



Australian Government Department of Industry, Science, Energy and Resources

Sample collection and analysis and data provision protocols (draft version 1)

National Soil Carbon Innovation Challenge

Soils and Vegetation Emissions Reduction Branch 4 January 2022

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About this document

These protocols apply to the collection, analysis and provision of soil organic carbon stock data and soil samples required of participants of the Development and Demonstration Round 1 grants under the National Soil Carbon Innovation Challenge.

Requirement to share data using these Protocols

In accordance with section G of the Development and Demonstration Round 1 grants under the National Soil Carbon Innovation Challenge Grant Agreement, it is a requirement for new soil carbon stock data collected as part of supported projects to be made available in accordance with these Protocols. Such data may be collected in various ways. Additionally, to the extent possible, existing data that is used in developing/testing the technologies should conform to these Protocols or be otherwise compatible with these Protocols, so as to ensure that existing data is of a quality that can be shared and developed in accordance with the Grant Agreement. Grantees should make that existing data available whenever that is possible.

For information on these requirements, refer to the:

- National Soil Carbon Innovation Challenge Development and Demonstration Round 1 grant opportunity Guidelines
- Sample Grant Agreement.

These Protocols have been developed to:

- enable the collection and provision of high quality data that may be used to develop new soil carbon stock and stock change measurement technologies
- ensure data is collected and organised to a minimum standard that supports quality control and assurance processes
- promote consistency between soil carbon stock data collection practices across Australian Government funded research and development activities
- support improved national soil data sharing in line with the objective of the National Soil Strategy to make Australian soil information and data available.

Data shared through the Innovation Challenge will be collated into a public dataset that will increase the availability of high quality, spatial and temporal carbon stock data for validation of soil carbon stock measurement technologies. A prerequisite of developing and deploying new soil carbon stock measurement technologies is an ability to assess both the accuracy and precision of the estimates produced. To achieve this prerequisite, soil carbon stock estimates produced by the technologies must be compared to corresponding spatial and temporal stock values obtained through conventional measurement of soil samples. The validity of such comparisons relies on the use of appropriate soil collection, preparation, and analysis protocols in the derivation of measured soil carbon stock values.

These protocols set minimum requirements for soil sampling, analysis and data provision that apply if you are collecting new data under the Innovation Challenge. For example, the minimum sampling size that is considered 'new data' required to be shared is at least 3 soil samples, from a soil collection site of at least 625 m² (i.e. 25 m x 25 m). These minimum requirements have been set to enable Innovation Challenge participants the flexibility to determine the appropriate sample size for their project, while ensuring that data generated by existing approaches such as that from the Soil Carbon Research Program (SCaRP) and Emissions Reduction Fund (ERF) methods can be used.

However, it is expected that more than the minimum required samples will be collected in order to obtain sufficient data to enable innovators to determine the level of confidence (uncertainty) that can be attributed to their measurement solution.

These protocols are intended to be flexible enough for use in other Australian Government programs that can support additional soil carbon stock data collection activities, such as the Pilot Soil Monitoring and Incentives Program, and the Soil Science Challenge (Department of Agriculture, Water and the Environment). The protocols are also intended to be flexible enough to allow, where appropriate, data collected in previous sampling programs and by ERF methods to be eligible for use. Ensuring consistency in soil carbon stock measurement approaches would facilitate increased availability of high-quality data that may be used to develop new soil carbon stock and stock change measurement technologies.

Note on participating in the Emissions Reduction Fund and other Australian Government funded soil programs

There are a number of linked soil programs being implemented by the Australian Government and it is important to understand how participation in these programs may affect the registration of projects under the Emissions Reduction Fund. This information is included to outline the links between the programs and is for information only, rather than advice or guidance.

The Australian Government is administering the following programs which may provide financial support for testing of soil carbon, trialling of new soil carbon measurement technology or trialling of management activities that increase soil carbon:

- the <u>National Soil Carbon Innovation Challenge</u> and the National Soil Carbon Data Program (Department of Industry, Science, Energy and Resources).
- the <u>Pilot Soil Monitoring and Incentives Program</u> and the <u>Soil Science Challenge</u> (Department of Agriculture, Water and the Environment).

There may be opportunities to register an Emissions Reduction Fund (ERF) soil carbon project as well as participating in one or more of these programs. If participating in these programs would involve undertaking activities on the same areas of land as those included in the soil carbon project, consider registering that land as an ERF soil carbon project before commencing any new management activity expected to sequester carbon in the soil. This will mean that increases in soil carbon gained because of this new activity may be eligible for crediting under the ERF.

In addition, if soil carbon testing is undertaken as part of participating in one of these programs, it could be used in reporting creditable increases in soil carbon provided that the soil sampling and analysis requirements of both the relevant ERF method and other program are satisfied (e.g. to calculate soil carbon stocks at the baseline and subsequent soil sampling rounds). This includes undertaking baseline sampling after applying to register the project under the ERF (in line with the ERF's sampling requirements).

If a soil carbon project has already been registered and participation in any of these soil programs is being considered, the program eligibility requirements should be reviewed as they are made available.

Definitions

The term *soil carbon* will be used throughout this document to refer solely to the organic carbon residing within the ≤ 2 mm particle size fraction of a soil.

A *soil collection site* refers to the spatial support from which a soil carbon stock value is derived.

A *soil core* describes a discrete portion of the soil within the soil collection site that is extracted with a coring device. The soil core may take the form of the entire soil collection site (e.g., where the soil carbon stock is derived based on only one georeferenced core) or it may represent only a small fraction of a soil collection site (e.g., where multiple cores have been collected from a defined area to estimate the soil carbon stock of the area).

A *sampling location* defines the position within the soil collection site where a soil core was taken.

A *soil layer* refers to the whole soil contained between the soil surface and a depth of soil sampling which must be at least 30 cm.

