



**Australian Government**  

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**Department of the Environment**

**CARBON FARMING INITIATIVE**

**SOIL SAMPLING AND ANALYSIS**

**METHOD AND GUIDELINES**

## Document revision history

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## GLOSSARY

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**Actual sampling depth** means the depth to which sampling occurs at each sampling site if the nominated sampling depth is or isn't achieved (the actual sampling depth must be less than or equal to the nominated sampling depth in each layer).

**Aggregate** means discrete clusters of soil grains.

**Air-dry soil** means soil that has been dried at approximately 40°C to constant mass.

**Australian Soil and Plant Analysis Council (ASPAC)** means the incorporated society by that name.

**Bulk density** means the mass of soil per unit volume.

**Carbon Estimation Area (CEA)** means the carbon estimation area as defined in the relevant methodology determination.

**Composite** means a sample created by bulking and thoroughly mixing individual soil cores collected from different sampling locations.

**Constant mass** means mass that does not change at the nominated drying temperature (air-dry at approximately 40 °C or oven-dry at 105 °C).

**Coring Device** means a cylindrically shaped device (mechanical or manual) used to extract a known volume of soil.

**Fine fraction** means particles of soil (<2 mm) that pass through a 2mm sieve, the portion of soil which is less than 2mm.

**Gravel** means particles of soil ( $\geq 2$  mm) that do not pass through a 2mm sieve.

**Gravimetric** means quantitative analysis by weight.

**Oven-dry soil** means soil that has been dried at 105°C to constant mass.

**National Association of Testing Authorities Australia (NATA)** means the national association by that name.

**Nominated sampling depth** means the depth that is chosen by the proponent for each CEA. For all CFI projects the minimum nominated sampling depth is 30 cm.

**Sample** means an individual soil core which is being analysed separately; or, each separate layer of soil from the soil core which is being analysed separately; or, a composite; or each separate layer of a composite which is being analysed separately.

**Sampling design** means instructions on the spatial layout of sampling locations, the number of samples and (in some cases) compositing of soil samples.

**Sieve** means a utensil consisting of a mesh (at intervals of 2mm) held in a frame, used to separate gravel from the fine fraction of soil.

**Soil core** means a discrete sample of soil that has been extracted with a coring device and includes the gravel ( $\geq 2$ mm) and the fine fraction (<2mm).

**Soil layer thickness** means a measure of the vertical length (depth) of the soil layer represented by a soil core or a segment of a soil core.

**Soil organic carbon (SOC)** means the form of carbon contained within soil organic matter.

**Sub-sample** means a representative portion (reduced mass) of the original (<2 mm fraction) sample upon which laboratory analyses are conducted.

# 1. INTRODUCTION

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## 1.1. CFI BACKGROUND

The Carbon Farming Initiative (CFI) allows farmers and other land managers to earn Australian Carbon Credit Units (ACCUs) by increasing carbon sequestration or reducing greenhouse gas emissions on the land. It is a condition of eligibility that offsets projects must use a methodology approved for use under the CFI. CFI methodologies set out the rules and instructions for undertaking CFI offsets projects, estimating abatement and reporting to the Clean Energy Regulator (CER).

Proposed methodologies are assessed by an independent expert committee, the Domestic Offsets Integrity Committee (DOIC). The DOIC is required to assess whether proposed methodologies meet the requirements of the offsets integrity standards as set out in section 133 of the Carbon Credits (Carbon Farming Initiative) Act 2011. For the purposes of this document, the key offsets integrity standards can be summarised as requiring that:

- the abatement should be measurable and verifiable;
- the method should not be inconsistent with the methods set out in the National Inventory Report;
- the method should be supported by relevant scientific results published in peer-reviewed literature;
- any estimation, assumption or projection in the methodology should be conservative.

## 1.2. OBJECTIVES AND SCOPE

The Soil Sampling and Analysis Method and Guidelines are intended for proponents who are developing a measurement-based soil carbon methodology, and for project proponents applying an approved measurement-based soil carbon methodology determination. In particular, the requirements of the method, as in force from time to time, are adopted by a number of provisions of the *Carbon Credits (Measurement-Based Methodology for Sequestering Carbon in Soils in Grazing Systems) Methodology Determination 2014*, including a number of calculations in Part 5 and sections 4.6, 4.7, 7.7, 7.12 and 7.17.

This document describes the steps required to obtain an estimate of soil organic carbon stocks within a project area through direct soil sampling. The steps described in this document include collection of soil samples, preparation of soil samples for analysis, and analysis of soil sub-samples at a laboratory. This document also presents the calculations required to convert raw data into the parameters required for the estimation of the stock of organic carbon contained in a defined depth of soil per hectare (including soil organic carbon content, bulk density and the gravimetric gravel content of soil samples).

These guidelines include *methods* and *general guidance* for sample collection, preparation and analysis, and *equations* to calculate the parameters required for soil organic carbon stock calculations.

- **The *method*** – presented in the blue boxes in Section 5 describe the compulsory steps and minimum requirements for all measurement-based soil carbon projects. The methods are minimum requirements designed to achieve results with a defined precision, enable independent verification of results, and ensure consistency with the methods set out in Australia's National Inventory Report. Methodology developers should ensure that the

relevant sections of the CFI methodology proposal Section 10 (data collection) and Section 12 (project monitoring and record keeping) require compliance with these standards.

- **The general guidance** - presented in Sections 2, 3 and 4 - provides an explanation of the methods and additional advice on *how* to undertake soil sampling and analysis in order to meet the method requirements. **The general guidance is intended to assist project proponents applying a measurement-based soil carbon methodology determination.** The general guidance section is intended to assist project proponents to make decisions on how to implement the soil sampling and analysis methods by providing additional explanation and advice on how to meet the minimum requirements.
- **The equations** – presented in Section 6 – describe how to calculate the parameters required for soil organic carbon stock calculations from each soil sample based on the analytical results obtained. Methodology developers should ensure that these equations are adapted if required and incorporated into Section 11 (Estimating Abatement) of the CFI methodology proposal. These equations may need to be adapted to comply with the particular requirements of a methodology proposal. .

Sample collection and the preparation of soil samples require skilled use of speciality equipment and knowledge of soils. Improper collection and preparation of soil samples can result in substantial errors, which can render the results of expensive sampling rounds unusable and compromise the integrity of the results. Therefore, the technician undertaking sample collection must be qualified in: implementing a sampling design; undertaking field-based sample collection; and preparing soil samples for submission to laboratories. Project proponents must use a qualified technician who has nationally accredited qualifications which address the competencies as set out in Appendix A to carry out sample collection and sample preparation.

Innovation will continue to drive improvements in soil sampling technologies. While these guidelines are currently restricted to destructive methods of soil sample analysis, additional guidance will be prepared in the future to encompass new technologies and techniques.

