BIDCARBON™

BidCarbon Standard Biochar for Soils

This Standard has in parts been based on the Australian Compost Standard AS 4454-2012, the European Biochar Certificate Guidelines (2019) and the IBI Standardized Product Definition and Product Testing Guidelines for Biochar That Is Used in Soil (2015).

Relevant local, territory, state and federal government regulations not referenced in this Standard, may detail additional requirements. In particular, prior to establishing a manufacturing facility, investigations may be required to ensure approval for location.

The terms "normative" and "informative" have been used in this document to denote the intended application of Annexes: a normative annex is an integral part of a Standard that constitutes requirements of the Standard, whereas an informative annex is only for information and guidance.

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Section 1—Scope and general

1.1. Name of Regulations

This Standard is the BidCarbon Standard Biochar for Soils.

1.2. Commencement

This Standard commences on the day after it is registered.

1.3. Scope

- (1) This Standard specifies requirements for biochar and biochar-based products that are used to amend the physical and chemical properties of natural and artificial soils and growing media.
- (2) It specifies physical, chemical, biological and labelling requirements for lump, powdered, granular, pelleted and liquid products whose carbon content is greater than 30% on a dry weight basis. This limit applies to biochar before they are mixed with other chemicals, biomass or minerals. It covers products marketed or distributed both in bags and bulk in all market sectors including domestic use, urban landscaping, agriculture, land and rehabilitation. It also applies to water remediation where the nutrient rich biochar after filtration is added back to the soil.
- (3) The Standard applies to the biochar that is mixed with compost, manure and sludges, minerals, chemicals and nutrient rich liquids before adding to soils.
- (4) Some biochar have a fertiliser value, and a purpose of the Standard is to enable the consumer to understand the fertiliser value of the material.
- (5) This Standard only covers the properties of biochar's before they are added to the soil. It recognises that the effect on soil properties, seed germination, toxicity, plant resistance to disease, yield and quality is a function of the method, frequency and application rate.
- (6) The Standard also applies to biochar that is used to filter water and air of nutrients and then applied to land. This Standard does not apply to biochar that is added to animal feed, construction materials, metal reductant, road base, an ingredient in food, an activated carbon or used as a fuel.
- (7) This Standard includes all high mineral biomass feedstocks including paper sludge, rice husks, straws and manures. All feedstocks that are used should meet State regulations related to land clearing and logging.
- (8) The carbon composition of the following biochar provides the rationale for the above limit of carbon in biochar that has not been blended with other amendments or fertilisers as 30%. These data are taken from Enders et al 2012, where C is measured using ASTM D1762-84.

1.4. Definitions

In this Instrument:

- (1) **AS 4454** means Australian Standard 4454-2012 Composts, soil conditioners and mulches, Standards Australia, ISBN 978-1-74342-036-2 or any new edition that supersedes the current publication.
- (2) *HJ 892* means Chinese Standard HJ 892-2017, Solid waste—Determination of polycyclic aromatic hydocarbons—High performance liquid chromatography, or any new edition that supersedes the current publication.
- (3) AS means Australian Standard.

- (4) *ISO* followed by a number (for example, ISO 10396:2007) means a standard of that number issued by the International Organization of Standardization and, if a date is included, of that date.
- (5) **ASTM** followed by a number (for example, ASTM D1762-84) means a standard of that number issued by the American Society for Testing and Materials and, if a date is included, of that date.
- (6) Activated Carbon also called activated charcoal is produced from low ash biomass (usually coconut shells and hardwood) at high temperatures to produce a high Carbon material with a high surface area of greater than 600 square meters per gram. Activation can be either be achieved, but not limited too, using steam of carbon dioxide at temperatures greater than 700°C or through treatment with some salts or acids.
- (7) Ash means the inorganic matter, or mineral residue of total solids, that remains when a sample is combusted in the presence of excess air. (Adapted from US Composting Council and US Department of Agriculture, 2001)
- (8) biochar means:
 - (i) (Material) charcoal for application to soil;
 - (ii) (Concept) "charcoal (biomass that has been pyrolysed in a zero or low oxygen environment. The resulting product will have an organic carbon content greater than 30% and a molar H/C ratio below 0.7) for which, owing to its inherent properties, scientific consensus exists that application to soil at a specific site is expected to sustainably sequester carbon and concurrently improve soil functions (under current and future management), while avoiding short- and long-term detrimental effects to the wider environment as well as human and animal health."
- (9) *pyrolysis* means the thermal degradation of biomass in the absence of oxygen leading to the production of condensable vapours, gases and charcoal.
- (10) solid means:
 - (i) (mass noun) the unconsolidated mineral or organic matter on the surface of the earth that has been subjected to and shows effects of genetic and environmental factors of: climate (including water and temperature effects), and macro- and microorganisms, conditioned by relief, acting on parent material over a period of time (ENVASSO, 2008);
 - (ii) (count noun) a spatially explicit body of soil, usually differentiated vertically into layers formed naturally over time, normally one of a specific soil class (in a specified soil classification system) surrounded by soils of other classes or other demarcations like hard rock, a water body or artificial barriers (ENVASSO, 2008)
- (11) Biosolids means solid, semi-solid or slurry material produced by the treatment of sewage.
- (12) **Condensate** means the liquid that either is captured from the flue gas from the pyrolysis process as it is cooled or from directly quenching of the biochar with water.
- (13) *Compost* means an organic product that has undergone controlled aerobic and thermophilic biological transformation through the composting process to achieve pasteurization and reduce phytotoxic compounds which achieves a specified level of maturity required for compost (definition as per AS 4544).
- (14) Contaminant means an undesirable material in a biochar material or biochar feedstock that compromises the quality or usefulness of the biochar or through its presence or concentration causes an adverse effect on the natural environment or impairs human use of the environment (adapted from Canadian Council of Ministers of the Environment, 2005). These can include heavy metals and compounds formed in the pyrolysis process including polyaromatic hydrocarbons, dioxins and furans.