A **soil sublayer** refers to the whole soil within a soil layer that has a thickness less than the soil layer (e.g., the 0-10 cm, 10-20 cm and 20-30 cm are sublayers of the 0-30 cm soil layer).

The *whole soil* means all the soil material within a soil layer or soil sublayer including gravel and fine earth.

The *fine earth* refers to the fraction of the whole soil having a particle size less than or equal to (\leq) 2 mm.

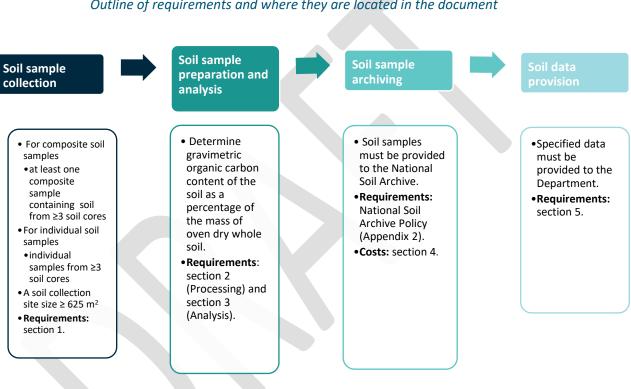
The gravel refers to the fraction of the whole soil having a particle size greater than (>) 2 mm.

The *spatial support* refers to the whole area of soil that a calculated soil carbon stock value is representative of. Where multiple soil cores have been collected over an area from random or stratified random locations and composited together, the spatial support of the single soil carbon stock value derived from the composite is the entire area from which the individual cores were collected. If the multiple soil cores, collected as described above, are kept separate for analysis, but used to calculate an average soil carbon stock and its sampling variance, then the spatial support is also the size of the entire area. If a soil carbon stock value is derived from a single soil core, then the spatial support is the surface area of the core tube used to collect the soil.

Summary of protocols

The way soil samples are collected is critical to defining how derived soil carbon stock values can be used to develop new measurement technologies. Considerations include the size of the area to be sampled, how many soil cores will be collected from the area, whether the samples derived from the soil cores will be kept separate or composited, and the depth of soil sampling.

These protocols are intended to be as flexible as possible to accommodate pre-existing and required differences in approach, whilst at the same time ensuring the validity of comparisons between soil carbon stock estimates derived from new technologies and pre-existing measured soil carbon stock data.



Outline of requirements and where they are located in the document

Soil sample collection 1.

The following requirements are mandatory for new data created under the National Soil Carbon Innovation Challenge, and are recommended for existing data used in validating new measurement technologies, in order to promote data portability and comparisons.

Minimum information requirements:

- 1. The date of soil collection and the organisation responsible for collecting the samples must be provided.
- 2. A geographic information system (GIS) shape file, consistent with the Geocentric Datum of Australia 2020 (GDA2020) latitudes and longitudes with a minimum of 5 decimal places,

must be provided to define the area of the soil collection site to which the measured soil carbon stock provided applies.

- i. The boundaries must have a spatial resolution ≤ 4 m.
- ii. Areas to which the measured soil carbon stock does not apply (e.g., rocky outcrops) must be excluded from the shape file.

Minimum sampling area requirements:

- 3. The spatial support of the soil collection site must be at least (\geq) 625 m² (i.e. 25 m x 25 m).
 - This will help ensure that any new estimation technologies developed can be applied to the area (i.e., the spatial resolution of the new technology is similar or finer than the collection site).
- 4. The sampling location must be cleared of living plants, plant litter and surface rocks, prior to collecting a soil core.

Minimum sampling requirements:

- 5. The soil samples must be collected according to one following approaches:
 - One composite soil sample prepared by mixing all of the soil obtained from ≥3 soil cores collected at random or stratified random locations within the soil collection site or
 - ii. All of the soil collected from ≥3 individual soil cores collected from random or stratified random locations within the soil collection site with the soil from each core being kept separate for preparation and analysis.

The following additional items should be considered when designing a sampling plan:

- a. Where soil cores are composited, it is recommended that ≥3 composite samples are used. Where soil cores are kept separate, it is recommended to collect > 3 cores. Increasing the number of composite samples or individual samples will allow improved estimates of the mean and sampling variance of the soil carbon stock that exists within the soil sampling site.
- b. If desired, the mathematical compositing approach defined in Appendix 10 can be applied to individual cores to derive soil carbon stock values that would be consistent with the collection of composite samples.
- c. Collection of multiple composite samples or multiple soil cores, greater than the minimum required (as outlined above) will allow more representative values to be derived for the mean soil carbon stock and its sampling variance for the sampling site. Deriving more representative values will increase the potential of detecting a statistically significant soil carbon stock change. Additionally, having more representative values for the mean and sampling variance will also allow an assessment of whether or not soil carbon stock estimated using a new technology is within defined confidence bounds of the measured values.
- A soil core must collect all soil within the 0-30 cm soil layer. Sampling can occur to depths
 >30 cm, in which case soil in the 0-30 cm and the >30 cm depth ranges become sublayers of the whole soil core and must be separated, prepared and analysed separately.
- A soil core can be divided into multiple sublayers if desired (e.g., 0-10 cm, 10-20 and 20-30 cm); however, a sublayer must not extend over the 30 cm depth to ensure that a 0-30 cm soil carbon stocks can be calculated.
- 8. Each soil layer or sublayer associated with a soil core must be:

- d. retained as an individual sample for subsequent sample preparation and analysis, or
- e. composited across multiple soil cores taken from the soil collection site.
- 9. The inner cutting edge of the coring device must have a minimum diameter of 38mm.
- 10. The coring device must be clean and can only be cleaned with water.
- 11. Only water can be used to assist with insertion and extraction of the coring device.

