if the particle size is above 3 mm.
- The new subsample has than to be homogenized thoroughly by turning and piling it 3 times upside-down.
- A further 15 sub-subsamples of 150 mL each have to be arbitrarily taken from different spots of the gathered subsample lot.
- The 15 sub-subsamples (totaling 2.25 L) have to be united and well mixed.
- The sample of 2.25 L has to be sent to an accredited laboratory.

As illustrated in Bucheli et al. (2014), such a sampling procedure my still not be sufficient to obtain truly representative samples, but assures a degree of accuracy (bias) and reproducibility (variance) affordable to compare analytical results with guide values set in this certificate.

Alternatively, an automated incremental cross-stream sample of 100 g could be taken every 30 min for at least 24 hours. Such an automated incremental cross-stream sample could replace the above-described sampling method.

Random Sampling
At each control visit, the controller takes a random sample of the biomass feedstock and the resulting biochar, seals both sample bags and let the producer send them to the certified laboratory.

Retention Sample

In addition to the EBC-analysis sample and random sample, the producers are obliged to take daily an incremental cross-stream sample of at minimum 100g. The time of the daily sample has to be marked in the production protocol. The daily cross-stream samples have to be collected in a monthly sample bag or case. After one month the sample bag has to be sealed and dated. The next 30 cross-stream samples will be collected in a new monthly bag or case. The incremental cross-stream sample can be taken manually or implemented. The incremental cross-stream sampling guaranties a most representative sampling of the product.

References:

Bucheli, T.D., Hilber, I., Schmidt, H.P., (2015). *Polycyclic aromatic hydrocarbons and polychlorinated aromatic compounds in biochar*, in: earthscan, London, U. (Ed.), Biochar for Environmental Management: Science and Technology eds J Lehmann and S Joseph

Thomas Bucheli et al. (2014) *On the heterogeneity of biochar and consequences for its representative sampling*, Journal of Analytical and Applied Pyrolysis 107 (2014) 25–30

APPENDIX F: ANALYTICAL METHODS FROM AS 4454

APPENDIX F(i): METHOD FOR DETERMINATION OF PH, ELECTRICAL CONDUCTIVITY, AMMONIUM, NITRATE AND SOLUBLE PHOSPHORUS CONTENT (AS4454 App B)

(Normative)

B1 GENERAL

This Appendix sets out methods for determining the pH, electrical conductivity, ammonium, nitrate and soluble phosphorus content of an as-received sample aliquot by extraction with deionized water.

NOTE: Tables 3.1 and 3.2 of the Standard (main document) specify compliance requirements.

B2 PRINCIPLE

An aliquot of the sample is shaken with water and the characteristics of the extract are measured.

B3 ANALYTICAL REFERENCES

The following references provide analytical procedures to be conducted on the water extract:

1. ISO 10390:2005, *Soil quality—Determination of pH*.

2. BS EN 13038, *Soil improvers and growing media—Determination of electrical conductivity*.

3. Greenan, N. S., Mulvaney, R.L. and Sims, G.K. *A microscale method for colorimetric determination of urea in soil extracts. Communications in Soil Science and Plant Analysis*. Vol 26, pp 2519-2529, 1995.
4. Page *et al*. *Methods of soil analysis (Part 2) Chemical and microbiological properties* (2nd edn), Soil Science Society of America, Inc, 1982.

5. Rayment, G.E. and Higginson, F.R. *Australian laboratory handbook of soil and water chemical methods*, Melbourne, Inkata Press, 1992.

6. Rayment, G.E. and Lyons, D.J. *Soil Chemical Methods—Australasia*. CSIRO publishing. Collingwood VIC Australia, 2011.

B4 APPARATUS

The following apparatus is required:

(a) Plastic extraction vessel with close-fitting lid. The volume should be sufficient to hold the sample plus five times its mass as volume of extractant, with sufficient airspace for easy shaking.

(b) Mechanical end-over-end shaker, optional.

(c) Filtration equipment, including low-ash fast filter papers.

NOTE: Whatman No. 41 papers have been found to be suitable for fast filtration, and No. 42 for slow filtration.

(d) Centrifuge with a maximum speed of greater than or equal to 3000 r/min.

(e) pH meter, accurate to 0.1 pH unit.

(f) Electrical conductivity (EC) meter, accurate to 0.05 dS/m.

(g) Means of determining the nitrate ion concentration in the extract to accuracy of 5 mg/L N (see Note below).