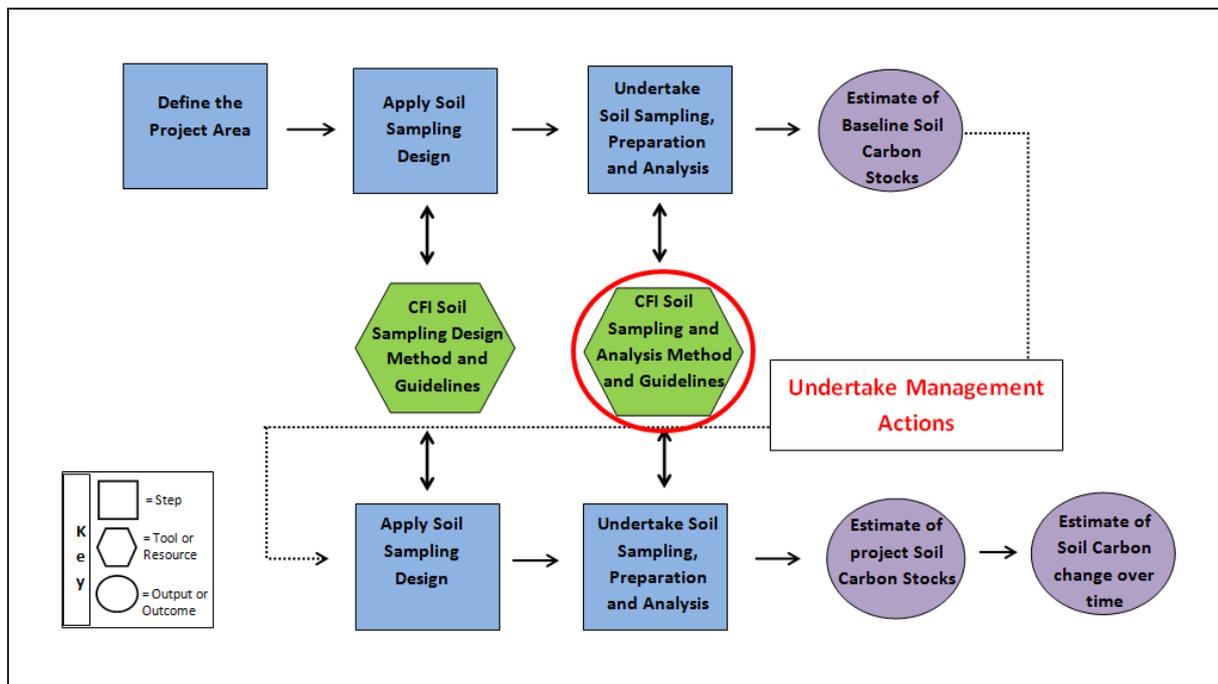
### 1.3. SAMPLING AND ANALYSIS FOR A MEASUREMENT-BASED SOIL CARBON METHODOLOGY

Several steps are required to estimate change in soil organic carbon stocks within a project area over time:

1. Develop a sampling plan for the Project Area based on a soil sampling design;
  - A sampling design provides instructions on the spatial layout of sampling locations, the number of samples, and (in some cases) the timing of sampling and compositing or bulking of soil samples.
2. Sample collection;
3. Sample preparation;
4. Laboratory analysis;
5. Calculation of the organic carbon content of soil samples and soil organic carbon stocks; and
6. Calculation of the change in soil organic carbon stocks over time within each Carbon Estimation Area (CEA).

This document provides guidance on Steps 2, 3, 4 and 5 only. An approved CFI methodology will include instructions on each of the Steps 1 to 6, plus additional instructions related to the particular abatement activity(ies) and project abatement calculation. Project proponents must undertake all steps, in the correct sequence, according to the instructions in the methodology determination.

A schema of a measurement-based approach to estimating changes in soil organic carbon stocks over time is presented in *Figure 1*. The Soil Sampling and Analysis Method and Guidelines (circled in red) are designed to inform the measurement of soil organic carbon stocks in the first sampling round to quantify baseline soil organic carbon stocks, and at subsequent sampling rounds to quantify stocks after project commencement.



*Figure 1: Role of Soil Sample Collection and Analysis Method and Guidelines in a measurement-based methodology.*

## 2. SAMPLE COLLECTION GUIDANCE

**This guidance refers to [Part A](#) of the Method**

Field-based sampling must be carried out by a qualified technician. Accordingly this document is not designed to provide step-by-step instructions on how to undertake field work, but to provide guidance on how to meet the standards set out in the methods section.

Instructions on implementing the sampling design will be found in the measurement-based methodology (or linked sampling design) being applied. This will include instructions for locating sampling sites, and any rules for re-locating the sampling site if obstructions such as trees or a large immovable rocks or rocky outcrops prevent sampling at the intended sample location.

### 2.1. SAMPLING EQUIPMENT

There are a range of mechanical and manual coring devices that can be used to extract individual soil cores. The inner cutting edge of the coring device must have a minimum diameter of 4 cm, to ensure that the sampling depth and bulk density of the soil cores collected are not compromised by compaction. To further minimise the risk of compaction it is recommended that the area ratio (outer body of the coring device divided by the inner body of the coring device) is 10 per cent or less. The coring device used to collect soil cores should be appropriate for the soil type, different diameters or cutting head configurations may be required to allow soil cores to be collected in different textured soils. In addition sampling should be undertaken when the soil moisture content is appropriate to extract an intact core, and this will vary for different soil types.

No lubricants (except water) should be used on coring devices due to potential carbon contamination. Core insertion and/or extraction in sticky clay soils may be aided by giving the inside of the core a light spray with water.

Coring devices must be cleaned after each soil core is collected. This is to ensure that there is no cross-contamination of soil cores.

### 2.2. SITE PREPARATION

Taking care not to disturb the soil surface or sub-surface, the sample site must be cleared of living plants, plant litter and surface rocks prior to sample collection. Surface litter can be considered part of the soil at such time that it becomes difficult to separate the dead surface litter from the mineral soil. Persons undertaking soil sampling should consult property maps and relevant authorities to ensure that sampling does not interfere with above ground or buried power lines, or water supply lines.

### 2.3. SAMPLING PROCEDURE

Australia's National Inventory Report monitors organic carbon in the top 30cm of the mineral soil according to IPCC recommendations which state that it is good practice to measure soil carbon to a depth of at least 30cm<sup>1</sup>. Sampling must occur to a minimum depth of 30cm. Proponents may nominate to sample to a depth greater than 30cm.

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<sup>1</sup> Penman, J, Gytarsky, M, Hiraishi, T, Krug, T, Kruger, D, Pipatti, R, Buendia, L, Miwa, K, Ngara, T, Tanabe, K and Wagner, F, (eds.), IPCC, 2003, [Good Practice Guidance for Land use, Land use change and Forestry](#), Chapter 3.4 Grassland.

Proponents must choose a nominated sampling depth and the technician must aim to sample to the same nominated depth at all sampling sites in a CEA. This will ensure that the estimation of change in organic carbon over time is accurate. At some sampling sites, it may not be possible to sample to the nominated sampling depth (30cm or greater), due to bedrock or impenetrable layers. The actual sampling depth must be recorded for each sampling site.