- (15) *Diluent/Dilutant* means Inorganic material that is deliberately mixed or inadvertently comingled with biomass feedstock prior to processing. These materials include soils and common constituents of natural soils such as clays and gravel that may be gathered with biomass or intermixed through prior use of the feedstock biomass (such as for animal bedding). Diluents/dilutants may be found in a diverse range of feedstocks, such as agricultural waste, manures, and municipal solid wastes. It should be noted that they could have other negative plant effects on growth or on the concentration of the heavy metals of the food that is grown with the biochar. Some soil diluents may be beneficial and add extra nutrients and organic matter. Testing is required to confirm any negative or positive effects.
- (16) *agricultural waste* has the meaning given by subsection 6(3) of the Biomass Energy (Small Technology) Specifications.
- (17) *dry matter* has the meaning given by the Fuel Supply Handbook for Biomass-Fired Power Projects. It was published by the World Bank Group on 2 September 2010.

The Fuel Supply Handbook for Biomass-Fired Power Projects website was http://documents.worldbank.org/curated/en/546111468019170010/China-Biomass-Cogeneration-Development-Project-fuel-supply-handbook-for-biomass-fired-power-projects

(18) Organic Carbon or C_{org} means:

Note:

- (1) Carbon in the organic matter of biochar feedstocks. Biochar feedstocks can contain such compounds as sugars, starches, proteins, fats, cellulose, and lignocellulose, which are thermochemically degradable.
- (2) Other organic carbon forms can include petroleum and petroleum by-products such as plastics and contaminated oils, which are, for the purposes of these Standards, included within the definition of contaminants. (Adapted from US Composting Council and US Department of Agriculture, 2001)
- (19) Inorganic Carbon or C_{inorg} means the carbon associated with inorganic C compounds typically calcium and magnesium carbonates (lime, dolomite).
- (20) *Feedstock* means the material undergoing the thermochemical process to create biochar. Feedstock material for biochar consists of biological material but may also contain diluents. (IBI, 2015)
- (21) *Fixed Carbon* means the carbon that remains after heating the biomass or biochar in a covered crucible to approximately 600°C. It should be noted that this test was developed for thermal coals and does not necessarily give an accurate measure of the stability of biochar in the soil.
- (22) *Forestry residues* has the meaning given by the Fuel Supply Handbook for Biomass-Fired Power Projects. It was published by the World Bank Group on 2 September 2010.
- (23) Labile or mineralizable carbon (in biochar) means the carbon that can be either extracted by washing with water or is available as a source of food for microbes.
- (24) *Hydrogen (H)* means the hydrogen content of the biomass or biochar.
- (25) *Minerals* means inorganic compounds derived from biomass or diluents. These inorganic compounds can be either amorphous or crystalline.
- (26) *municipal solid waste* or *MSW* has the same meaning as in the Carbon Farming Standard for Waste and Resource Recovery Data and Reporting.
- (27) *Polycyclic Aromatic Hydrocarbons* or *PAHs* means a family of compounds built from two or more benzene rings. Sources of PAHs include fossil fuels and incomplete combustion of organic matter, in auto engines, incinerators, forest fires, charcoal grilling, or other biomass burning. PAHs are usually found as a mixture containing two or more of these compounds, such as soot. Out of hundreds of different PAH compounds, only a few are

- considered to be highly toxic and of regulatory concern. (Adapted from USGS, 2012). For a detailed description of the type and toxicity of PAH, please refer to Appendix A.
- (28) *Torrefaction* means a thermal pretreatment of biomass that increases energy density, HHV (higher heating value), grindability and hydrophobicity. Temperatures range from 220°C to 350°C, and the time for torrefaction can vary from less than 1 minute to greater than 12 hours.
- (29) **Volatile Matter** means defined as a product, exclusive of moisture, given off by a material as a gas or vapor, determined by prescribed methods that vary according to the nature of the material. (Adapted from Milne et al, 1990).
- (30) *straw* has the meaning given by the Fuel Supply Handbook for Biomass-Fired Power Projects. It was published by the World Bank Group on 2 September 2010.

Note: The Fuel Supply Handbook for Biomass-Fired Power Projects website was http://documents.worldbank.org/curated/en/546111468019170010/China-Biomass-Cogeneration-Development-Project-fuel-supply-handbook-for-biomass-fired-power-projects

(31) **Wood vinegar** means a red-brown liquid formed in the distillation/pyrolysis of wood. It principally contains acetic acid, methanol, acetone and wood oils. It consists of over 200 compounds and is used as a biopesticide and a stimulant for seed germination and plant growth.

1.5. Application

This Standard is intended for use by all levels of government, those who manufacture or supply biochar and biochar-based products.

1.6. Objective

This Standard sets out the requirements for the physical and chemical properties, toxicity testing, Occupational Health and Safety (OHS) properties related to storage, transportation, application, labelling and marking of biochar and biochar-based products. It is intended for use by manufacturers, suppliers, customers and government bodies. The Standard also provides a classification of biochar to help purchasers decide if the biochar or biochar products are fit for purpose. It is recommended that biochar compost products be tested using AS 4454.

Section 2—Specific Requirements

2.1. Product Classification

- This product classification is based on extensive analysis of the literature on the properties and use of the biochar. The principal defining feature is the total carbon and ash content. It should be noted that this is not a hierarchical classification as each type of biochar will have specific applications for which it is fit for purpose. The H/C_{org} ratio is an indicator for the stability of biochar in soil (refer Appendix B). The lower the ratio the greater is the percentage of the biochar remaining after 100 years in the soil.
- (2) Product classification criteria are specified in <u>Table 2.1</u>, with additional requirements given in <u>Table 2.2</u>.