2. Soil sample processing

The collected soil samples derived from soil layers or sublayers must be prepared according to this section and as presented in **Error! Reference source not found.**

- 1. Air-dry the entire whole soil sample (single or composite sample of a layer or sublayer) at 40°C to constant weight and record the mass of the air-dried whole soil ($M_{ad_{ws}}$).
- 2. Crush the entire air-dried whole soil to break up any soil aggregates.
- Sieve the entire air-dried whole soil sample to separate the air-dried fine earth (≤ 2 mm soil material) from the gravel (>2 mm soil material). Note that it is important to ensure that no soil aggregates are retained within the gravel.
- 4. Record the mass of gravel retained on the 2 mm sieve (M_a) .
- 5. Thoroughly mix the air-dried fine earth (≤2 mm soil material). The mass of the air-dried fine earth to be retained will be defined by considering all analyses that are to be performed on the sample. For soil carbon stock assessment alone, retaining 100 g would be adequate. However, for the purposes of archiving and completing additional analyses, additional air-dried fine earth will be required.

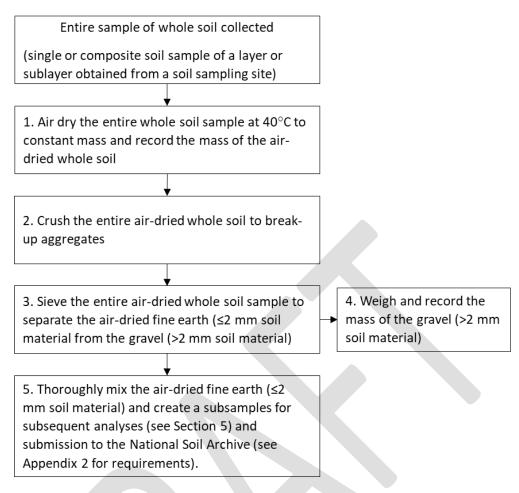


Figure 1. Visual representation of the soil preparation process

If data is to be generated for a composite sample, it is possible to process the individual soil samples to be composited separately to reduce the mass of soil processed at any one time. If soil samples to be composited are processed separately, the masses of air-dried whole soil (Step 1) and the masses of gravel (Step 4) should be added together to give the final values to be used for the composite sample. In addition, the air-dried fine earth material from each sample needs to be calculated and mixed as part of Step 5. Also the thickness of the composite sample needs to be calculated as the sum of the thicknesses of each individual soil sample. It is also possible to process all samples individually, analyse them individually, and then mathematically calculate the appropriate values that would have been obtained if the samples were composited (see Appendix 1).

3. Soil sample analysis and calculations to derived soil carbon stock values

All soil analyses to be applied to soil samples collected within the project must be completed by a laboratory accredited by ASPAC for the analyses required to be performed.

a. Analysis of the content of organic carbon in the oven dried whole soil

It is a requirement to determine and express the gravimetric organic carbon content of the soil as a percentage of the mass of oven dry whole soil. This will involve determining the gravimetric organic carbon content of the fine earth material, determining the water content of the fine earth material, and determining the gravimetric gravel content of the whole soil as depicted in Figure 2 (Boxes 1, 2 and 6, respectively). The analytical process, data collected, and all calculations provided are consistent with the ERF 2021 soil carbon method but do not use the same identifiers within the equations and have combined multiple equations into single equations.

i. Analysis of the gravimetric organic carbon content of the air-dried fine earth

The analysis of the gravimetric organic carbon content of the air-dried fine earth ($OC_{ad_{fe}}$) obtained for each soil sample must be completed by (and as indicated in Box 1 of Figure 2):

- 1. Obtain a subsample (≥10 g) of the homogenised air-dried fine earth (Box 5, Error! Reference source not found.).
- 2. Grind the subsample to ≤ 0.5 mm.
- 3. Determine the gravimetric organic carbon content of the air-dried fine earth $(OC_{ad_{fe}})$ by dry combustion analysis (method 6B2 for soils without carbonate and method 6B3 without the removal of charcoal for soils with carbonate (Rayment and Lyons 2011)).
- 4. Express the organic carbon content as a percentage of the oven-dry mass of air-dried fine earth.

ii. Analysis and calculation of the gravimetric water content of the air-dried fine earth soil material

The gravimetric water content of the air-dried fine earth soil material obtained for each soil sample must be completed by (as indicated in Box 2 of Figure 2):

- Obtain a subsample (≥20 g) of the air-dried fine earth (Box 5, Error! Reference source not found.).
- Weigh and record the mass of a container to which the air-dried fine earth will be added to ≥2 decimal places.
- 3. Weigh and record the mass of air-dried fine earth and container to ≥ 2 decimal places.
- 4. Oven dry the air-dried fine earth at 105°C to constant mass.
- 5. When the sample heated to 105 °C is removed from the oven, place it into a desiccator to allow it to cool before weighing it.
- 6. Weigh and record the mass of the oven-dried fine earth and container to ≥ 2 decimal places
- 7. Calculate the gravimetric water content of the air-dried fine earth to 4 decimal places according to Equation [1].

$$W_{ad_{fe}} = \frac{(M_2 - M_3)}{(M_3 - M_1)}$$
[1]

where:

- $W_{ad_{fe}}$ is the gravimetric water content of the air-dried fine earth for the layer or sublayer (g water/g oven dry fine earth).
- M_1 is the mass of the container used to hold the air-dried fine earth during the oven drying (g).
- M_2 is the mass of air-dried fine earth and container (g).

 M_3 is the mass of oven-dried fine earth and container (g).

iii.

Calculation of the gravimetric gravel content of the oven-dry whole soil

The gravimetric gravel content of the oven-dry whole soil must be calculated according to Equation [2]:

$$G_{od_{ws}} = \frac{M_g}{M_{od_{ws}} + M_g} = \frac{M_g}{\left(\frac{(M_{ad_{ws}} - M_g)}{(1 + W_{ad_{fe}})} + M_g\right)}$$
[2]

where:

- $G_{od_{ws}}$ is the gravimetric gravel content of the oven-dry whole soil for the layer or sublayer (g gravel/g oven-dry whole soil).
- M_g is the mass of gravel retained on the 2 mm sieve when the entire layer or sublayer was sieved from Box 4 in **Error! Reference source not found.** (g).
- $M_{od_{ws}}$ is the oven-dry mass of the whole soil in the layer or sublayer (g).
- $M_{ad_{ws}}$ is the measured air-dried mass of the entire whole soil for the layer or sublayer from Box 1 in **Error! Reference source not found.** (g).