NOTE: Suggested instruments are an auto-analyser, ion chromatograph, other colorimetric methods or nitrate strips such as the Merkoquant® nitrate test strip and reader system. Nitrate strip readers are convenient for 'in-house' quality assurance testing and may be used provided they are correctly calibrated on each occasion of use. Nitrate ion electrodes have been found to give inaccurate results where soluble organic matter exists.

(h) Means of determining the ammonium ion concentration in the extract to accuracy of 5 mg/L N.

NOTE: Suggested apparatus includes distillation plus titration apparatus, distillation plus a spectrophotometer and colorimetric tests using an auto-analyser. Ammonium analyser

strips may be used to indicate the presence of ammonium ions but they are not acceptable for determining their concentration.

(i) Means of determining the orthophosphate-P concentration in the extract to accuracy of 1 mg/L.

B5 PROCEDURE

The procedure shall be as follows:

(a) Take an aliquot of the specimen for test (SFT) in as-received condition and determine the moisture content by drying to constant mass in an oven at 105°C in accordance with Appendix I.

(b) Determine the pH, EC, Nitrate-nitrogen concentration, Ammonium-nitrogen concentration, and Orthophosphate-P concentration.

(c) Take an aliquot of the SFT in as-received condition that is representative of the product and approximately 200 mL to 2 L in volume depending on the coarseness of the product (see Table A6.2 in Appendix A AS4454).

(d) Weigh between 20 g and 40 g (oven dry equivalent) of the material into the extraction vessel (see Paragraph B4(a)) and record aliquot mass.

(e) Add a mass of deionized or distilled water (see Paragraph B3) that is five times the dry mass of the aliquot.

(f) Seal the vessel and shake by hand to disperse the material through the water. Alternatively, shake on a mechanical end-over-end shaker (see Paragraph B4(b)) rotating at less than 10 r/min, for 90 min.

(g) Determine the pH of the suspension with the pH meter (see Paragraph B4(e).

(h) If filtration will give a separation within a few minutes, filter the solution through a low-ash fast filter. Otherwise, centrifuge the suspension at about 3000 r/min for 5 min and then filter through a low-ash fast filter.

(i) If the filtrate has no discernable turbidity, it can be used as the test solution. Cloudy filtrates should be re-filtered through slow filter paper (see Paragraph B4(c) or centrifuged.

(j) The clear filtrate or centrifugate is the test solution.

(k) Determine the electrical conductivity of the test solution to the nearest 0.05 dS/m with the EC meter.

(l) Determine the nitrate-nitrogen concentration of the test solution to the nearest 5 mg/L using one of the methods referred to in Rayment and Lyons or Rayment and Higginson.

NOTE: For further information see Paragraph B4(g).

Dilute the test solution if necessary, to give a solution with a nitrate ion level in the working range of the instrument. Adjust the measured nitrate ion concentration to compensate for any dilution made to the test solution.

If necessary, calculate the nitrogen concentration present in the test solution as nitrate ions from the following equation:

$N\text{n} = 0.226 \times NO_3$ . . . B5.1 where Nn = nitrogen concentration present in the test solution as nitrate ions, in milligrams of nitrogen per litre NO_3 = nitrate ion concentration in the test solution, in milligrams per litre.

NOTE: Any pink or purple colour present on the upper patch of the nitrate test strip indicates the presence of nitrite ions. Plant operators should be warned that this might indicate anaerobic conditions or immature compost.

(m) Determine the ammonium-nitrogen concentration of the test solution by a standard laboratory procedure listed in Paragraph B3. Calculate the nitrogen concentration present in the test solution as ammonium ions from the following equation:

$N\text{am} = 0.78 \times A$ . . . B5.2 where Nam = nitrogen concentration present in the test solution as ammonium ions, in milligrams of nitrogen per litre A = ammonium ion concentration in the test solution, in milligrams per litre.

(n) If required, determine the orthophosphate-P concentration of the test solution to the nearest 1 mg/L using an appropriate colorimetric method listed in Paragraph B3.

(o) Convert test results from mg/L of test solution to mg/kg dry mass equivalent of the sample using the volume of water extractant (equivalent to volume of deionized water added to the sample plus the moisture in the sample when analysed) and dry mass of the sample analysed (in kg).

B6 TEST REPORT

The test report shall contain the following:

(a) Sample identification, including sufficient details to show the time elapsed between the manufacture and testing of the product.