Table 2.1

Table 2.1: Classification Criteria for Biochar

Biochar Classification Type	Carbon Content $(\%C_{org})$	Ash Content (%Ash)	Produced from (examples)	H/C _{org}	Notes
High Carbon low ash biochar (HCB)	>70	<10	wood, bamboo, switch grass and nut shells	0.1 < 0.7	Typically low nutrient content
Medium Carbon medium ash biochar (MCB)	60-70	5-25	straw/stalks, grasses, kernels and cobs, some poultry litter and manures	0.3 – 0.7	Relatively high nutrient content and pH value and may be used as a liming agent.
Low Carbon high ash (LCB)	30-60	>25	sludge, rice husk, some poultry litter and manures, and greenhouse waste	> and/or <0.7	Relatively high nutrient content and pH value and may be used as a liming agent.
Partially Combusted Organic Material (Not biochar)	<30	NA	NA	NA	NA

Note:

All biochar are made at pyrolysis temperatures of between 350 – 750 $^{\circ}$ C. Biomass thermally treated below 350 $^{\circ}$ C will be considered as being torrefied.

- (3) The test methods are taken from AS 4454, IBI classification publication and from McLaughlin¹ on the issues related to measuring moisture content and ash composition. The sampling methods are set out in <u>Appendix E</u>.
- (4) Most biochar are very difficult to ignite unless they are bone dry, however it is recommenced for both OHS and flammability issues that the biochar has a moisture content greater than 10%. For long term storage the biochar should have a moisture content of less than 15% to ensures little chance of the growth of pathogens or self-ignition.

Table 2.2

Table 2.2: Chemical, Physical and Basic Agronomic Testing of Biochar

For high carbon, low ash biochar, there is very little available N and P. Therefore, to reduce testing costs, no measurements are required. It should be noted that any deviation from AS 4454 must be justified by research showing that specific analytical techniques are more appropriate to give accurate measurements. Specific tests methods from AS 4454 are given in Appendix F.

Characteristi c and unit of measuremen t	Units	НСВ	МСВ	LCB	Test Method
Moisture	% of total mass, dry basis	>10%	>10%	>10%	Dry at 150°C as per ASTM D2867 Standard test for moisture for activated carbon ² . This is more appropriate than the moisture test method in the compost standard (AS 4454).
Total Carbon and Hydrogen	%	declare	declare	declare	Total C and H analysis by dry combustion-elemental analyser.
Organic Carbon (C_{org})	%	declare	declare	declare	Total C and H analysis by dry combustion-elemental analyser. Inorganic C analysis by determination of CO ₂ -C content with 1N HCl, as outlined in ASTM D4373 Standard Test Method for Rapid Determination of Carbonate Content of Soils. Organic C calculated as Total C – Inorganic C
H/C _{org}	%	<0.7	<0.7	> or <0.7	Refer Appendix B of the IBI Biochar Standards

Total Ash	%	<10%	<25%	>25%	dry at 150°C as per ASTM D2867 then open crucibl and ash at 550°C
рН		declare	declare	declare	As per AS 4454
Total CaCO ₃ equivalent % dry matter	%	declare	declare	declare	ISO 10693 (1995): Soil Quality - Determination of carbonate content
Electrical Conductivity dS/m	dS/m	declare	declare	declare	As per AS 4454
Available Phosphorous (p)	mg/kg	optional All wood biochar have very little available P	declare	declare	 2% formic acid follow by spectrophotometr (Wang et al. 2012)³ Bray P Extraction (Bra No1 Extract Test)
Total P	mg/kg	optional	declare	declare	Modified dry ashing (Enders and Lehmann 2012) ⁴ . Elements in the digest determined by common analytical techniques
Total Nitrogen (N)	mg/kg	optional	declare	declare	Dry combustion-element analyzer following the same procedure for total and H above.
Available N as nitrate	mg/kg	optional	declare	declare	2M KCl extraction follow by spectrophotometry (Rayment and Lyons 201
Available N as ammonium	mg/kg	optional	declare	declare	2M KCl extraction follow by spectrophotometry (Rayment and Lyons 201
Total potassium	mg/kg	optional	declare	declare	Modified dry ashing (Enders and Lehmann 2012) ⁴ . Elements in the digest determined by common analytical techniques or total elements by ICP-MS

Silica	mg/kg	optional	optional Unless being sold as a high Si amendment	optional Unless being sold as a high Si amendment	NaOH 550°C fusion followed by ICPOES analysis ⁶
Total Calcium Magnesium, Iron and Sulphate-S	mg/kg	declare	declare	declare	1M HCl extraction (Camps Arbestain et al. 2015) ⁷ . Elements in the digest determined by common analytical techniques.
Particle size distribution	%	declare	declare	declare	Progressive dry sieving with 50 mm, 25 mm, 16 mm, 8mm, 4mm, 2 mm, 1 mm, and 0.5 mm sieves.
Surface area	m²/g	optional	optional	optional	BET CO2 adsorption or NMR cryoporosimetry,
Germination Inhibition Assay Worm avoidance test and germination tests	Pass/fail	Pass/fail	Pass/fail	Pass/fail	OECD methodology (1984) using three test species, as described by Van Zwieten et al. (2010)8 or Environment and Climate Change Canada's biological test method. See Appendix C for further information. See Appendix C for further information.

Table 2.3

Table 2.3: Measurement of potential heavy metal toxic elements in biochar using limits and test method in AS 4454 or GB/T 23349

If the heavy metal concentration is higher than recommended Standard in AS 4454 then undertake the Toxicity Characteristic Leaching Procedure (TCLP) (USEPA method 1311, (1992), USEPA, SW-846) (Appendix D). If this is undertaken it is recommended that all parameters required for TCLP are also analysed for Total Concentration to facilitate analysis (proponents should check to ensure such). See further comments in Appendix D regarding potential limitations of TCLP testing and additional beneficial test options available to biochar producers which can be further considered where appropriate.