 $W_{ad_{fe}}$ is the gravimetric water content of the air-dried fine earth for the layer or sublayer calculated according to Equation [1].

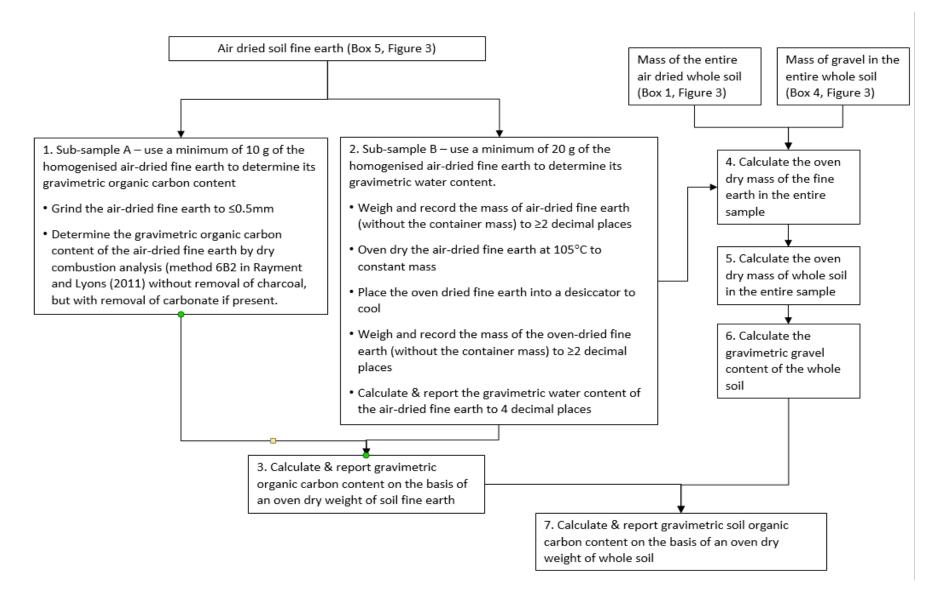


Figure 2. Schematic representation of the steps required for the determination the organic carbon content of the whole soil in the collected soil samples.

iv. Calculation of the gravimetric organic carbon content of the oven-dry whole soil

The gravimetric carbon content of the oven-dry whole soil must be calculated according to Equation [3]:

$$OC_{od_{ws}} = OC_{ad_{fe}} \times \left(1 + W_{ad_{fe}}\right) \times \left(1 - G_{od_{ws}}\right)$$
[3]

where:

- $OC_{od_{ws}}$ is the gravimetric organic carbon content of the oven-dry whole soil for the layer or sublayer (g soil organic carbon/100 g oven dry whole soil)
- $OC_{ad_{fe}}$ is the gravimetric organic carbon content of the oven-dry fine earth for the layer or sublayer obtained from the dry combustion analysis (g soil organic carbon/100 g oven-dry fine earth)
- $W_{ad_{fe}}$ is the gravimetric water content of the air-dried fine earth for the layer or sublayer calculated according to Equation [1] (g water/g oven dry fine earth).
- $G_{od_{ws}}$ is the gravimetric gravel content of the oven dry whole soil for the layer or sublayer calculated according to Equation [2] (g gravel/g oven dry whole soil)

b. Calculation of the oven-dry bulk density of the whole soil

1) The volume of soil collected for the sample must be calculated according to Equation [4]

$$V = \pi \times r^2 \times t \tag{4}$$

where:

V is the volume of soil collected for the layer or sublayer (cm^3) .

r is the radius of the core tube used to collect the soil core (cm).

- *t* is the thickness of the soil layer or sublayer sampled (cm). For a composite sample, *t* is calculated as the average thickness across all samples that were composited.
- 2) The oven-dry bulk density of the whole soil must be calculated according to Equation [5]

$$BD = \frac{M_{od_{ws}}}{V} = \frac{\left(\frac{\left(M_{ad_{ws}} - M_g\right)}{\left(1 + W_{ad_{fe}}\right)} + M_g\right)}{V}$$
[5]

where:

- *BD* is the oven dry bulk density of the whole soil in the layer or sublayer (g oven-dry whole soil/cm³ soil collected)
- $M_{od_{ws}}$ is the oven-dry mass of the whole soil in the layer or sublayer (g).
- *V* is the volume of soil collected for the layer or sublayer calculated according to Equation [4] (cm³)
- $M_{ad_{ws}}$ is the measured air-dried mass of the entire whole soil for the layer or sublayer from Box 1 in **Error! Reference source not found.** (g).
- $W_{ad_{fe}}$ is the gravimetric water content of the air-dried fine earth for the layer or sublayer calculated according to Equation [1].
- M_g is the mass of gravel retained on the 2 mm sieve when the entire layer or sublayer was sieved from Box 4 in **Error! Reference source not found.** (g).

c. Calculation of the organic carbon stock in the whole soil

1) The soil carbon stock associated with the collected soil sample must be calculated according to Equation [6].

$$SOC = OC_{od_{ws}} \times BD \times t$$
 [6]

where:

SOC is soil organic carbon stock within an individual soil layer or sublayer (tonnes of soil carbon/ha).

- OC_{odws} is the gravimetric concentration of organic carbon determined for the layer or sublayer according to Equation [3] (%, g organic carbon/100 g oven dry whole soil).
- *BD* is the oven-dry bulk density determined for the layer or sublayer according to Equation [5] (g oven dry whole soil/cm³ whole soil).
- t is the thickness of the layer or sublayer sampled (cm). For a composite sample, t is calculated as the average thickness across all samples that were composited.