(b) pH to the nearest 0.1 unit.

(c) Electrical conductivity (EC) to the nearest 0.05 dS/m.

(d) Concentrations of nitrate and ammonium-N in mg/L of test solution, to the nearest 5 mg/L and mg/kg dry mass equivalent, to the nearest 5 mg/kg.

(e) Concentration of orthophosphate-P in mg/L of test solution, to the nearest 1 mg/L, and mg/kg dry mass equivalent, to the nearest 1 mg/kg.

(f) Test method analytical reference used.

(g) Reference to this test method, i.e. Appendix B of AS 4454.

NOTE: 'mg/L in test solution' results obtained using this method may differ from results obtained under the previous extraction method due to the change in the dilution factor. The difference will depend on the density of the sample ('as is' and 'as compacted' for the extraction), as well as the changed dilution ratio (now 1:5 dry mass: mass of deionized water, changed from 1:1.5 compressed volume: volume of deionized water). As composts, soil conditioners and mulches are sold on a volume basis (bulk, in cubic meters; packaged, in litres), the conversion from mg/kg dry mass equivalent to kg/m3 or mg/L will depend on the moisture content of the material and its bulk density.

For example: A compost with 40% moisture content, a bulk density of 0.5 kg/L and a phosphorus content of 5 mg/kg dry mass basis would convert to the following:
Phosphorus content × solids content × bulk density = 5 mg/kg × (1-0.4) × 0.5 kg/L = 1.5 mg/L compost.

APPENDIX F(ii): *METHOD FOR DETERMINATION OF TOTAL CARBON AND NITROGEN CONTENT (AS4454 App C)*

(Normative)

C1 GENERAL

This Appendix sets out methods for determining the organic carbon (and hence organic matter) and total nitrogen contents of a product.

NOTE: Table 3.1(A) specifies the compliance requirements.

C2 PRINCIPLE

A dried and ground whole sample of the product is analysed by means of an instrument such as a carbon/nitrogen furnace or by Wet Chemical methods.

C3 ANALYTICAL REFERENCES

The following references contain suitable methods for the determination of carbon or nitrogen content:

1. ISO 10694:1995, *Soil quality—Determination of organic and total carbon after dry combustion (elementary analysis)*.
2. 2 ISO 11261:1995, *Soil quality—Determination of total nitrogen—Modified Kjeldahl method*.
3. ISO 13878:1998, *Soil quality—Determination of total nitrogen content by dry combustion ("elemental analysis")*.
4. Page *et al*. *Methods of soil analysis (Part 2) Chemical and microbiological properties* (2nd ed) Soil Science Society of America, Inc, 1982.
5. Rayment, G.E. and Higginson, F.R. *Australian laboratory handbook of soil and water chemical methods*, Melbourne Inkata Press, 1992.
6. Rayment, G.E. and Lyons, D.J. *Soil Chemical Methods—Australasia*. CSIRO publishing. Collingwood VIC Australia, 2011.
7. Thompson, W.H. (Ed). *Test Methods for the Examination of Composting & Compost (TMECC)*, The US Composting Council Research & Education Foundation & the U.S Dept of Agriculture, 2001.
8. Walkley, A. *A Critical Examination of a Rapid Method for Determination of Organic Carbon in Soils—Effect of Variations in Digestion Conditions and of Inorganic Soil Constituents*. Vol 63:251-257, 1947.

9. Walkley, A. and Black, I.A. *An Examination of Degtjareff Method for Determining Soil Organic Matter and a Proposed Modification of the Chromic Acid Titration Method, Vol 37:29-37, 1934.*

C4 APPARATUS

The following apparatus is required:

(a) Means of determining carbon (see Note 1 below).

NOTE:

1: A possible method for determining carbon is an Induction Furnace method using an instrument such as a LECO CNS-2000 or equivalent, or by a Wet Chemical method using oxidation and a spectrophotometer or equivalent, such as that described by Rayment and Higginson (1992). Wet Chemical methods are less suitable for products with more than 20% organic carbon, as very small samples need to be taken.

2: Induction Furnace methods measure total carbon (i.e. organic and inorganic carbon), including carbonates. When determining total organic carbon by Induction Furnace methods on alkaline samples, pre-treatment of the sample with acid to remove carbonates is required.

3: Where significant levels of plastic contaminants occur in the product, for example with mixed waste compost, the Wet Chemical oxidation methods (as described by Walkley Black and similar) is considered the only acceptable method.