Parameter	Maximum Allowed Limit (Total Concentration) (mg/kg dry wt)	Test Method
Arsenic	20	GB/T 23349-2020 or AS 4454-2012
Cadmium	1	GB/T 23349-2020 or AS 4454-2012
Chromium	100	GB/T 23349-2020 or AS 4454-2012
Boron	100	AS 4454-2012

Copper	150	AS 4454-2012 or HJ 892
Lead	150	GB/T 23349-2020 or AS 4454-2012
Mercury	1	GB/T 23349-2020 or AS 4454-2012
Selenium	5	AS 4454-2012
Zinc	100	AS 4454-2012 or HJ 892
Boron	Declaration	TMECC (2001)

Table 2.4

Table 2.4: Measurement of potential toxic organic compounds. Testing will be carried out if required by state EPA.

Parameter	Requirement	Units	Test Method/ Requirement
Polycyclic Aromatic Hydrocarbons (PAHs), total (sum of 16 US EPA PAHs)	As per state EPA regulations applied for specific biomass amendments applied to soil	mg/kg (dry wt)	As per state EPA regulations applied for specific biomass amendments
Dioxins/Furans (PCDD/ Fs)	As above	ng/kg WHO-TEQ (dry wt)	As per state EPA regulations applied for specific biomass amendments
Polychlorinated Biphenyls (PCBs)	As above	mg/kg (dry wt)	As per state EPA regulations applied for specific biomass amendments

2.2. Process Criteria

2.2.1. Biochar Heating and Cooling Temperature

- (a) For biochar that are produced in batch kilns, the temperature throughout the feedstock/biochar should not vary by more than 100°C during the heating and cooling cycle to avoid condensation of liquids on the biochar that may contain polyaromatic hydrocarbons.
- (b) Biochar must be cooled to less than 120°C before being exposed to air to prevent any possibility of chemisorption of air and spontaneous combustion.
- (c) Carbonaceous materials (coals compost and charcoal) left at specific temperatures have been known to spontaneously combust (https://en.wikipedia.org/wiki/Spontaneous_combustion). Although research Pagni et al (2002)⁹ has indicated that biochar and charcoal, that is either wet or dry, will not spontaneously combust at temperatures below 80°C it is recommended that solid biochar, that is to be stored in large volumes (>500 m³.), is dry (approximately 15% m.c.) before leaving the pyrolysis kiln. This will minimize the risk of spontaneous combustion. It should also be noted that fungi and bacteria can form on wet biochar that has been exposed to air and alter the properties of the biochar, especially those produced from manures

and sludges which can potentially be a health hazard. There are no known hazards associated with storing biochar that is completely immersed in water.

2.3. Physical and chemical requirements

(1) Products shall comply with the physical and chemical requirements for the classification as set out in Table 2.2 and 2.3.

Note:

For high carbon biochar produced from woody biomass it is optional to give NPK concentrations as they are usually very small. It is recommended that concentrations of Mg, Ca, Si and Fe are given as these can be important for liming effect and for improving strength and nutrient availability of plants. Surface area and pore volume is an important aspect of high carbon biochar and for high carbon woody biochar can be carried out using CO₂ BET method (See IBI characterisation guidelines). For medium and high ash content biochar, the surface area and pore volume measurements may not reflect the true value unless more expensive NMR measurements (Rawal et al. 2016)¹⁰ are carried out.

(2) Toxicity Tests

- (a) All biochar should be tested using worm avoidance and germination tests as specified in Van Zwieten et al 2010. In addition, other plant tests are recommended to be carried out as specified in AS 4454 and as per section 2.3.(2) (these tests are optional).
- (b) Many papers have been prepared on the impact of biochar on the availability of heavy metals and adsorption of PAH. If required by EPA or other authorities it is recommended to use USEPA 1311 Toxicity Characteristic Leaching Procedure (TCLP) test. NATA certified laboratories can undertake this test.

(3) Additional Plant Tests (Optional)

- (a) Previous research has shown that the efficacy of biochar is a function of how much and how often biochar is added to the soil. In particular too much biochar can cause growth inhibition usually due to either locking up of nitrogen and phosphorus (especially in high carbon high temperature biochar) or a salting effect from a high nutrient content biochar where too high a concentration of salts has been transported to the roots of a plant. AS 4454 provides a test for composts to determine a maximum application rate based on the EC value (see Appendix F(i)). To allow consumers to understand the effects of too high an application rate it is recommended that suppliers carry out rate trials similar to that carried out by Yin Chan et al. (2008)¹¹.
- (b) For biochar added to raw materials (whether or not they are dried) for composting and vermicomposting, tests must be carried out in accordance with AS 4544.

Section 3—Verification of requirements and testing

3.1. Product Testing

Storage before testing

(1) Products should be tested as soon as possible after reaching a lab. Samples should be stored in fridge at 4°C. Samples should be dried to 0% moisture content at 40°C over 2 days and the moisture content recorded then kept in sealed containers in the fridge at 4°C until tests carried out.

Physical and Chemical Testing

- (2) Products shall be tested prior to distribution at least once a year or if major process conditions are changed.
- (3) Test methods for the analysis of the properties of the biochar specified in <u>Table 2.1</u> and elsewhere in this Standard shall be in accordance with relevant aspects of Appendices A-F.

Note: Methods for sampling and preparation of samples prior to analysis are given in Appendix E.

Appendix F gives guidance on test methods for assessing the nutrient value of biochar.

Appendix G gives guidance on the determination of compliance with this Standard.

3.2. Claiming Compliance

Documentation shall be retained to demonstrate the product complies with the requirements in Table 2.1, 2.2 and 2.3.

- (1) Manufacturers making a statement of compliance with this Australian Standard (AS) on a product, its packaging, or promotional material related to that product, are advised to ensure that such compliance is capable of being verified.
- (2) Acceptable evidence of compliance with this Standard should include (but is not limited to) the following:
 - (a) Three test reports on samples representative of products produced under normal production conditions.
 - (b) Certification license numbers and certificates.
 - (c) Independent evidence of conformance with the processing, testing and record keeping activities associated with the production of the products claimed to be compliant with this Standard.