2) If the 0-30 cm soil layer was sampled in sublayers, the soil carbon stock of the 0-30 cm layer must be calculated according to Equation [7]. If the 0-30 cm soil layer was sampled as a single layer, then $SOC_{30} = SOC$.

$$SOC_{30} = \sum_{i=1}^{n} SOC_i$$
^[7]

where:

- SOC_{30} is the stock of soil organic carbon present in the 0-30 cm soil layer (tonnes of soil carbon/ha).
- SOC_i is the stock of soil organic carbon present within sublayer *i* calculated according to Equation [6] (tonnes of soil carbon/ha).

i is a designator for the soil sublayer.

n is the total number of sublayers sampled within the 0-30 cm soil layer.

4. Submission of soil samples to Australia's National Soil Archive

A portion of each fine earth soil sample collected and analysed as part of the projects supported by the National Soil Carbon Innovation Challenge must be submitted to the <u>National Soil Archive</u>. For details and instructions concerning the submission of samples to the archive refer to Appendix 2.

Costs

There are costs associated with archiving soil samples. This is to partially cover the costs of stock taking, labelling, transfer and storage. The CSIRO National Soil Archive is a small organisation that holds many tens of thousands of specimens. In many circumstances, the Archive could not provide value-added services free of charge to researchers without reducing the amount of effort going into making soil specimens available for further research.

For the purposes of the National Soil Carbon Innovation Challenge, the soil archiving costs can be estimated on the following basis:

- 50 samples or less: \$40/sample
- 51 samples or more: \$30/sample.

It is recommended to budget for Archive Submission Costs when planning your project. Additional costs which should be factored in, include transport and freight, data processing, packaging and preparation of quarantine certificates in accordance with biosecurity requirements. Further details are in Appendix 2.

5. Data provision

Additionally, as noted in the Grant Agreement and Grant Guidelines, participants in the Innovation Challenge must provide to the Commonwealth the following data (Required Data) obtained by the protocols outlined in sections 1–3:

- 1. soil organic carbon stock measurements at a particular location, as determined by the Commonwealth and notified to the Grantee from time to time; and
- 2. copies of submission request form for submission of soil specimens to the CSIRO National Soil Archive.

For the purposes of point 1, the following information must be provided to the Department.

For each soil collection site:

- 1. Date of sampling
- 2. Organisation that collected the samples
- 3. Size (area)

- 4. Shape File
- 5. Indication of site type, that corresponds to an analysis of either:
 - a. Single composite sample for a 0-30cm layer from multiple cores
 - b. Single composite sample for each sublayer from multiple cores
 - c. Multiple composite samples for the 0-30cm layer from multiple cores
 - d. Multiple composite samples for each sublayer from multiple cores
 - e. Individual soil core for the 0-30cm layer
 - f. Each sublayer of an individual soil core
 - g. The 0-30cm layer from multiple individual soil cores
 - h. Sublayers from multiple individual soil cores.

Depending on the site type, for each sample within a soil collection site (layer or sublayer):

- 6. Number of cores
- 7. GPS Coordinates for each core/sample (if applicable)
- 8. Upper and lower boundary of the soil layer or sublayer sampled (cm)
- 9. Diameter of the core tube used to collect the soil (cm)
- 10. Mass of the entire air dried whole soil sample
- 11. Mass of gravel (>2mm soil material) in the entire air-dried whole soil sample
- **12.** Gravimetric organic carbon content of the air-dried fine earth (g OC/100 g air-dried fine earth)
- 13. Mass of the container used when determining water content of the air-dried fine earth (g)
- **14.** Mass of the container + air-dried fine earth used when determining the water content of the air-dried fine earth (g)
- **15.** Mass of the container + oven-dried fine earth used when determining the water content of the air-dried fine earth (g)
- **16.** Calculations for SOC stock of the sample:
 - a. Gravimetric water content of the air-dried fine earth (g water/g oven-dry fine earth) $(W_{ad_{fe}})$
 - b. Gravimetric gravel content of the oven dry whole soil (g gravel/g oven dry whole soil) (G_{od_ws})
 - c. Gravimetric organic carbon content of the oven-dry whole soil (g OC/100g oven-dry whole soil)(OC $_{od_ws}$)
 - d. Oven dry bulk density of the whole soil (g oven dry whole soil/cm3 whole soil) (BD_{ws})
 - e. Soil carbon stock of the layer or sublayer sampled (tonnes soil carbon/ha) (SOC).

To the extent that any Intellectual Property Rights subsist in the Required Data:

- a. the participants in the Innovation Challenge will retain those rights; and
- b. the participants in the Innovation Challenge must grant to the Commonwealth, and to any other potential user of the Required Data, an irrevocable, worldwide licence (including a right to sublicense) to use, publish, modify, access, communicate, exploit and commercialise the Required Data for any purpose.

Further, participants in the Innovation Challenge must agree that the Commonwealth may provide the Required Data to any subcontractor or nominee to host on, and provide access to, the Required Data through the Australian Soil Resource Information System (ASRIS) or any other system that may be notified by the Commonwealth to the Grantee from time to time.

6. References

Rayment GE, Lyons DJ (2011) 'Soil Chemical Methods - Australasia.' (CSIRO Publishing: Collingwood, Vic, Australia)

Appendix 1. Equations to be used to perform mathematical compositing.

Mathematical compositing allows soil samples obtained from individual soil cores to be collected, prepared, and analysed separately and to then derive a soil carbon stock consistent with what would have been obtained had the samples been composited.