(b) Means of estimating organic matter.

NOTE: A possible method for estimating organic matter is a Loss on Ignition method such as described by Page et al (1982), suitable for products with more than 20% carbon (about 40% organic matter). As it is assumed that Loss on Ignition = Organic Matter, correction should be made for carbonate content and water of constitution (structural water) as required.

(c) Means of determining total nitrogen.

NOTE: A possible method for determining total nitrogen is an Induction Furnace method, using an instrument such as a LECO CNS-2000® or equivalent, or by a Wet Chemical method, using digestion plus distillation and titration such as the Total nitrogen—semi-

micro Kjeldahl with steam distillation method, as described by Rayment and Higginson (1992) and Rayment and Lyons (2011), or http://www.iso.org/iso/catalogue_detail.htm?csnumber=19239

C5 PROCEDURE

C5.1 Carbon/Organic matter

Determine the carbon in the test aliquot in as-received condition using one of the methods for determination of carbon defined in the method references listed in Paragraph C3, such as Induction Furnace or Wet Chemical. Alternately, estimate the organic matter using a method for determination of organic matter defined in one of the method references listed in Paragraph C3, such as Loss on Ignition using a dried and ground whole sample.

C5.2 Total nitrogen

Determine the total nitrogen in the test sample using an appropriate method, such as Induction Furnace or Wet Chemical (see Paragraph C3), using a dried and ground whole sample.

C6 CALCULATION OF ORGANIC MATTER

Where carbon has been determined by Induction Furnace or Wet Chemical methods, calculate organic matter with one of the following equations:

(a) organic matter = 1.7 × % C (Induction Furnace); . . . C6.1

(b) organic matter = 2.2 × % C (Wet Chemical); or . . . C6.2

(c) by other laboratory derived equations.

NOTE: The relationship between carbon and organic matter varies according to organic matter type and soil type if soil is present in the product. Commonly used conversion factors range from 1.65 to 2.2

C7 TEST REPORT

The test report shall contain the following:

(a) Sample identification, including sufficient details to show the time elapsed between the manufacture and testing of the product.

(b) Total organic carbon content as % dry weight basis.

(c) Organic matter content as % dry weight basis.

(d) Total nitrogen content as % dry weight basis.

(e) The C/N ratio being C/N where C = total organic carbon and N = total nitrogen.

(f) Analytical reference used.

(g) Reference to this test method, i.e. Appendix C of AS 4454

APPENDIX F(iii): *METHOD FOR DETERMINATION OF TOTAL CONTENT OF NUTRIENTS, CONTAMINANT METALS AND OTHER ELEMENTS (AS4454 App D)*

(Normative)

D1 GENERAL

This Appendix discusses and lists appropriate methods for determining the total elemental content of a ground air-dried aliquot of product and the organic contaminants on an as received aliquot. Generally, the methods will be those of oxidizing acid digestion with appropriate analytical finish using flame atomic absorption spectroscopy (AAS) and/or inductively coupled plasma arc spectrometry (ICP). The oxidizing acid digestion methods cited in Paragraph D5 for elemental analysis are generally suitable for the determination of total phosphorus (P), boron (B), calcium (Ca),magnesium (Mg), sodium (Na) as required in **Tables 3.1 and 3.2** of the Standard and for the metallic contaminants required from **Table 3.4** of the Standard (see main document).

The digest may also be used for the determination of the total nutrient contents such as total potassium (K), sulfur (S), iron (Fe), manganese (Mn), molybdenum (Mo), selenium (Se) and iodine (I) as may be required under Appendix P of AS4454.

The methods specified in Paragraph D5 require the use of non-pretreated as-received samples and suffer significant analytical error discussed in Paragraph A7 of AS4454. Ensure that the results for organic contaminants have been corrected and presented on a dry weight basis.

D2 PRINCIPLE—DIGESTION FOR ELEMENTAL ANALYSIS

A representative dried and ground aliquot of the product of a size determined from Table A6.2 of AS4454 is prepared and digested in aqua regia or similar oxidizing acid mixture and the diluted liquor analysed for the required elements.

The most usual analytical technique will be Inductively Coupled-Optical Emission Spectroscopy (ICP-OES) or Inductively Coupled-Mass Spectrometry (ICP-MS) depending on the sensitivity required. Some laboratories may use flame Atomic Absorption Spectroscopy (AAS).