Sampling depth can be determined by using a depth gauge on the soil corer. Soil samples to the minimum depth of 30cm must be extracted in a single soil core. If the nominated sampling depth is greater than 30cm, soil samples can be extracted in a single soil core, or as two separate soil cores, one to a depth of 30cm and the other to the depth greater than 30cm. If two soil cores are being extracted from the same hole (the 0-30cm layer first, followed by the 30+cm layer), any soil found to have migrated to lower depths on the sides of coring equipment should be physically removed from the soil core. Any soil found to have fallen in from the side wall of the core hole should also be removed from the top of the soil core. In order to prevent large scale contamination of the soil core from the side walls of soils with a high sand content, it is recommended that a sample sheath be used to extract the 30+cm soil core.

Sampling when the soil moisture content is appropriate should prevent significant compaction of the soil in the core. To check if compaction has occurred within the core, the length of the extracted soil core and the depth of the core hole should be measured: these measurements should be similar. If this is not the case, and compaction is considered to be significant, another core should be extracted at the same site (adjacent to the initial core hole) or sampling should be undertaken at a time when the soil moisture content is more appropriate.

The 0-30cm and 30+cm soil samples must be prepared and analysed separately to each other. This will potentially enable proponents to transition to a model-based methodology in the future. It will also enable proponents to determine whether soil organic carbon stocks are increasing over time in the 30+cm layer and assist proponents to decide whether to continue sampling to a depth greater than 30cm in future sampling rounds.

Proponents may choose to split the extracted soil core into multiple layers (for example, 0-10cm, 10-20cm, 20-30cm) to obtain estimates of the organic carbon content in each depth horizon.

Proponents that are using a sampling design which involves compositing or bulking should combine the soil cores into a single sample prior to undertaking the sample preparation steps described in Section 3. If the proponent is using a sampling design which involves compositing, and the soil core has been split into two or more layers, the same layer from each soil core must be combined to form a composite for that layer.

*From this point forward a soil sample refers to a soil sample that is being separately prepared and analysed for organic carbon content. A soil sample could be a single soil core; a single soil core layer (e.g. 0-30cm layer or 30+cm layer); a composite; or a composite of a soil core layer.*

To estimate the bulk density of soil, it is necessary to obtain accurate measurements of the mass of soil collected and the total soil volume from which that mass was collected. Care must be taken during core extraction to make sure that no soil is lost from the core. If soil is lost during extraction or if other factors prevent extraction of a complete core, a new sample should be taken as close as possible to the initial extraction location at a point where no disturbance was caused by the initial extraction.

## 2.4. LABELLING AND DATA COLLECTION

The inner diameter of the cutting tip of the coring device and the actual sampling depth (if the nominated sampling depth cannot be achieved) must be measured and recorded, in order to calculate sample volume.

Each soil sample must be clearly labelled and the sample's GPS location, date of extraction, sample depth, carbon estimation area (CEA) identifier and soil core identifier must be recorded. If project proponents are using a sampling design method that involves stratification and/or compositing, the sample's strata and/or composite identifier must also be recorded.

### 3. SAMPLE PREPARATION GUIDANCE

This guidance refers to [Part B of the Method](#)

All laboratory analysis requires that representative sub-samples of soil samples are prepared and submitted to a laboratory for analysis. Each soil sample, which may be comprised of a single soil core, a single soil core layer, a composite of soil cores, or a composite of a soil core layer, must be prepared and sub-sampled prior to laboratory analysis. Sample preparation steps include air-drying, weighing, crushing, sieving, mixing and sub-sampling cores or composites as illustrated in *Figure 2*. These steps can be carried out prior to submitting sub-samples to a laboratory by a qualified technician who has the necessary equipment, or alternatively these steps can be carried out at a laboratory. Proponents that are using a sampling design which involves compositing or bulking, and who intend to send samples to a laboratory for sample preparation, should check whether the lab has the capacity to process large samples. Some labs may request that samples are air-dried prior to submission.

At each stage of sample preparation, effort should be made to minimise the loss of soil from the sample and to prevent cross-contamination of samples. It is recommended that drop sheets or non-porous surfaces are used to catch and recover spilt soil. It is also recommended that each soil sample is handled separately and that equipment is thoroughly cleaned after each sample is processed.

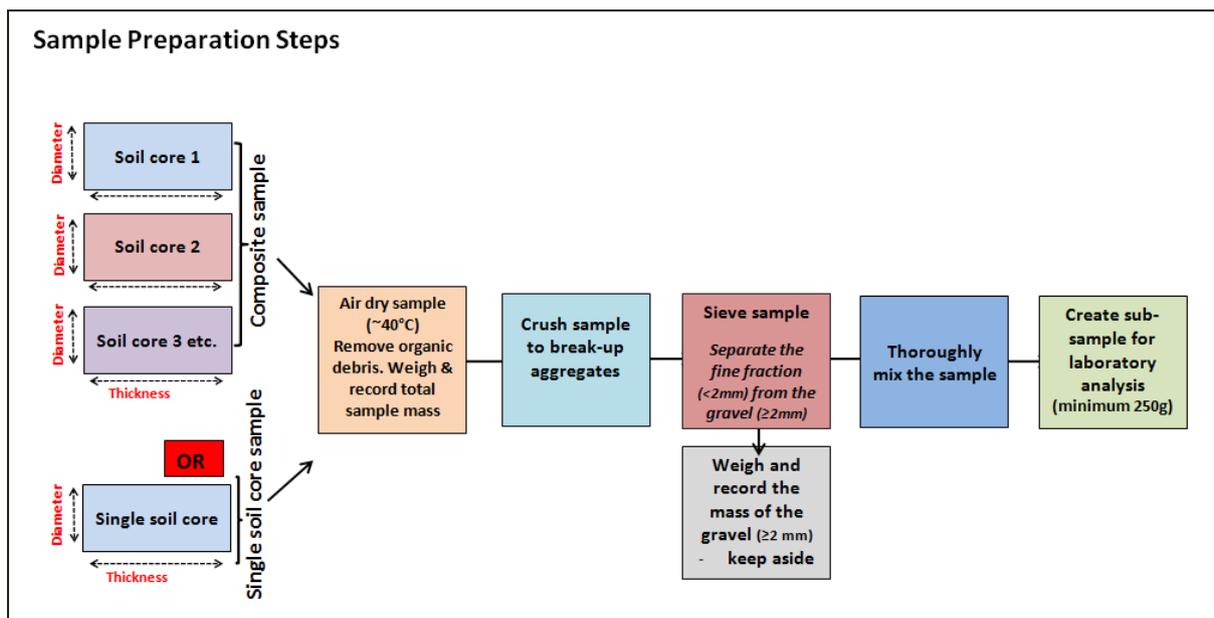


Figure 2: Steps involved in preparing soil sub-samples for laboratory analysis

#### 3.1. AIR-DRYING

Each soil sample must be air-dried at approximately 40° C, for 48 hours or until constant mass has been achieved. It is advisable to keep air drying temperatures at around 40° C because temperatures above 65 ° C can activate carbon oxidation. There are a number of ways to air-dry soil samples, for example within heat controlled ovens or desiccators, or by exposing soil to the air in a large temperature controlled room. It can assist the drying process to break-up any large soil aggregates, and to spread the soil onto sheets or shallow trays.