Section 4—Packaging marking and documentation

4.1. Packaging

Volume—Packaged materials

- (1) All products shall be packaged in multiples of a litre and the volume shall be measured in accordance with the method in Appendix F(vi) to this Standard, which is consistent with Appendix J of AS 4454.
- (2) When measured in accordance with this method, the actual volume of packaged product shall not be less than the volume stated on the primary package.

Note: Attention is drawn to relevant state or territory and federal regulations governing size and weight requirements that are not within the scope of this Standard.

Volume—Bulk materials

- (3) Details on the determination of volume of bulk product are subject to agreement between the supplier and the purchaser, e.g. loose or settled volume, or weight at a specified moisture content.
- (4) The method for determining volume and bulk density of bulk materials using a small sample (minimum 10 L) is described in <u>Appendix F(vi)</u>. Using the bulk density determined as in <u>Appendix F(vi)</u>, the mass of a load of product, and the volume of product dispatched can be determined.

Note: Parties need to be aware that settling will occur during transport.

Protection

(5) Both packaged and bulk products should be protected so that, under normal conditions of handling, storage and transport, the contents do not become contaminated by extraneous matter (e.g. plant propagules, pathogens and pests) and the contents of packages are not released unintentionally. Packaging shall contain perforations to allow for pressure equalization, and ease and safety of handling.

4.2. Required marking or documentation

(1) Any product supplied in accordance with this Standard shall be accompanied by information supplied either on the primary packaging or, where unavailable, a separate information sheet that is permanently and legibly marked with the information listed below.

Note: In this Standard, the term 'information sheet' can mean an invoice or other

document that provides the information in a location where the consumer is most

likely to read it.

- (2) On packages, the letters shall be no less than 9 point in size and in a prominent position.
- (3) This information shall accompany the product in order to verify compliance with this Standard and include the following:
 - (a) Name or registered trademark and full street address of the manufacturer, packer or distributor.
 - (b) Volume of contents, in litres (to the nearest litre), printed in letters a minimum of 18 point in size. The only statement of volume shall be the volume determined according to the method for the determination of volume in Appendix J of AS 4454.

Note: Packages should **not** be marked with codes such as '**No.1 pack**', or similar, as a substitute for stating the actual number of litres of contents in the package. Mass of contents should not be marked as a substitute for stating the actual volume in litres, although a mass may be provided in addition to volume.

- (c) If the product contains fine particles that could cause respiratory problems or pathogenic micro- organisms then a health warning label and hazardous information label for customers on the handling of the products on the package for bagged product or the information sheet/invoice in the case of bulk products shall be provided. The label must be permanently and legibly marked as follows:
 - (i) Appearance The warning specified in Item (c)(ii) shall be printed in a contrasting colour to other non-warning printing, have a border in bold and shall appear in accordance with Items (A), (B) or (C) as appropriate:
 - (A) On 15 L or larger packages: in 16 point Arial bold or other Sans Serif
 - (B) On packages of less than 15 L: in 12 point Arial bold or other Sans Serif font.
 - (C) On the information sheet/invoice: in 10 point Arial bold or other Sans Serif font.
 - (ii) *Health warning label* The following warning* shall be printed in a prominent position on the front of the package for bagged product or bulk product on the information sheet/invoice.

Example:

Health Warning

- This product contains dust particles
- · Keep Product moist when handling to avoid dust
- Avoid breathing dust- wear particulate mask if dusty
- Wear appropriate gloves and footwear as a precautionary measure
- · Wash hands immediately after use
- Read detailed warning label on this bag and refer to manufacturers Material Safety Data Sheet (MSDS)

4.3. Required marking-conditional upon product claims or test results

- (1) The information sheet or primary package shall be permanently and legibly marked with the following information, depending on the conditional statement for that particular component, written in a prominent position, in letters no less than 9 point in size, set out as follows:
 - (a) Instructions on the maximum application rate for a given soil and plant;
 - (b) If the manufacturer intends that the product is to be used as a nutrient supplement then details of the total and available P and N and total K;
 - (c) If the product total heavy metal content (total concentration) exceeds the relevant state EPA requirements and those listed in <u>table 2.3</u> of the draft Standard but the leachable heavy metal contents are below the EPA requirements this needs to be noted.

Maximum application rate of product with different salinities

(2) <u>Table 2.1</u> sets out maximum application rates of product with different salinities for plants of different sensitivities to salinity.

Table 2.1

Table 2.1: Maximum Application Rate of Biochar Product with Different Salinities for Plants of Different Sensitivities to Salinity

EC range (see Appendix F(i))	Sensitive plants	Tolerant plants
dS/m	L/m ²	L/m ²
0-1	Unlimited	Unlimited
1-2	< 15	< 60
2-4	< 8	< 32
4-8	< 4	< 16
8-12	< 2.5	< 10
> 12	< 2	< 8

Note 1: These rates are for mulches or for incorporation into soil to a depth of 5 cm. When incorporated into the soil to a depth of at least 10 cm these amounts can be doubled.

Note 2: The maximum rate of application of product is to be stated on the primary package or information sheet (as per Clause 4.3(1)(a)), based on the electrical conductivity (EC) result, as in the following example:

The concentration of soluble plant nutrients in this product is such that the maximum rate of application on one occasion should be no more than 4 L per square meter for sensitive plants and no more than 16 L per square meter for tolerant plants. Repeat applications may be made after several weeks.

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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/C_{org} 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 biochar (Spokas, 2010);
- (3) H/C and O/C ratios are closely related (for low-ash biochar <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 biochar. These inorganic carbonates do not form aromatic groups distinctive of biochar materials.