D4 PROCEDURE

Determine the total content of nutrients, contaminant metals and other elements and for the determination of organic contaminants and pathogen indicators in accordance with the relevant test methods specified in paragraph D5**.**

D5 METHOD REFERENCES

D5.1 Metallic and other elements

1 US EPA Method 3050B, Acid digestion of Sediments, Sludges, Soils.
2 US EPA Method 3051, Microwave assisted Acid Digestion/Sludges, Soils.
3 BS EN 13650, Soil Improvers and Growing Media. Extraction of aqua regia soluble elements.
4 ISO 11466:1995, Soil quality—Extraction of trace elements soluble in aqua regia.
5 ISO 16772:2004, Soil quality—Determination of mercury in aqua regia soil extracts with cold-vapour atomic spectrometry or cold-vapour atomic fluorescence spectrometer.
6 ISO 22036:2008, Soil quality—Determination of trace elements in extracts of soil by inductively coupled plasma—atomic emission spectrometry (ICP - AES)

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D6 TEST REPORT

The test report shall contain the following:

(a) Sample identification, including sufficient details to show the time elapsed between the manufacture and testing of the product.

(b) Total nutrient levels in mg/kg dry mass, including the following:

(i) Nitrogen (N).
(ii) Phosphorous (P).
(iii) Potassium (K).

(iv) Carbon (C).
(v) Calcium (Ca).
(vi) Magnesium (Mg).
(vii) Sulfur (S).
(viii) Iron (Fe).
(ix) Manganese (Mn).

(c) Total contaminant metals and other elements, in mg/kg dry mass, including the following:

(i) Arsenic (As).
(ii) Cadmium (Cd).
(iii) Boron (B).
(iv) Chromium (Cr).
(v) Copper (Cu).
(vi) Lead (Pb).
(vii) Mercury (Hg).
(viii) Nickel (Ni).
(ix) Selenium (Se).
(x) Zinc (Zn).
(xi) Molybdenum (Mo).

APPENDIX F(iv): METHOD FOR DETERMINATION OF PARTICLE SIZE GRADING

(Normative)

G1 GENERAL

This Appendix sets out a method for determining the particle size grading of a product and the classification of the product based on this.

NOTE: **Table 3.1** of the Standard (refer main document) specifies the compliance requirements.

G2 PRINCIPLE

Soil conditioners, coarse mulches and fine mulches are assessed using sieves of standard size apertures.

G3 APPARATUS

The following apparatus is required:

(a) Stacking sieve with square apertures of 16 mm.

(b) Stacking sieve with square apertures of 5 mm.

(c) A receiving pan for the sieves stack.

(d) Balance accurate to 0.01 g.

(e) Means of measuring in millimeters.

G4 PROCEDURE

G4.1 Sieve and weigh particle size fractions

The procedure shall be as follows:

(a) Select a representative aliquot of a size determined from Table A6.1, Appendix A, from the bag or batch of product being assessed. Air or oven dry at no higher than 40°C +/- °C.

(b) Determine mass of dry material sample to the nearest 1 g.

(c) Place the entire dry material sample on a sieve stack comprising the 16 mm sieve on top then the 5 mm aperture sieve then the receiving pan on the bottom.

(d) Shake in a horizontal plane for 1 to 2 min until no more material falls through the 5 mm sieve.

(e) Separately weigh material retained by 16 mm sieve (the >16 mm fraction), the material retained by 5 mm sieve (the >5 mm to <16 mm fraction) and the material that has passed through the 5 mm sieve (the <5 mm fraction) to the nearest 1 g and record each of the weights.

(f) Calculate each of the sieved fractions as a percentage (%) by weight of the total dry material sample as follows:
  (i) >16 mm fraction.
  (ii) >5 mm to <16 mm fraction.
  (iii) <5 mm fraction.

G5 TEST REPORT

The test report shall contain the following:

(a) Sample identification, including sufficient details to show the time elapsed between the manufacture and testing of the product.

(b) The product classification by particle size grading and the proportion of each fraction as a percentage of the total dry material sample shall be reported.

(c) Reference to this test method, i.e. Appendix G of AS 4454.

APPENDIX F(v): *METHOD FOR DETERMINATION OF TOTAL CARBONATE CONTENT*

(Normative)

H1 GENERAL

This Appendix sets out a method for determining the total carbonate content of a product using standard reference methods.