Each soil sample must be dried separately.

It is highly recommended that samples be air-dried on the same day that samples are extracted. If this is not possible, it is recommended that samples be stored at low temperatures (in a refrigerator or stored with ice or cooling blocks) until air-drying can be carried out. Storage of moist soil samples in warm conditions may result in the loss of carbon due to ongoing chemical and biochemical processes.

Once air-drying is complete any visible organic debris in the soil including leaves, stalks and roots must be removed. Each air-dried soil sample must be weighed to obtain the total air-dry mass of the sample. The scales used to weigh the soil must provide results in grams. For large samples, the soil can be weighed in batches that can be added together after weighing. When samples are weighed, the mass of the sample must be adjusted for the mass of the container.

If the air-drying step is being carried out at a laboratory the soil samples must be submitted to the laboratory within four weeks of sample extraction. The proponent must request that the laboratory weighs the air-dry sample and that the total mass of each air-dried sample is reported.

## 3.2. CRUSHING, SIEVING AND MIXING

Aggregates within the soil must be crushed and broken up. Aggregates may be crushed with a variety of manual or mechanical implements for example a mortar and pestle, shovel or jaw crushing equipment. Soil can be placed into cloth bags or sturdy plastic bags to minimise spillage during manual crushing. Further crushing is likely to be carried out at the laboratory with precision (as described in Section 5), so crushing need only be carried out in order to break-up aggregates at this stage.

Each soil sample must be sieved to separate the <2 mm fraction from any gravel or larger detritus ( $\geq 2$  mm). If the sample contains a large amount of soil (for example, a composite), sieving should be carried out in small batches to prevent soil loss. The sieve should be gently shaken to allow the soil to pass through. The  $\geq 2$  mm fraction that does not pass through the sieve should be collected and set aside. If soil aggregates are present in the  $\geq 2$  mm fraction after sieving, the crushing and sieving steps should be repeated. The gravel fraction ( $\geq 2$  mm) from each core or composite sample must be weighed separately and this mass must be recorded.

If the sieving step is being carried out at a laboratory the proponent must request that the laboratory weighs the gravel fraction of each sample and that the mass of gravel from each sample is reported.

The <2 mm fraction of soil from each core or composite sample must be mixed thoroughly to produce a homogenous sample. Thorough mixing is important to ensure that the sub-samples are representative of the content within each sample. If samples are not mixed thoroughly, the variance in soil carbon between sub-samples is likely to be greater; this will make it more difficult to detect statistically significant changes in soil organic carbon stocks over time.

Manual equipment, such as a bucket and spade; or mechanical equipment, such as a lapidary tumbler, cement mixer or rotating drum, can be used to mix samples. Equipment made of galvanised metal will contaminate the soil sample with zinc. If the methodology being followed requires the soil to be tested for micronutrients, galvanised metal must not be used for sample collection or preparation. All the soil in each sample must be mixed together. The equipment used to mix each sample should be appropriate for the quantity of soil to be mixed; equipment used to mix composite samples will require a larger capacity than equipment used to mix samples comprised of individual cores. In order to manage the volume of soil in a composite and to gain a more homogeneous mix, the soil can be

added to the mixing container in smaller batches. The soil should be mixed thoroughly after each batch is added until all soil from the composite has been incorporated.

### 3.3. SUB-SAMPLING

The aim of sub-sampling is to obtain a small but representative quantity of the soil sample for laboratory testing. A range of methods can be used to create representative sub-samples, such as riffle splitters/sample dividers or manual coning and quartering.

At least one sub-sample must be taken from each sample and must be submitted to the laboratory for analysis. Proponents must retain at least one additional sub-sample per sample for insurance against lost sub-samples, or in case the need arises to repeat laboratory analyses. Proponents may also wish to consider retaining additional sub-samples for future fractionation analysis which will facilitate a transition to a potential, future model-based methodology.

Each sub-sample submitted to the laboratory must contain a minimum of 250 grams (g) of air-dry soil. Each sub-sample must be placed into a sturdy plastic bag or plastic jar and sealed. Each sub-sample must be clearly labelled with its sample and sub-sample identifier. Sub-samples retained for future analysis and insurance purposes should be kept in sealable UV resistant containers (plastic bags or jars) and should be stored out of direct sunlight to preserve the carbon content of the soil. Sub-samples must be stored away from the sunlight or in UV resistant containers prior to laboratory submission.

When submitting soil sub-samples to a laboratory, project proponents must nominate the organic carbon analysis method that they want the laboratory to use. A description of the different methods that are currently available as of the date of publication is provided in Section 4.1.

## 4. LABORATORY ANALYSIS GUIDANCE

This guidance refers to [Part C](#) of the Method

Two separate verification standards currently apply to organic carbon testing in Australia: Australasian Soil and Plant Analysis Council (ASPAC) certification; and National Association of Testing Authorities (NATA) accreditation. These processes provide a measure of confidence in analytical results. The certification process by ASPAC is designed to verify the technical proficiency of participating laboratories through a national quality assurance program. The accreditation process by NATA is designed to verify the technical proficiency of participating laboratories through procedural validation of both the laboratory and the analysis.

Accordingly sub-samples must be analysed in laboratories:

- certified by the Australasian Soil and Plant Analysis Council (ASPAC) using a method that is certified through ASPAC for organic carbon analysis and gravimetric water content; OR
- using an organic carbon method and gravimetric water content method that has been accredited for that laboratory by NATA under ISO-IEC 17025 (Chemical Testing).

Organic carbon and water content analysis must be carried out on sub-samples taken from the <2 mm fraction of the soil sample. Laboratory analysis steps include organic carbon analysis and calculation of the gravimetric water content of the air-dry soil as illustrated in *Figure 3*. Analyses of the organic carbon content and the gravimetric water content of the soil are two discrete analyses that must be carried out separately; therefore each sub-sample must be split into at least two further sub-samples at the laboratory.