The molar H/C_{org} 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/C_{org} 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/C_{org} below 0.7. These materials have a greater proportion of fused aromatic ring structures. Other thermochemically processed materials that have an H/C_{org} 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.

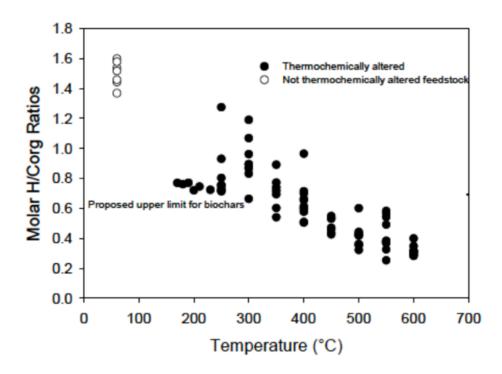


Figure A7.1. Relationship between molar H/C_{org} ratios and temperature of thermochemically altered organic matter in comparison to untreated biomass. The dashed line is the upper limit of 0.7. Data points below the 0.7 line are thermochemically altered materials that are considered to be thermochemically "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 biochar 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

- (1) Toxicity testing of biochar-amended soils is conducted using the Organisation for Economic Cooperation and Development (OECD) <u>earthworm avoidance method</u> (OECD 1984) or or Tests for Measuring Avoidance Behaviour or Reproduction of Earthworms (Eisenia andrei or Dendrodrilus rubidus) Exposed to Contaminants in Soil (Method Development and Applications Unit Science and Technology Branch Environment and Climate Change Canada), 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.
- (2) 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 were counted.

Germination inhibition study

- (3) 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 were incubated in the glasshouse for 22d prior to addition of the seed. All trays were 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.
- (4) 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

(1) What is TCLP?

Toxicity Characteristic Leaching Procedure (TCLP) is designed to determine the mobility of both organic and inorganic compounds present in liquid, solid as well as multiphasic samples. It is a method that simulates what happens when a sample is disposed in a landfill and is utilized to determine whether the contaminated soil meet the EPD landfill disposal guideline.

(2) Why using TCLP analysis?

Since direct disposal of contaminated soil to landfill is not allowed and it must be qualified the Landfill disposal Criteria prior to disposal. TCLP is one of EPD approved method used to assess the soil contamination concentration. If any TCLP test results of the sample exceed the disposal criteria, the contaminated soil must first be pre-treated or stabilized before disposal.

(3) TCLP Process

- (a) The TCLP analysis 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.
- (b) 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).
- (c) The following list is the TCLP regulatory limit and the laboratory accredited LOR.

Parameter (dissolved analyte)	TCLP Regulatory Limit (mg/L)	ALS accredited LOR (mg/L)
Cadmium (Cd)	10	0.01
Chromium (Cr)	50	0.1
Copper (Cu)	250	0.1
Nickel (Ni)	250	0.1
Lead (Pb)	50	0.1
Zinc (Zn)	250	0.1
Mercury (Hg)	1	0.002
Tin (Sn)	250	0.1
Silver (Ag)	50	0.1
Antimony (Sb)	150	0.1
Arsenic (As)	50	0.1

Beryllium (Be)	10	0.1	
Thallium (TI)	50	0.01	
Vanadium (V)	250	0.1	
Selenium (Se)	1	0.02	
Barium (Ba)	1000	0.1	
Others accredited TCLP parameter			
Cobalt (Co)		0.1	
Manganese (Mn)		0.1	
Molybdenum (Mo)		0.1	

Appendix E: Method for sampling, sampling handling and preparation prior to analysis

(Taken from EU guidelines (EBC))

- (1) 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.
- (2) 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.
- (3) 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

(4) 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

(5) 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

(i) 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.

(ii)	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 (AS 4454 App B)

B1 General

(1) 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 <u>2.1</u> and <u>2.2</u> of the Standard (main document) specify compliance requirements.

B2 General

- (2) An aliquot of the sample is shaken with water and the characteristics of the extract are measured. *B3 Analytical References*
- (3) The following references provide analytical procedures to be conducted on the water extract:
 - (i) ISO 10390:2005, Soil quality—Determination of pH.
 - (ii) BS EN 13038, Soil improvers and growing media—Determination of electrical conductivity.
 - (iii) 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.
 - (iv) Page et al. Methods of soil analysis (Part 2) Chemical and microbiological properties (2nd edn), Soil Science Society of America, Inc, 1982.
 - (v) The Australian Laboratory Handbook of Soil and Water Chemical Methods, authored by G.E. Rayment and F.R. Higginson, was published in Melbourne, Victoria, by Inkata Press in 1992. The ISBN is 9780909605681.
 - (vi) Rayment, G.E. and Lyons, D.J. Soil Chemical Methods—Australasia. CSIRO publishing. Collingwood VIC Australia, 2011.

B4 Apparatus

- (4) The following apparatus is required:
 - (i) 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.
 - (ii) Mechanical end-over-end shaker, optional.
 - (iii) 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.

- (iv) Centrifuge with a maximum speed of greater than or equal to 3000 r/min.
- (v) pH meter, accurate to 0.1 pH unit.
- (vi) Electrical conductivity (EC) meter, accurate to 0.05 dS/m.
- (vii) 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.

(viii) 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.

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

B5 Procedure

- (5) The procedure shall be as follows:
 - (i) 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.
 - (ii) Determine the pH, EC, Nitrate-nitrogen concentration, Ammonium-nitrogen concentration, and Orthophosphate-P concentration.
 - (iii) 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 AS 4454).
 - (iv) Weigh between 20 g and 40 g (oven dry equivalent) of the material into the extraction vessel (see Paragraph B4(i)) and record aliquot mass.
 - (v) Add a mass of deionized or distilled water (see <u>Paragraph B3</u>) that is five times the dry mass of the aliquot.
 - (vi) 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(ii)) rotating at less than 10 r/min, for 90 min.
 - (vii) Determine the pH of the suspension with the pH meter (see <u>Paragraph B4(v)</u>).
 - (viii) 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.
 - (ix) 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(iii) or centrifuged.
 - (x) The clear filtrate or centrifugate is the test solution.
 - (xi) Determine the electrical conductivity of the test solution to the nearest 0.05 dS/m with the EC meter.
 - (xii) 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 <u>Paragraph B4(vii)</u>.