NOTES:

1. This method presents the results as if the alkalinity present is present as calcium carbonate and hence, yields approximate values of liming equivalence. This is considered to be accurate
   enough for products covered by this Standard.

2. Table 3.1 of the Standard (refer main document) specifies the compliance requirements.

H2 PRINCIPLE

The product is treated with excess dilute hydrochloric acid and the unreacted acid is titrated with sodium hydroxide.

H3 METHOD ANALYTICAL REFERENCES

The analytical methods that may be used for this test are listed below:

1) ISO 10693:1995, *Soil quality—Determination of carbonate content—Volumetric method*.
2) Rayment, G.E. and Higginson, F.R., *Australian laboratory handbook of soil and water chemical methods*, Melbourne, Inkata Press, 1992. METHOD 19A1, *Carbonates-Rapid Titration*.
3) Association of Official Analytical Chemists (AOAC) Method 955.01, *Neutralizing value for liming material.*

H4 SAMPLE ALIQUOT

Use dried and ground whole sample (see Appendix A, Paragraph A6.3(c)).

H5 TEST REPORT

The test report shall contain the following:

(a) Sample identification, including sufficient details to show the time elapsed between the manufacture and testing of the product.

(b) Percentage of liming materials in the dry product, expressed as % CaCO3 equivalent, to the nearest 1%.

(c) Method analytical reference used for the test.

(d) Reference to this test method, i.e. Appendix H of AS4454.

APPENDIX F(VI): *METHOD FOR THE MEASUREMENT OF A VOLUME OF PACKAGED PRODUCT AND OF THE VOLUME AND BULK DENSITY OF BULK PRODUCT (AS4454 App J)*

(Normative)

J1 GENERAL

This Appendix sets out the method for the measurement of a volume of packaged product and for the measurement of the volume and bulk density of bulk product. This is the method recommended for use in determining quantities at point of sale for both packaged and bulk product.

NOTE: **Clause 3.1** of the Standard (main document) specifies the compliance requirement.

J2 PRINCIPLE

The packaged product is poured into a rigid, calibrated container and the contents levelled. The bulk product is poured into a rigid, calibrated container and dumped to compact the material, which is levelled off to a known, calibrated volume. The test uses product as received.

J3 APPARATUS

J3.1 For packaged product

Rigid, straight-sided pails (see Note) made from translucent plastic, calibrated in litres from the bottom with the following nominal capacities should be used:

(a) For packages of 11 L or more ..... 10 L to 20 L.
NOTE: The contents of the package may require more than one pail (see Paragraph J4.1(c)).
(b) For packages of 6 L to 10 L .....10 L.
(c) For packages of 2 L to 5 L. .....5 L.

NOTE: If necessary, grind off protrusions of plastic on the base of the pail so that it sits flat.

J3.2 For bulk product

For assessing bulk product, the following apparatus should be used:

(a) A rigid, straight-sided container made from translucent plastic, calibrated in litres from the bottom with a minimum capacity of 10 L.
(b) Balance or scales capable of measuring the mass of the sample within 50 g.

J4 PROCEDURE

J4.1 For packaged product

The procedure shall be as follows:

(a) Calibrate the pail by pouring into it water in 2 L increments and clearly marking the incremental 2 L quantities on the inside wall of the pail with a permanent marker. Include a small amount of detergent in the water to allow wetting of the container. Allow the water in the pail to come to rest then mark the container at the surface of the water. Continue adding further water to the nominal volume of the largest packages to be measured. More than one pail may be required.

(b) Choose one package at random from a batch of packages as the specimen for test (SFT). Sit the package upright on the floor. Completely cut off the upper end of the package to expose the contents.

(c) Empty the package loosely into the chosen pail and allow its contents to flow out. Use more than one pail if necessary.

(d) Level off the surface of the product and read the volume to the nearest 1 L.

(e) Add the volume for each pail used if more than one pail was required for a single package of product, calculating the sum to the nearest 1 L.

J4.2 For bulk product

Processors and manufacturers should establish and maintain a program of regular assessment & documentation of bulk density data for their various compost products. As bulk density can vary substantially, especially with change in moisture content, it is recommended that bulk density of finished product be assessed at least weekly. The procedure shall consist of the following steps:

(a) Calibrate a container (with a capacity of at least 10 L for fine products and 20 L for coarse mulch product) using a marked measuring jug or flask to progressively fill the container with water in 1 L increments, clearly marking the incremental 1 L quantities on the container with a permanent marker.