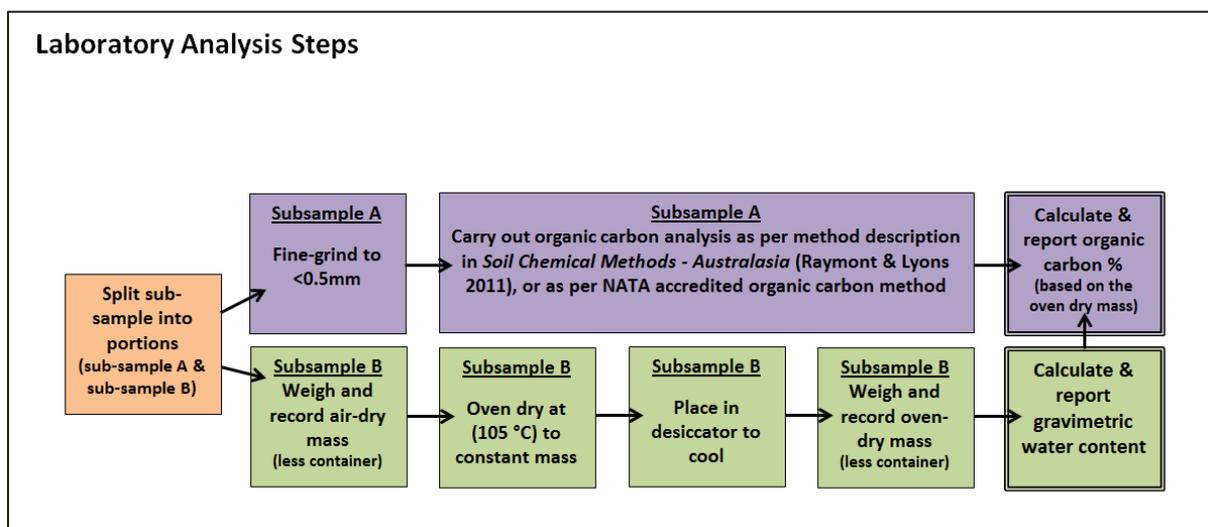


Figure 3: Steps involved in laboratory analysis of organic carbon.

### 4.1. SELECTING AN ORGANIC CARBON ANALYSIS METHOD

There are currently two different organic carbon analysis techniques that are ASPAC-certified or NATA-accredited: dry combustion and wet oxidation. Other techniques, such as infrared diffuse reflectance spectroscopy, are in development and may allow for faster turnaround and reduced analysis costs in the future. As new methods become ASPAC-certified or NATA-accredited they may be used to analyse the organic carbon content of soil samples under the CFI.

The organic carbon analysis methods that are currently ASPAC-certified or NATA-accredited - which use dry combustion or wet oxidation techniques – are briefly described here to assist proponents in selecting a method. Soil Chemical Methods Australasia (2011)<sup>2</sup> provides a full description of organic carbon analysis methods.

The **Dumas high temperature dry combustion method**, determines the total organic carbon content of a soil sample through high temperature (>1000 °C) conversion of organic carbon to carbon dioxide and volumetric or infrared measurement of the resulting carbon dioxide. The Dumas dry combustion method requires additional steps to physically remove charcoal and chemically remove carbonates prior to combustion.

**Wet oxidation techniques used** to determine the organic carbon content of soil include the Heanes (1984)<sup>3</sup> and the Walkley and Black (1934)<sup>4</sup> methods. Wet oxidation methods use sulphuric acid to generate heat and an acidic dichromate solution to oxidise the organic carbon. The carbon content is measured by titration or through photometric means by measuring absorbance at 600 nm. The Walkley and Black method can potentially return a lower organic carbon measurement than the Heanes method due to incomplete oxidation, and will often require conversion to a total organic carbon measurement. The Heanes method includes an additional controlled heating step to enhance the oxidation process, and therefore produces a measurement of total organic carbon content. Wet oxidation methods require an additional step to correct for saline soils.

In choosing an organic carbon analysis method, project proponents should consider the relative precision of different methods. Table 1 below shows estimates of the precision that can be expected from Dumas with pre-treatment of charcoal and carbonates (6B3), Heanes (6B1) and Walkley & Black (6A1), analyses produced within ASPAC-certified laboratories.

**Table 1: Precision of ASPAC Certified Organic Carbon Laboratory Methods**

| Method                              | Organic carbon measure total or incomplete | Standard deviation of results within ASPAC laboratories <sup>b</sup> | Standard deviation of results between comparison laboratories <sup>c</sup> |
|-------------------------------------|--|--|--|
| Dumas (6B3 dry combustion)          | Total                                      | 5–8%   | 7-10%  |
| Heanes (6B1 wet oxidation)          | Total                                      | 8-10%  | 10-12%   |
| Walkley & Black (6A1 wet oxidation) | Incomplete                                 | 8-12%  | 15%  |

Source: Rayment & Lyons, 2010, Soil Chemical Methods – Australasia, CSIRO Publishing and D Lyons pers. comm.

<sup>b</sup> Data Sourced from two major Australian soil testing laboratories, expressed as  $\pm 2$  times reproducibility relative standard deviation %

<sup>c</sup> Data sourced from the ASPAC inter-laboratory proficiency program, expressed as % robust coefficient of variation (CV)

<sup>2</sup> Rayment D, and Lyons, D, 2011, Soil Chemical Methods – Australasia, CSIRO Publishing.

<sup>3</sup> Heanes, D, 1984, Determination of total organic-c in soils by an improved chromic acid digestion and spectrophotometric procedure, *Communications in Soil Science and Plant Analysis*, Vol. 15, pp. 1191-1213.

<sup>4</sup> Walkley, A & Black, I, 1934, An examination of the Degtjareff method for determining soil organic matter and a proposed modification of the chromic acid titration method, *Soil Science*, Vol. 37, pp. 29-38.

Project proponents may also wish to consider the recommendations of the Intergovernmental Panel on Climate Change (IPCC), which states that it is good practice to use the dry combustion technique to measure carbon in situations where the measurement of soil carbon is significant to the project<sup>5</sup>.

## 4.2. MEASUREMENT OF ORGANIC CARBON

The sub-sample intended for organic carbon analysis must be fine ground to pass through a 0.5mm sieve in preparation for organic carbon analysis. The laboratory must conduct organic carbon analysis on the finely-ground sub-sample in accordance with ASPAC-certified or NATA-accredited procedures.

## 4.3. MEASUREMENT OF WATER CONTENT FOR BULK DENSITY CALCULATION

Bulk density is a measurement of the oven-dry mass of the soil sample divided by the volume of the sample and is required to calculate the organic carbon stocks of an area. To determine the oven-dry mass of the soil sample, it is necessary to measure the water content of the air-dry sub-sample.

The laboratory must undertake water content analysis on one air-dry sub-sample per core or composite sample. An air-dry sub-sample must be oven-dried at the maximum temperature of 105°C to constant mass. Once the sub-sample has reached constant mass it must be placed in a desiccator to cool, and weighed quickly on extraction from the desiccator so it does not re-absorb moisture from the air. The initial air-dry mass and final oven-dry mass of each sub-sample analysed for water content must be measured and recorded by the laboratory, in order to calculate the water mass of the sub-sample. The gravimetric water content of the air-dry sub-sample is calculated as a ratio of the water mass to the oven-dry soil mass in the sub-sample.

Laboratory reports must include the gravimetric water content of each sub-sample submitted for analysis.

## 4.4. ORGANIC CARBON REPORTING

The laboratory must state if the organic carbon content of the soil sub-sample is reported on an oven-dry (105° C) or an air-dry (40° C) mass basis. If results are reported on an air-dry mass basis, an additional calculation must be carried out to convert the results to an oven-dry mass basis (see Section 6, Equation 4). All calculations to determine soil organic carbon stocks must be based on the oven-dry mass of the soil because clay has a higher water holding capacity than the coarser components of soil. If calculations were based on the air-dry mass, the mass of any clay-based soils would include residual water and would result in an inaccurate measure of the soil organic carbon content.