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:

$$Nn = 0.226 \times NO_3 \qquad \dots B5.1$$

where:

 $\bf A$ = 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.

(xiii) 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:

 $Nam = 0.78 \times A$...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.

- If required, determine the orthophosphate-P concentration of the test solution to the (xiv) nearest 1 mg/L using an appropriate colorimetric method listed in Paragraph B3.
- (xv) 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: (6)
 - Sample identification, including sufficient details to show the time elapsed between the manufacture and testing of the product.
 - (ii) pH to the nearest 0.1 unit.
 - Electrical conductivity (EC) to the nearest 0.05 dS/m. (iii)
 - (iv) 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.
 - (v) 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.
 - (vi) Test method analytical reference used.
 - (vii) 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 \times solids content \times bulk density = 5 mg/kg \times (1-0.4) \times 0.5 kg/L = 1.5 mg/L compost.

Appendix F(ii): Method for determination of total carbon and nitrogen content (AS 4454 Appendices C)

C1 General

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

Note: Table 2.1(A) specifies the compliance requirements.

C2 Principle

(2) 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

- (3) The following references contain suitable methods for the determination of carbon or nitrogen content:
 - (i) ISO 10694:1995, Soil quality—Determination of organic and total carbon after dry combustion (elementary analysis).
 - (ii) ISO 11261:1995, Soil quality—Determination of total nitrogen—Modified Kjeldahl method.
 - (iii) ISO 13878:1998, Soil quality—Determination of total nitrogen content by dry combustion ("elemental analysis").
 - (iv) Page et al. Methods of soil analysis (Part 2) Chemical and microbiological properties (2nd ed) Soil Science Society of America, Inc, 1982.
 - (v) The Australian Laboratory Handbook of Soil and Water Chemical Methods, authored by G.E. Rayment and F.R. Higginson, was published in Melbourne, Victoria, by Inkata Press in 1992. The ISBN is 9780909605681.
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 - (vii) 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.
 - (viii) 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.
 - (ix) 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

- (4) The following apparatus is required:
 - 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.

Note 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.

Note 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.

(ii) 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.

(iii) 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

(5) 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.

(6) Total nitrogen:

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

C6 Calculation of Organic Matter

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

(1) organic matter = 1.7 × % C (Induction Furnace); ... C6.1
 (2) organic matter = 2.2 × % C (Wet Chemical); or ... C6.2

(3) 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

- (8) The test report shall contain the following:
 - (i) Sample identification, including sufficient details to show the time elapsed between the manufacture and testing of the product.
 - (ii) Total organic carbon content as % dry weight basis.
 - (iii) Organic matter content as % dry weight basis.
 - (iv) Total nitrogen content as % dry weight basis.
 - (v) The C/N ratio being C/N

where

C = total organic carbon; and

N = total nitrogen.

- (vi) Analytical reference used.
- (vii) 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 (AS 4454 App D)

D1 General

- (1) 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 2.1 and 2.2 of the Standard and for the metallic contaminants required from Table 2.4 of the Standard (see main document).
- (2) 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 AS 4454.
- (3) The methods specified in Paragraph D5 require the use of non-pretreated as-received samples and suffer significant analytical error discussed in Paragraph A7 AS 4454. Ensure that the results for organic contaminants have been corrected and presented on a dry weight basis.

D2 principle—digestion for elemental analysis

- (4) A representative dried and ground aliquot of the product of a size determined from Table A6.2 in Appendix A AS 4454 is prepared and digested in aqua regia or similar oxidizing acid mixture and the diluted liquor analysed for the required elements.
- (5) 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).

D3 procedure

(6) 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 D4.

D4 procedure

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

D5 Test Report

- (8) The test report shall contain the following:
 - (i) Sample identification, including sufficient details to show the time elapsed between the manufacture and testing of the product.
 - (ii) Total nutrient levels in mg/kg dry mass, including the following:
 - (a) Nitrogen (N).
 - (b) Phosphorous (P).
 - (c) Potassium (K).
 - (d) Carbon (C).

- (e) Calcium (Ca).
- (f) Magnesium (Mg).
- (g) Sulfur (S).
- (h) Iron (Fe).
- (i) Manganese (Mn).
- (iii) Total contaminant metals and other elements, in mg/kg dry mass, including the following:
 - (a) Arsenic (As).
 - (b) Cadmium (Cd).
 - (c) Boron (B).
 - (d) Chromium (Cr).
 - (e) Copper (Cu).
 - (f) Lead (Pb).
 - (g) Mercury (Hg).
 - (h) Nickel (Ni).
 - (i) Selenium (Se).
 - (j) Zinc (Zn).
 - (k) Molybdenum (Mo).

Appendix F(iv): Method for determination of particle size grading

G1 General

(1) 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 2.1 of the Standard (refer main document) specifies the compliance requirements.

G2 Principle

(2) Soil conditioners, coarse mulches and fine mulches are assessed using sieves of Standard size apertures.

G3 Apparatus

- (3) The following apparatus is required:
 - (i) Stacking sieve with square apertures of 16 mm.
 - (ii) Stacking sieve with square apertures of 5 mm.
 - (iii) A receiving pan for the sieves stack.
 - (iv) Balance accurate to 0.01 g.
 - (v) Means of measuring in millimetres.