(b) Weigh the empty, calibrated container of known volume on the scales (see Paragraph J3.2(b)).

(c) Fill the container of known volume (see Paragraph J4.2(a)) with a representative sample of the compost or mulch product in as-received condition, until it overflows.

(d) Tamp the container to settle the product by raising the container 5 cm above a flat, level and solid surface (such as a concrete floor) and dropping the full container such that the base lands flat on the solid surface. Repeat this tamping exercise five times.

(e) Remove compost from the top of the container as required so that the container is filled level with a calibration line.

(f) Weigh the full container (i.e. container and representative sample) (*m*f).

(g) Calculate the apparent density of the compost product using the following equation:

$$AD = \frac{Mf - Mc}{V} \quad \text{. . . J4.2(1)}$$

where

AD = apparent density (in kilograms per litre)
*M*f = mass of full bucket (in kilograms)
*M*c = mass of empty bucket (in kilograms)
V = volume in the container (in litres).

Example:

*M*c = 0.45 kg
*M*f = 7.45 kg
V = 10 L
AD = (7.45–0.45)/10
= 0.70 kg/L, or
= 0.70 t/m^3 (since 1000 kg = 1 t; 1000 L = 1 m^3)

APPENDIX G: MEANS OF DEMONSTRATING COMPLIANCE WITH THIS STANDARD (AS4454 App Q)

(Informative)

Q1 SCOPE

This Appendix sets out the following different means by which compliance with this Standard can be demonstrated by the manufacturer or supplier:

(a) Evaluation by means of statistical sampling.
(b) The use of a product certification scheme.
(c) Assurance using the acceptability of the supplier's quality system.
(d) Other such means proposed by the manufacturer or supplier and acceptable to the customer.

Q2 STATISTICAL SAMPLING

Statistical sampling is a procedure which enables decisions to be made about the quality of batches of items after inspecting or testing only a portion of those items. This procedure will only be valid if the sampling plan has been determined on a statistical basis and the following requirements are met:

(a) The sample needs to be drawn randomly from a population of product of known history. The history needs to enable verification that the product was made from known materials at essentially the same time, by essentially the same processes and under essentially the same system of control.

(b) For each different situation, a suitable sampling plan should be defined. A sampling plan for one manufacturer of given capability and product throughput may not be relevant to another manufacturer producing the same items.

In order for statistical sampling to be meaningful to the customer, the manufacturer or supplier needs to demonstrate how the above conditions have been satisfied. Sampling and the establishment of a sampling plan should be carried out in accordance with AS 1199.1, guidance to which is given in AS1199.0.

Q3 PRODUCT CERTIFICATION

The purpose of product certification is to provide independent assurance of the claim by the manufacturer that products comply with the stated Standard. The certification scheme should meet the criteria described in Table 3.1 in that, as well as full type testing from independently sampled

production and subsequent verification of conformance, it requires the manufacturer to maintain effective quality planning to control production.
The certification scheme serves to indicate that the products consistently conform to the requirements of the Standard.

Q4 SUPPLIER'S QUALITY MANAGEMENT SYSTEM

Where the manufacturer or supplier can demonstrate an audited and registered quality management system complying with the requirements of the appropriate or stipulated Australian or international Standard for a supplier's quality management system or systems, this may provide the necessary confidence that the specified requirements will be met. The quality assurance requirements need to be agreed between the customer and supplier and should include a quality or inspection and test plan to ensure product conformity. Information on establishing a quality management system is set out in AS/NZS ISO 9001 and AS/NZS ISO 9004.

Q5 OTHER MEANS OF ASSESSMENT

If the above methods are considered inappropriate, determination of compliance with the requirements of this Standard may be assessed from the results of testing coupled with the manufacturer's guarantee of product conformance. Irrespective of acceptable quality levels (AQLs) or test frequencies, the responsibility remains with the manufacturer or supplier to supply products that conform to the full requirements of the Standard.

Q6 REFERENCED DOCUMENTS

AS:

1199	Sampling procedures and tables for inspection by attributes
1190.0	Part 0: Introduction to the ISO 2859 attribute sampling system
1199.1	Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection

AS/NZS ISO:

9001	Quality management system—Requirements
9004	Quality management system—Guidelines for performance improvements
HB 18	Guidelines for third-party certification and accreditation
HB 18.28	Guide 28—General rules for a model third-party certification system for products