Laboratory reports must include the method of organic carbon analysis, as the use of the Walkley and Black method requires the application of an additional conversion factor (See Section 4.5 below).

## 4.5. CONVERSION OF WALKLEY AND BLACK RESULTS

The Walkley and Black wet oxidation method will return a lower organic carbon content measurement than the Heanes wet oxidation or Dumas dry combustion total organic carbon methods. Consequently, it is recommended that a project proponent uses either the Walkley and Black method

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<sup>5</sup> Penman, J, Gytarsky, M, Hiraishi, T, Krug, T, Kruger, D, Pipatti, R, Buendia, L, Miwa, K, Ngara, T, Tanabe, K and Wagner, F, (eds.), IPCC, 2003, [Good Practice Guidance for Land use, Land use change and Forestry](#).

for the duration of the project, or a total organic carbon method (Heanes or Dumas) for the duration of the project.

If project proponents use the Walkley and Black method at any sampling round during the project, proponents must convert the incomplete oxidation results produced through the Walkley and Black method to a total organic carbon measurement (unless this conversion has been performed by the laboratory). Results from the Walkley and Black and total organic methods (Dumas and Heanes) reported in ASPAC proficiency rounds have been compared to determine a conversion factor for ASPAC certified laboratories. For ASPAC certified laboratories, the organic carbon content results attained via Walkley and Black method must be multiplied by a factor of 1.1359 for conversion to total organic carbon (See Equation 6 in Section 6).

## 5. METHOD

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This section details the minimum standards that are required for the purposes of carrying out a soil-carbon CFI measurement-based methodology. Methodology developers should ensure that the relevant sections of the CFI methodology proposal (Section 10 (data collection) and Section 12 (project monitoring and record keeping) require compliance with these standards. This method is adopted by a number of provisions of the *Carbon Credits (Measurement-Based Methodology for Sequestering Carbon in Soils in Grazing Systems) Methodology Determination 2014*.

### GLOSSARY

**Actual sampling depth** means the depth to which sampling occurs at each sampling site if the nominated sampling depth is or isn't achieved (the actual sampling depth must be less than or equal to the nominated sampling depth in each layer).

**Aggregate** means discrete clusters of soil grains.

**Air-dry soil** means soil that has been dried at approximately 40°C to constant mass.

**Australian Soil and Plant Analysis Council (ASPAC)** means the incorporated society by that name.

**Composite** means a sample created by bulking and thoroughly mixing individual soil cores collected from different sampling locations.

**Constant mass** means mass that does not change at the nominated drying temperature (air-dry at approximately 40 °C or oven-dry at 105 °C).

**Coring Device** means a cylindrically shaped device (mechanical or manual) used to extract a known volume of soil.

**Gravel** means particles of soil ( $\geq 2$  mm) that do not pass through a 2mm sieve.

**Gravimetric** means quantitative analysis by weight.

**National Association of Testing Authorities Australia (NATA)** means the national association by that name.

**Nominated sampling depth** means the depth that is chosen by the proponent for each CEA. For all CFI projects the minimum nominated sampling depth is 30 cm.

**Sample** means an individual soil core which is being analysed separately; or, each separate layer of soil from the soil core which is being analysed separately; or, a composite; or each separate layer of a composite which is being analysed separately.

**Sieve** means a utensil consisting of a mesh (at intervals of 2mm) held in a frame, used to separate gravel from the fine fraction of soil.

**Soil core** means a discrete sample of soil that has been extracted with a coring device and includes the gravel ( $\geq 2\text{mm}$ ) and the fine fraction ( $< 2\text{mm}$ ).

**Soil layer thickness** means a measure of the vertical length (depth) of the soil layer represented by a soil core or a segment of a soil core.

**Soil organic carbon (SOC)** means the form of carbon contained within soil organic matter.

**Sub-sample** means a representative portion (reduced mass) of the original ( $< 2\text{ mm}$  fraction) sample upon which laboratory analyses are conducted.

## **PART A: SAMPLE COLLECTION**

**Note:** See section 4.6 of the *Carbon Credits (Measurement-Based Methodology for Sequestering Carbon in Soils in Grazing Systems) Methodology Determination 2014*.

### **A.1 QUALIFICATIONS OF TECHNICIANS**

The person undertaking sample collection or sample collection and sample preparation must hold nationally-accredited qualifications or formal recognition of prior learning that addresses the competencies listed in Appendix A.

### **A.2 SAMPLING EQUIPMENT**

The coring device used to collect soil samples must:

- (a) have a consistent diameter, with the exception of the cutting tip;
- (b) have a minimum inner cutting head diameter of 4 cm; and
- (c) be clean and free of any lubricants excluding water.

### **A.3 SITE PREPARATION**

The sample site must be cleared of living plants, plant litter and surface rocks prior to sample collection with minimal disturbance to the soil surface or sub-surface.

### **A.4 SAMPLE EXTRACTION**

- (1) The nominated sampling depth must be a minimum of 30 centimetres (cm).
- (2) Sampling must occur to the same nominated sampling depth across the CEA in each sampling round, unless bedrock or impenetrable layers prevent sampling to this depth.
- (3) In circumstances where bedrock or impenetrable layers prevent sampling to the nominated sampling depth, the actual sampling depth must be recorded.
- (4) All soil in the soil core to the nominated or actual sampling depth must be retained.
- (5) Soil from the 0-30 centimetre (cm) layer and the 30+ centimetre (cm) layer must be extracted separately or must be separated prior to the sample preparation step.

## A.5 RECORD KEEPING

- (1) The name and qualifications of the person carrying out sample collection must be recorded.
- (2) The person in subclause (1) must sign a statutory declaration which states the sample collection or sample collection and preparation has been undertaken in accordance with this method.  
*Note:* See section 7.15 of the *Carbon Credits (Measurement-Based Methodology for Sequestering Carbon in Soils in Grazing Systems) Methodology Determination 2014*.
- (3) The inner diameter of the cutting head on the coring device(s) must be measured and recorded in centimetres (cm) to three significant figures.
- (4) The details of each soil core collected must be recorded including:
  - (a) nominated sampling depth, or actual sampling depth in centimetres (cm) to three significant figures;
  - (b) the coordinates of the actual sampling location expressed in the format required by the methodology being implemented, or if the methodology does not specify a format then in decimal degrees to at least 5 decimal places as Eastings and Northings;
  - (c) if more than one cutting head is used to sample within a CEA, the inner diameter of each cutting head used at each sampling location must be recorded;
  - (d) date of collection;
- (5) The details of each soil sample must be recorded including:
  - (a) CEA identifier
  - (b) strata identifier (if using a stratified sampling design);
  - (c) core or composite identifier; and
  - (d) soil layer identifier (if more than one soil layer is being sampled).
- (6) If the nominated sampling depth is greater than 30 centimetres (cm), or if soil cores are further separated into multiple layers for separate organic carbon and water content analysis, the thickness of each soil layer being separately analysed must be recorded in centimetres (cm) to three significant figures.