1) The gravimetric organic carbon content of the composite soil sample must be calculated from the data derived from the soil layer or sublayers associated with each individual soil core to be composited according to Equation [8].

$$OCmc_{od_{ws}} = \frac{\sum_{i=1}^{n} \left(OC_{od_{ws},i} \times M_{od_{ws},i} \right)}{\sum_{i=1}^{n} \left(M_{od_{ws},i} \right)} \times 100$$
[8]

where:

- $OCmc_{od_{ws}}$ is soil gravimetric organic carbon content of the mathematical composite sample (%, g organic carbon /100g oven dry whole soil).
- $OC_{od_{ws},i}$ is the gravimetric concentration of organic carbon determined for the layer or sublayer of soil core *i* calculated according to Equation [3] (%, g organic carbon/100 g oven dry whole soil).
- $M_{od_{ws},i}$ is the oven-dry mass of whole soil collected for the layer or sublayer of core *i* calculated according to numerator of Equation [5] (g oven dry whole soil).
- *i* is a designator for each core from which a soil layer or sublayer was derived.
- n is the total number of cores from which layers or sublayers are to be composited.

2) The bulk density of the composite soil sample must be calculated from the data derived from the soil layers or sublayers associated with each individual soil core to be composited according to Equation [9].

$$BDmc = \frac{\sum_{i=1}^{n} (M_{od_{ws,i}})}{\sum_{i=1}^{n} (V_i)}$$
[9]

where:

BDmc is oven dry whole soil bulk density of the mathematical composite sample (g oven dry whole soil /cm³ oven dry whole soil).

- $M_{od_{ws},i}$ is the oven-dry mass of soil collected for the layer or sublayer of core *i* calculated according to numerator of Equation [5] (g oven dry whole soil).
- V_i is the volume of soil collected for the layer or sublayer of soil core *i* calculated according to Equation [4] (cm³ oven dry whole soil).
- *i* is a designator for each core from which a soil layer or sublayer was derived..

n is the total number of cores from which layers or sublayers are to be composited.

3) The average thickness of soil collected for the mathematical composite sample must be calculated according to Equation [10]

$$tmc = \frac{\sum_{i=1}^{n} t_i}{n}$$
[10]

where:

tmc is the average thickness of soil collected across all the layers or sublayers included in the composite sample (cm).

 t_i is the thickness of soil collected for the layer or sublayer of soil core i (cm).

i is a designator for each core from which a soil layer or sublayer was derived.

n is the total number of cores from which layers or sublayers are to be composited.

4) The soil carbon stock associated with the mathematical composite soil sample must be calculated according to Equation [11].

$$SOCmc = OCmc_{od_{ws}} \times BDmc \times tmc$$
^[11]

where:

- *SOCmc* is soil organic carbon stock of the mathematical composite for the layer or sublayer (tonnes of soil carbon/ha).
- $OCmc_{od_{ws}}$ is the gravimetric concentration of organic carbon determined for the mathematical composite for the layer or sublayer according to Equation [8] (%, g organic carbon/100 g oven dry whole soil).
- *BDmc* is the oven-dry bulk density determined for the mathematical composite for the layer or sublayer according to Equation [9] (g oven dry whole soil/cm³ whole soil).
- *tmc* is the average thickness of the layer or sublayer calculated according to Equation [10] (cm).

5) If the 0-30 cm soil layer was sampled in sublayers, the soil carbon stock of the 0-30 cm layer must be calculated according to Equation [12]. If the 0-30 cm soil layer was sampled as a single layer, then $SOCmc_{30} = SOCmc$.

$$SOCmc_{30} = \sum_{i=1}^{n} SOCmc_i$$
[12]

where:

- $SOCmc_{30}$ is the soil organic carbon stock of the mathematical composite present in the 0-30 cm soil layer (tonnes of soil carbon/ha).
- $SOCmc_i$ is the stock of soil organic carbon present within sublayer *i* calculated according to Equation [11] (tonnes of soil carbon/ha).

i is a designator for the soil sublayer within the 0-30 cm soil layer.

n is the total number of sublayers sampled within the 0-30 cm soil layer.

Appendix 2. Submission of soil samples to Australia's National Soil Archive

<u>These Policies are developed by CSIRO and sourced from the team which manages Australia's</u> <u>National Soil Archive.</u>

Policy on the submission of soil specimens

The <u>CSIRO National Soil Archive</u> stores around 71,000 soil specimens. Archiving of stored soil specimens involves stock taking, labelling, transfer of soil material, and storage. For each archived specimen archive information is available in the database: how much material is present, whether as whole soil or fine earth, and on which shelf the specimen can be found. The label on every archived specimen indicates the agency, project, observation ID, site ID, horizon (name), depth, sample (where there are more specimens per horizon) and layer. Together these variables provide a unique ID for every specimen, which is expressed in the barcode on the label. In 2009, 22,000 of the 71,000 stored soil specimens had been archived. Archiving of stored soil specimens is on-going.

Policy statement

To maintain important historic and contemporary soil specimens from around Australia for future analysis, the CSIRO National Archive supports continuing submission of additional collections of soil specimens to expand the Archive collection where possible.

Procedure

Specimens submitted to the CSIRO National Soil Archive are required to meet a number of criteria to become part of the collection. Organisations wanting to submit soil specimens need to describe (using the Submission Form), the type and number of specimens, the amount of material per specimen, the amount of data, the data format, the research to which these specimens contributed and justifications for archiving. Upon receiving this request, the CSIRO National Soil Archive Committee will review and assess the submission.

Priority will be given to specimens:

- supporting priority initiatives (e.g. Soil Organic Carbon Monitoring)
- representing important landscapes
- addressing knowledge gaps, either temporally or spatially
- used for widely publicised research
- with data available in NatSoil (Microsoft Access)

Specimens submitted to the CSIRO National Soil Archive may be accepted if they are within the scope of CSIRO's scientific interests and in good condition, have adequate documentation and if the CSIRO National Soil Archive can provide for their care. To assist with the capture and storage of soil information in the appropriate format, the archive provides data entry (NatForm) and storage (NatSoil) databases (Microsoft Access) upon request. Documentation must be submitted to the Database Manager at the time the specimens are submitted, or before.