G4 Procedure

- (4) The sieving and weighing of particle size fractions must be carried out in the following way:
 - (i) Select a representative aliquot of a size determined from Table A6.1, Appendix A AS 4454, from the bag or batch of product being assessed. Air or oven dry at no higher than 40°C +/-°C.
 - (ii) Determine mass of dry material sample to the nearest 1 g.
 - (iii) 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.
 - (iv) Shake in a horizontal plane for 1 to 2 min until no more material falls through the 5 mm sieve.
 - (v) 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.
 - (vi) Calculate each of the sieved fractions as a percentage (%) by weight of the total dry material sample as follows:
 - (a) >16 mm fraction.
 - (b) >5 mm to <16 mm fraction.
 - (c) <5 mm fraction.

G5 Test Report

- (5) The test report shall contain the following:
 - (i) Sample identification, including sufficient details to show the time elapsed between the manufacture and testing of the product.
 - (ii) 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.
 - (iii) Reference to this test method, i.e. Appendix G of AS 4454.

Appendix F(v): Method for determination of total carbonate content

H1 General

- (1) This Appendix sets out a method for determining the total carbonate content of a product using Standard reference methods.
 - Note 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.
 - **Note 2:** Table 2.1 of the Standard (refer main document) specifies the compliance requirements.

H2 Principle

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

H3 Method Analytical References

- (3) The analytical methods that may be used for this test are listed below:
 - (i) ISO 10693:1995, Soil quality—Determination of carbonate content—Volumetric method.
 - (ii) The Australian Laboratory Handbook of Soil and Water Chemical Methods, authored by G.E. Rayment and F.R. Higginson, was published in Melbourne, Victoria, by Inkata Press in 1992. The ISBN is 9780909605681.
 - (iii) Association of Official Analytical Chemists (AOAC) Method 955.01, Neutralizing value for liming material.

H4 Sample Aliquot

(4) Use dried and ground whole sample (see Paragraph A6.3(C) in Appendix A AS 4454).

H5 Test Report

- (5) The test report shall contain the following:
 - (i) Sample identification, including sufficient details to show the time elapsed between the manufacture and testing of the product.
 - (ii) Percentage of liming materials in the dry product, expressed as % CaCO₃ equivalent, to the nearest 1%.
 - (iii) Method analytical reference used for a the test.
 - (iv) Reference to this test method, i.e. Appendix H of AS 4454.

Appendix F(vi): Method for the measurement of a volume of packaged product and of the volume and bulk density of bulk product (AS 4454 App J)

J1 General

(1) 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 1: Subdivision 2.1 of the Standard (main document) specifies the compliance requirement.

J2 Principle

(2) 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

- (3) For packaged products, rigid, straight-sided pails (see Note) made from translucent plastic, calibrated in litres from the bottom with the following nominal capacities must be used:

 - (ii) For packages of 6 L to 10 L10 L.
 - (iii) For packages of 2 L to 5 L.5 L.

Note 2: If necessary, grind off protrusions of plastic on the base of the pail so that it sits flat.

- (4) The following apparatus must be used to assess bulk products:
 - (i) A rigid, straight-sided container made from translucent plastic, calibrated in litres from the bottom with a minimum capacity of 10 L.
 - (ii) Balance or scales capable of measuring the mass of the sample within }50 g.

J4 Procedure

- (5) The procedure for *packaged products* is as follows:
 - (i) 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.
 - (ii) 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.
 - (iii) Empty the package loosely into the chosen pail and allow its contents to flow out. Use more than one pail if necessary.
 - (iv) Level off the surface of the product and read the volume to the nearest 1 L.
 - (v) 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.
- (6) Bulk product processors and manufacturers must establish and maintain a programme of regular assessment and documentation of bulk density data for their various compost products. Given the substantial variation in bulk density, especially with change in moisture content, it is essential that the bulk density of the finished product be assessed at least weekly. The procedure shall consist of the following steps:

- (i) 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.
- (ii) Weigh the empty, calibrated container of known volume on the scales (see <u>Paragraph J(4)</u> (ii)).
- (iii) Fill the container of known volume (see <u>Paragraph J(6)(i)</u>) with a representative sample of the compost or mulch product in as-received condition, until it overflows.
- (iv) 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.
- (v) Remove compost from the top of the container as required so that the container is filled level with a calibration line.
- (vi) Weigh the full container (i.e. container and representative sample) (mf).
- (vii) Calculate the apparent density of the compost product using the following equation:

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

where:

AD = apparent density (in kilograms per litre)

Mf = mass of full bucket (in kilograms)

Me = mass of empty bucket (in kilograms)

V = volume in the container (in litres).

Example:

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Me = 0.45 \text{ kg}
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Mf = 7.45 kg

V = 10 L

AD = (7.45-0.45)/10

= 0.70 kg/L. or

 $= 0.70 \text{ t/m}^3 \text{ (since } 1000 \text{ kg} = 1 \text{ t; } 1000 \text{ L} = 1 \text{ m}^3\text{)}$

Appendix G: Means of demonstrating compliance with this Standard (AS 4454 App Q)

Q1 Scope

- (1) This Appendix sets out the following different means by which compliance with this Standard can be demonstrated by the manufacturer or supplier:
 - (i) Evaluation by means of statistical sampling.
 - (ii) The use of a product certification scheme.
 - (iii) Assurance using the acceptability of the supplier's quality system.
 - (iv) Other such means proposed by the manufacturer or supplier and acceptable to the customer.

Q2 Statistical Sampling

- (2) 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:
 - (i) 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.
 - (ii) 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.
- (3) 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

- (4) 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 2.1 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.
- (5) The certification scheme serves to indicate that the products consistently conform to the requirements of the Standard.

Q4 Supplier'S Quality Management System

(6) 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 ISO 9001 and ISO 9004.

Q5 Other Means of Assessment

(7) 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

(8)	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
(9)	ISO	
	9001	Quality management system—Requirements
	9004	Quality management system—Guidelines for performance improvements
(10)	ISO/IEC	
	Guide 16	Guidelines for third-party certification and accreditation
	Guide 28	General rules for a model third-party certification system for products

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