## A.6 OFFSETS REPORT REQUIREMENTS

Offsets reports for a project must contain the following information:

- (a) The name and qualifications of the person undertaking sample collection or sample collection and preparation.
- (b) The statutory declaration signed by the person in subclause A.5(1) stating that the sample collection or sample collection and preparation has been undertaken in accordance with this method.
- (c) The date of sample collection in each CEA. If the date of the sample collection occurs over a number of days, report the first and last dates of sample collection.
- (d) The starting date in which sample collection was commenced within the project area.
- (e) The final date in which sample collection was completed for the entire project area.
- (f) The nominated sampling depth for each CEA in each sampling round.

## **PART B SAMPLE PREPARATION**

*Note:* See section 4.6 of the *Carbon Credits (Measurement-Based Methodology for Sequestering Carbon in Soils in Grazing Systems) Methodology Determination 2014*.

### **B.1 AIR-DRYING SAMPLES**

- (1) Each soil sample must be dried at the approximate temperature of 40 degrees centigrade (°C) for 48 hours or until constant mass is achieved.
- (2) If soil samples are not air dried prior to laboratory submission, the soil samples must be submitted to the laboratory within four weeks of sample extraction.

### **B.2 ORGANIC DEBRIS**

Visible organic debris must be removed from the soil sample.

### **B.3 CRUSHING**

Aggregates present in each soil sample must be crushed and broken up.

### **B.4 SEPARATING SOIL FRACTIONS**

The soil from each sample must be sieved with a clean 2 millimetre (mm) sieve.

### **B.5 CORE OR COMPOSITE MIXING**

The <2 millimetre (mm) fraction of soil from each core or composite must be mixed with clean equipment.

### **B.6 SUB-SAMPLING AND LABELLING**

- (1) At least one sub-sample of 250 grams (g) must be taken from the <2 millimetre (mm) fraction of each sample.
- (2) Each sub-sample must be placed into a sturdy plastic container and sealed.
- (3) Each sub-sample must be labelled with the sample and sub-sample identifiers.

### **B.7 STORAGE AND HANDLING**

Each sub-sample must be stored away from the sunlight or in UV resistant containers prior to laboratory submission.

### **B.8 RECORD KEEPING**

- (1) Each air-dry soil sample must be weighed and the mass recorded in grams (g) to three significant figures.
- (2) The gravel ( $\geq 2$ mm fraction) content of each soil sample must be weighed and the mass recorded in grams (g) to three significant figures.

## PART C ORGANIC CARBON AND WATER CONTENT ANALYSIS

*Note:* See section 4.7 of the *Carbon Credits (Measurement-Based Methodology for Sequestering Carbon in Soils in Grazing Systems) Methodology Determination 2014*.

### C.1 LABORATORY AND METHOD STANDARDS

- (1) Analysis of soil sub-samples must be undertaken by a laboratory that is;
  - (a) certified for organic carbon analysis by the Australasian Soil and Plant Analysis Council (ASPAC) and using a method certified for organic carbon analysis and gravimetric water content by ASPAC, or
  - (b) using an organic carbon method and gravimetric water content method that has been accredited for that laboratory by the National Association of Testing Authorities Australia (NATA) under ISO-IEC 17025 (Chemical Testing).
- (2) Physical removal of charcoal and chemical removal of carbonates must be carried out in the laboratory prior to organic carbon analysis if the Dumas dry combustion method is used.

*Note:* See description of Dumas dry combustion method on page 15 of this guide.

### C.2 ORGANIC CARBON ANALYSIS

The laboratory must carry out organic carbon analysis on the air-dry soil from a sub-sample.

### C.3 WATER CONTENT ANALYSIS

The laboratory must carry out water content analysis on the air-dry soil from a sub-sample.

### C.4 CONVERSION OF WALKLEY AND BLACK RESULTS

For project proponents using ASPAC-certified laboratories, the organic carbon content results obtained from the Walkley and Black method must be converted to total organic carbon by multiplying the results by a factor of 1.1359 unless the laboratory reports state that a conversion factor has been applied (see subclause C.5(4)).

*Note:* See description of Walkley and Black method on page 15 of this guide.

## C.5 RECORD KEEPING

- (1) ASPAC certification or NATA accreditation for the organic carbon method and the laboratory carrying out organic carbon and water content analysis of sub-samples must be recorded.
- (2) All laboratory reports for each sampling round must be retained for verification.
- (3) Laboratory reports must state the method of organic carbon analysis.
- (4) Laboratory reports must state whether a conversion factor has been applied to convert organic carbon content results produced through the Walkley and Black method to total organic carbon.
- (5) Laboratory reports must state the organic carbon content in each soil sample submitted for analysis as a percentage of the mass of the soil sub-sample (%) or in grams of carbon per kilogram of soil (g/kg).
- (6) Laboratory reports must state if the organic carbon content is reported on an oven-dry (105° C), or an air-dry (40° C) mass basis.
- (7) Laboratory reports must state the gravimetric water content of each sub-sample submitted for analysis in grams of water per grams of oven dry soil (g/g).
- (8) Proponents must record the calculations used to convert incomplete oxidation results produced through the Walkley and Black method to total organic carbon in accordance with clause C.4, unless the laboratory reports state that a conversion factor has been applied, in accordance with subclause C.5(4).

## C.6 OFFSETS REPORT REQUIREMENTS

Offsets reports for a project must contain:

- (a) Proof of ASPAC certification or NATA accreditation for the organic carbon method and the laboratory carrying out organic carbon and water content analysis of sub-samples.

## 6. CALCULATIONS

The following equations derive the parameters required to calculate organic carbon stocks (organic carbon content per hectare) of each (single core or composite) soil sample using data collected during the extraction, preparation and analysis of the samples as per the method in Section 5.

The calculation of organic carbon stocks in a soil sample requires the following parameters: the thickness of the soil layer (6.1), the gravimetric gravel content of the soil sample (6.2), the bulk density of the soil sample (6.3), the organic carbon content of a sub-sample (6.4). This calculation is not included below as a methodology may modify the equation depending on how soil layers and sampling depths are treated. For example, the organic carbon stock calculations in a methodology may be based on the nominated and/or the actual thickness of the soil layer.

### 6.1. Calculate the average actual thickness of the soil layer for a composite sample

This equation should be used to calculate the actual average thickness of a composite sample for a given soil layer. Where a sample is comprised of a single soil core, actual thickness of the sample is  $t$ .

|                                      |            |
|--------------------------------------|------------|
| $T_a = \frac{1}{n} \sum_{i=1}^n t_i$ | Equation 1 |
|--------------------------------------|------------|

Where:

$T_a$  = the average actual thickness of the composite sample for a given soil layer (cm).

$t$  = the actual thickness of each soil core for a given soil layer (cm).

$n$  = the number of cores in a composite sample:

$i$  = a/each specific soil core.