More information on the National Soil Archive: <u>https://www.csiro.au/en/research/natural-environment/land/soil-archive</u>

REQUIRED DATA

LOCATION

- Coordinates of the site location where the specimens were obtained
- The datum of the coordinates. In the GDA 94 datum, latitudes and longitudes, with latitudes as negative in the Southern Hemisphere. In the Universal Transverse Mercator (UTM) coordinate system, the grid zone, northing, easting and datum are required.
- The method used to acquire the coordinates needs to be recorded as one of the following: map reference, GPS or Survey.

TYPE OF SOIL OBSERVATION

The type of soil observation from which the specimens are extracted needs to be recorded as one of the following: soil pit, existing vertical exposure, relatively undisturbed soil core or auger boring (NCST, 2009 p. 147).

DEPTH INTERVAL

Upper and lower depth (m) of the soil specimens.

COLLECTOR

Name and contact details of the person responsible for the specimens.

DATE COLLECTED

DESIRED DATA

MEASURED SOIL CHEMISTRY PROPERTIES, where known

MEASURED SOIL PHYSICAL PROPERTIES, where known

METHOD OF ANALYSES, must be specified for each measurements result or field value

The method of analysis must be recorded and this must reference a published source (e.g. Rayment and Higginson, 1992, McKenzie et al. 2002). If a published source is unavailable, complete documentation is required.

DATA FORMAT: Microsoft Access, Microsoft Excel or ASCII

REFERENCES TO RELEVANT REPORTS AND JOURNAL PAPERS

OPTIONAL DOCUMENTATION

The presence of morphological descriptions greatly increases the utility of archived soil specimens. In addition to the mandatory dataset outlined above, the CSIRO National Soil Archive Committee suggests the following attributes are collected during sampling, in accordance with the Australian Soil and Land Survey Field Handbook (3rd ed)(page numbers are indicated below).

HORIZONS

Horizons (A1, B2, etc) are recorded with upper and lower depth (m) (p. 148)

HORIZON COLOURS AND MOTTLES (p.159)

FIELD TEXTURE (p.161)

COARSE FRAGMENTS (p.170)

STRUCTURE (p.171)

FIELD PH

SOIL PERMEABILITY

DRAINAGE

AUSTRALIAN SOIL CLASSIFICATION ORDER AND SUBORDER

Physical requirements for acceptance into the Archive

Soil specimens are recommended to be a minimum of 200 g and maximum of 1 kg in weight.

Soil specimens need to be:

- non-toxic
- air-dried at 40 degrees Celsius or oven-dried
- stored in air-tight containers

Specimens that do not meet these requirements will not be accepted.

Data requirements for acceptance into the Archive

Supporting documentation is of primary importance to the scientific value of specimens. Pertinent data must be documented and **clearly linked to each specimen**. Detailed documentation ensures that a specimen's field data, history of use, physical condition and identification are known. These records may actually have to substitute for a specimen should it run out. See text box on previous page for the data submission criteria.

When the Submission Request Form has been received, the Steering Committee of the CSIRO National Soil Archive convenes to evaluate the submission. After the submitting organisation has been advised of a positive assessment, details of data and specimen transfer are arranged with the archivist. The contributing organisation is responsible for documentation and must provide the CSIRO National Soil Archive with all available data and publications, while realising that the data will eventually be freely accessible to future archive users. Specimen transport to the CSIRO National Soil Archive will be at the submitting agency's cost unless otherwise negotiated.

Once the transfer is completed, the specimens will become and remain property of the CSIRO National Soil Archive. Specimens cannot be given out as 'permanent loans' as such arrangements are ambiguous with respect to control and financial responsibility. Submitting organisations will be able to access specimens that were previously their property according to the Policy on Specimen Use.

Submission Request Form

For the proposed submission of soil specimens to the CSIRO National Soil Archive

The mission of the CSIRO National Soil Archive is to provide facilities and protocols for conserving the long-term scientific value of soil specimens and associated soil data, and to make these specimens and their data available for public research, both now and into the future when new analytical techniques may be brought to bear on the specimens.

The information to be provided below will allow the CSIRO National Soil Archive Committee to review and assess the proposed submission. Please provide the following:

- 1. Name, Organisation, Address, Email Address and Phone number.
- 2. How many specimens are to be submitted, from how many sites?
- 3. How many cubic metres does the collection currently occupy?
- 4. How many grams of material are available, on average, per specimen?
- 5. Is the soil material sieved to 2 mm? Does whole soil or coarse fragment materials exist?
- 6. In what type of airtight containers are the specimens currently held?
- 7. Are the specimens non-toxic?
- 8. Are the specimens air- or oven-dried? If oven-dried, to what temperature?
- 9. In what region were the specimens sampled?
- 10. Is accurate location information available?
- 11. Is accurate sampling information available?
- 12. What analyses were performed? Provide method references.
- 13. What data are available?
- 14. In what format is the data currently stored?
- 15. What data use or licensing requirements are in place, if any?
- 16. What was the research context?
- 17. What is the justification for these soil specimens to be part of a national archive?
- 18. Provide references or copies of published work relevant to the specimens.

Policy on the use of soil specimens in the CSIRO National Soil Archive

The CSIRO National Soil Archive balances the preservation of historic soil specimens and associated soil data (for future research) with the role of making these soil specimens and associated data available for current public research. Potential users of the archived soil specimens must adhere to the Archive guidelines as a matter of common courtesy and ethical responsibility. If the guidelines are not followed, the CSIRO National Archive Committee reserves the right to halt further use of the specimens until inconsistencies are solved and guidelines are adhered to.

Policy statement

The archived soil specimens have been collected for the purposes of scientific research. Therefore they are expected to be subject to treatments and analytical procedures required to advance science, at the discretion of the CSIRO National Soil Archive Steering Committee, even if this results in consumption or alteration of the specimen. Users need to be keenly aware however, that the quantity of soil material per specimen is limited, and exercise particular restraint when analysis is destructive.

To ensure the integrity of the CSIRO National Soil Archive, subsampling is taken very seriously as great care is needed to ensure sufficient material is available for users in future decades. As such our priorities are:

- 1. to maintain the chemical and physical integrity of the specimens;
- 2. to maintain documentary records of the specimens;
- to document the use of the specimens, and promote new research results (e.g. calibration method of new analytical methods, survey of previously measured electrolytes, confirmation of previous measurements);
- 4. to report any changes or factors that affect future use of the specimens (e.g. quantity available, storage conditions, accidents);
- 5. to ensure acknowledgement of used archive specimens in any resulting publications

Use of Specimens

Access to soil specimens may be provided for research purposes at the discretion of the Steering Committee and is in part dependent on the archive user's past adherence to Archive Policies. Loan requests may be declined, in whole or in part, if the Steering Committee considers the request excessive, disruptive or incompatible with existing or proposed Archive programs, or suspects that specimens might be subject to misuse, damage or deterioration.

Procedure

For detailed information about the specimens in our collection, and their possible availability for further research, contact the CSIRO National Soil Archivist. Arrangements can also be made for browsing and querying the NatSoil database that contains information on all archived specimens. Following the lodging of the User Request Form, the borrower will be provided with a copy of the CSIRO National Soil Archive Policies and be informed of the likely costs, if any.

- 1. The CSIRO National Soil Archive Committee reviews the information on the User Request Form.
- 2. If the request is approved, an Agreement is drafted and signed by the borrowing agency.
- 3. Subsampling is normally done by archive staff, and the subsamples are sent to the requesting organisation.

- 4. The normal loan period is 12 months, but an extension may be granted upon written request.
- 5. Specimens are not to be forwarded to third parties without advance permission from the Archive.
- 6. Both the CSIRO National Soil Archive and the original sources of the collection of soil specimens are to be cited in any publications resulting from research on Archive specimens.
- 7. Any new research pertaining to the soil archive specimens is added to the NatSoil database to improve the quality of collection.
- 8. Any soil material remaining from the specimens must be returned to the Archive as back-up material, with a copy of the Agreement, as soon as possible after the completion of the study.
- 9. On return, a loan should be sent by traceable freight, and carefully packaged to prevent damage in transit.

Note that loans are made to institutions, not to individuals. The head of the borrowing organisation will take responsibility for the security, documentation and safe return of all specimens, and for adherence to the conditions of the loan.

For each loan, an agreement must be signed by both parties. The agreement will itemise the materials loaned and analyses to be performed, and state the duration and other conditions of the loan, including limitations of the purpose of the loan, responsibilities of the parties, and options for renewal. It will also form a contract between CSIRO and the borrower's institution. Loans will not be dispatched until the conditions stated in the agreement are agreed to.

Use of Data

Access to soil data may be provided for educational and research purposes when approved by the Steering Committee. The CSIRO National Soil Archive has ownership of the data and access to it is at the discretion of the Steering Committee. Use of the information is subject to CSIRO's collaborator and intellectual-property agreements. Upon approval of the data request, data records will be made available through applications developed for search and retrieval of the database.

Costs

Organisations may be charged Archive Access Costs of \$10 per subsampled specimen, particularly for large user requests, to partially cover the costs of retrieval, subsampling, packaging and transport. The CSIRO National Soil Archive is a small organisation that holds many tens of thousands of specimens. In many circumstances, the Archive could not provide value-added services free of charge to researchers without reducing the amount of effort going into the archiving of more specimens. It is recommended to budget for Archive Access Costs when planning research based on archived soil specimen.

User Request Form – Soil Specimens

For the proposed use of soil specimens from the CSIRO National Soil Archive

The mission of the CSIRO National Soil Archive is to provide facilities and protocols for conserving the long-term scientific value of soil specimens and associated soil data, and to make these specimens and their data available for public research, both now and into the future when new analytical techniques may be brought to bear on the specimens.

Users of the archive need to be keenly aware that the quantity of specimen is limited and great care is needed to ensure sufficient material is available for users in future decades and centuries.

Therefore we ask that you adhere to our guidelines as a matter of common courtesy and ethical responsibility. If we observe that the guidelines are not being followed, we maintain the right to halt further use of the specimens until inconsistencies are solved and guidelines adhered to.

The information you provide below will allow the CSIRO National Soil Archive Committee to review and assess your submission. Please provide the following:

- 1. Name, Organisation, Address, Email Address and Phone number.
- 2. How many specimens are requested?
- 3. From which sites, which original sampling agency, which original sampling project, which depth? List or append the results of the NatSoil queries in the following format: Agency, project code, Site ID, Observation ID, Horizon no, Sample no.
- 4. How many grams of material are requested per specimen? In some cases the requested amount may exceed allowable limits.
- 5. Choose a preference for either whole soil or fine earth (<2 mm) (if available).
- 6. For what research do you plan to use these specimens? Append project proposal
- 7. List analyses to be conducted and whether they are destructive.
- 8. Who is responsible for subsampling? For large orders, CSIRO National Soil Archive staff may require assistance by staff of the agency requesting the specimens.
- 9. Suggest a method of sub sampling. Procedure to prevent contamination must be detailed and strictly followed.

User Request Form – Soil Data

For the proposed use of soil data from the CSIRO National Soil Archive

The mission of the CSIRO National Soil Archive is to provide facilities and protocols for conserving the long-term scientific value of soil specimens and associated soil data, and to make these specimens and their data available for public research, both now and into the future when new analytical techniques may be brought to bear on the specimens.

The information you provide below will allow the CSIRO National Soil Archive Committee to review and assess your submission. Please provide the following:

- 1. Name, Organisation, Address, Email Address and Phone number.
- 2. What data is requested?
- 3. From which sites, which sampling agency, which sampling project, which depth?
- 4. For what research do you plan to use this data? Append project proposal.