### 6.2. Calculate the gravimetric gravel content of the oven-dry soil sample

|                                       |            |
|---------------------------------------|------------|
| $P_{gravel} = \frac{M_{gravel}}{M_s}$ | Equation 2 |
|---------------------------------------|------------|

Where:

$P_{gravel}$  = the gravimetric gravel content of the oven-dry soil sample (g).

$M_{gravel}$  = the mass of gravel ( $\geq 2$  mm) in the soil sample (g).

$M_s$  = the total oven-dry mass of the soil sample (g).

### 6.2.1. Calculate the total oven dry-mass of the soil sample

|                               |              |
|-------------------------------|--------------|
| $M_s = M_{<2mm} + M_{gravel}$ | Equation 2.1 |
|-------------------------------|--------------|

Where:

$M_s$  = the total oven dry-mass of the soil sample (g).

$M_{<2mm}$  = the oven-dry equivalent mass of the fraction of the soil sample that is <2 mm (g).

$M_{gravel}$  = the mass of gravel ( $\geq 2$  mm) in the soil sample (g).

### 6.2.2. Calculate the oven-dry equivalent mass of the <2 mm fraction of soil

|  |              |
|--|--------------|
| $M_{<2mm} = \frac{M_{AD<2mm}}{1 + \theta_m}$ | Equation 2.2 |
|--|--------------|

Where:

$M_{<2mm}$  = the oven-dry equivalent mass of the fraction of the soil sample that is <2 mm (g).

$M_{AD<2mm}$  = the air-dry mass of the fraction of the soil sample that is <2mm (g).

$\theta_m$  = the gravimetric water content of the <2 mm air-dry sub-sample (g water /g oven-dry soil mass) as reported by the laboratory.

### 6.2.3. Calculate the air-dry mass of the <2mm fraction of soil

|                                    |              |
|------------------------------------|--------------|
| $M_{AD<2mm} = M_{AD} - M_{gravel}$ | Equation 2.3 |
|------------------------------------|--------------|

Where:

$M_{AD<2mm}$  = the air-dry mass of the fraction of the soil sample that is <2mm (g).

$M_{AD}$  = the air-dry mass of the soil sample (g).

$M_{gravel}$  = the mass of gravel ( $\geq 2$  mm) in the soil sample (g).

### 6.3. Calculate the bulk density of the soil sample

|                                     |            |
|-------------------------------------|------------|
| $BD = \frac{M_s}{\sum_{i=1}^n V_i}$ | Equation 3 |
|-------------------------------------|------------|

Where:

$BD$  = the bulk density of the oven-dry soil sample ( $\text{g/cm}^3$ ).

$M_s$  = the oven dry-mass of the soil sample (g).

$V$  = the volume of a/each core ( $\text{cm}^3$ ).

$n$  = the number of cores in the sample:

- where the sample is comprised of one soil core,  $n = 1$ ;
- where the sample is a composite,  $n$  = the number of soil cores included in the composite.

$i$  = a/each specific soil core.

### 6.3.1. Calculate core volume

|                 |              |
|-----------------|--------------|
| $V = \pi r^2 t$ | Equation 3.1 |
|-----------------|--------------|

Where:

$V$  = the volume of a/each core ( $\text{cm}^3$ ).

$r$  = the radius of the cutting head of the core (cm).

$t$  = the actual thickness of each sample (cm).

## 6.4. Organic carbon content of the sub-sample

A value for the oven-dry organic carbon content of the sub-sample, expressed as a percentage (%), ( $OC_{OD}$ ) is required for organic carbon stock calculations. The following equations may be required to convert the organic carbon content reported by the laboratory to percentage oven-dry organic carbon content ( $OC_{OD}$ ).

### 6.4.1. Convert air-dry organic carbon to oven-dry organic carbon

To be carried out only if organic carbon results are reported on an air-dry basis.

|   |            |
|---|------------|
| $OC_{OD} = OC_{AD} \times (1 + \theta_m)$ | Equation 4 |
|---|------------|

Where:

$OC_{OD}$  = the oven-dry organic carbon content of the sub-sample, expressed as a percentage (%).

$OC_{AD}$  = the air-dry organic carbon content of the sub-sample, expressed as a percentage (%) as reported by the laboratory.

$\theta_m$  = the gravimetric water content of the <2 mm air-dry sub-sample (g water /g oven-dry soil mass) as reported by the laboratory.

### 6.4.2. Convert grams per kilogram organic carbon to a percentage

To be carried out only if organic carbon results are reported on a gram per kilogram (g/kg) basis.

|                             |            |
|-----------------------------|------------|
| $OC = \frac{OC_{g/kg}}{10}$ | Equation 5 |
|-----------------------------|------------|

Where:

$OC$  = the organic carbon content (air-dry or oven-dry) of the sub-sample, expressed as a percentage (%).

$OC_{g/kg}$  = the organic carbon content (air-dry or oven-dry) of the sub-sample, expressed in grams per kilogram (g/kg), *as reported by the laboratory*.

10 = the conversion factor required to convert g/kg to a percentage.

### 6.5. Convert Walkley and Black results to a dry combustion equivalent

To be carried out if Walkley and Black analysis method has been used at an ASPAC certified laboratory.

|                                |            |
|--------------------------------|------------|
| $OC_{OD} = W\&B \times 1.1359$ | Equation 6 |
|--------------------------------|------------|

Where:

$OC_{OD}$  = the oven-dry organic carbon content of the sub-sample, expressed as a percentage (%).

$W\&B$  = Walkley and Black results (%), *as reported by the laboratory*.

1.1359 = the conversion factor.

## Appendix A:

# SOIL TECHNICIAN COMPETENCIES

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The soil technician undertaking field-based sample collection and preparation of soil samples for submission to a laboratory must have qualifications from a nationally accredited course, or formal recognition of trade or prior learning (from a nationally accredited institution), in the following competencies:

### 1. Implementation of sampling design:

- Set up and use a GPS in the field.
- Location of specific sites according to pre-determined coordinates.

### 2. Site preparation and equipment for soil sampling

- Identification of work health and safety (WH&S) hazards, identify risks and implementation of controls to avoid accidents (including selecting suitable personal protection equipment, avoiding overhead wiring and consulting local authorities to locate underground services).
- Selection of appropriate tools, equipment and machinery for soil sampling according to required sampling depth and site conditions (including soil physical properties).
- Carrying out pre-operational and safety checks on tools, equipment and machinery according to manufacturer's specifications.

### 3. Soil sampling

- Safe and accurate operation of appropriate tools, equipment and machinery.
- Extraction of soil cores intact with minimum disturbance to the soil core and the surrounding field site.
- Accurate documentation of details of extracted cores.

### 4. Preparation of soil samples for laboratory submission

- Selection of appropriate techniques, tools, equipment and machinery with which to prepare representative sub-samples of soil for soil carbon and water content analysis in a laboratory.
- Safe and accurate operation of the appropriate tools, equipment and machinery required to prepare representative sub-samples of soil for laboratory submission.
- Use of appropriate techniques to prepare representative sub-samples of soil for submission to laboratories for soil carbon and water content analysis.
- Accurate documentation of details of samples and subsamples.