

Standard Operating Procedure

Soil Inorganic Carbon by Modified Pressure Calcimeter

Scope

This document outlines the measurement of inorganic carbon (C) concentrations in mineral soil samples by pressure calcimeter. The measurement procedure is described in general terms, as the operation of till vary. This methodology repeats the sample preparation of Total Carbon and the same fine ground sample can be used and Inorganic C should be reported on an oven-dry (105 °C) soil mass basis.

Equipment

2-mm (10 mesh) sieve
Air-drying facility (minimum temperature: 30 °C)
Coarse-grinding apparatus (e.g., rolling pin, flail mill)
Drying tins
Drying oven
Desiccator
Analytical balance (± 0.1 mg sensitivity)
Tin foil cups/capsules
180- μm (80 mesh) sieve
Fine-grinding apparatus (e.g., ball mill, agate mortar and pestle)
Consult methods-specific SOP for additional materials

Reagents

Consult instrument-specific SOP for reagents (catalysts, carrier gases, etc.)

Procedure

1. Air-dry the bulk soil sample at 30-40 °C. Transfer the sample to a paper bag or spread out on a tray before drying. Forced air circulation is recommended to remove humidity. Soils that form very strong aggregates upon drying (e.g., Andisols) should be dried at lower temperatures (<25 °C). When in doubt, consult the sample submitter.
2. Break up the bulk soil sample with a rolling pin or coarse-grinding mill (e.g., flail mill) and pass through a 2-mm sieve. When working with large samples (e.g., composite samples), a subsample may be taken prior to sieving using a sample splitter or by spreading the soil sample across a flat surface, dividing the soil into sections (e.g., a rectangular grid or pie), and collecting all material within multiple non-adjacent sections. Visually verify that the proportions

of large to small aggregates/particles are similar for the subsample and the bulk soil sample, as soil aggregates will separate by size during sample handling.

3. Weigh and record the mass of an empty, labeled drying tin. Add 10.0 g sieved soil to drying tin and record total mass of tin and soil. Dry the 10.0 g subsample in an oven at 105 °C for a minimum of 24 hours. After drying, place sample in desiccator to cool to room temperature, and record the total oven-dry mass of the tin and soil.
4. Finely grind a subsample (minimum 1.0 g) of sieved soil to ensure sample homogeneity and measurement repeatability. The method of grinding should **leave the subsample fine enough to pass a 180- μ m sieve**.¹ (Note: passing a 180- μ m sieve implies that all coarse and medium sand and a fraction of the fine sand have been pulverized.) The desired maximum particle size can be achieved using various mill designs (e.g., ball, cross beater, roller) or by hand-grinding in an agate mortar and pestle. Verify the effectiveness of the grinding method at least once by grinding and sieving (180- μ m sieve) a test sample with >10% sand.

The following steps concern sample weighing, combustion, and detection of combustion products. For details, consult the instrument-specific SOP.

5. Tare a tin foil cup, including any additives indicated by the instrument SOP (e.g., tungsten oxide). Add the SOP-specified mass of soil sample to the cup, record mass, and seal tin foil cup. Enter sample mass into instrument software.
6. Combust sample and measure combustion products according to instrument SOP. Combustion temperature should be at least 950 °C. (Note: temperatures of at least 1300 °C are recommended to maximize recovery of carbon from the thermal decomposition of carbonates.²) The most common elemental analyzers measure N₂ released by combustion using a thermal conductivity cell and use either an infrared gas analyzer or a thermal conductivity cell to measure CO₂.

Calculations

1. $AD/OD = (\text{mass of air-dry soil and tin} - \text{mass of empty tin}) / (\text{mass of oven-dry soil and tin} - \text{mass of empty tin})$. Commonly referred to as a moisture correction.
2. $C = C_{ar} \times AD/OD$
where:
C = C (%), oven-dry basis
 $C_{ar} = C$ (%) as reported by instrument software
3. $N = N_{ar} \times AD/OD$
where:
N = N (%), oven-dry basis
 $N_{ar} = N$ (%), as reported by instrument software

QA/QC

1. After every 20 samples, run a duplicate analysis on one of the preceding 20 samples selected at random. Include data for QC duplicates in final data report. The difference in C concentration

between duplicate readings of the same sample should be less than 20% of the average of the two readings.³

1. In addition to instrument-specific calibrations, which may not use soil materials, analysis of a minimum of four soil standard reference materials is recommended every six months. Soil standards that have been analyzed at an external laboratory will be provided by SHI. Additional soil standards can be developed in-house but must be analyzed at least once at an external laboratory to obtain independent reference values.

References

1. USDA NRCS. (2014). Kellogg Soil Survey Laboratory Methods Manual, Soil Survey Investigations Report No. 42, Version 5.0, Lincoln, NE.
2. Wright, A. F., & Bailey, J. S. (2001). Organic carbon, total carbon, and total nitrogen determinations in soils of variable calcium carbonate contents using a Leco CN-2000 dry combustion analyzer. *Communications in Soil Science and Plant Analysis*, 32(19–20), 3243–3258. <https://doi.org/10.1081/CSS-120001118>
3. Soil Environmental Chemistry Program, The Ohio State University. (nd). Standard Operating Procedure: Soil Organic Carbon and Nitrogen Determination by Dry Combustion, Version 5.0. Columbus, Ohio.

NOTE

This SOP was developed by SHI, for SHI communication, and developed after “Soil Total Carbon and Nitrogen Determination by Dry Combustion” (v5) of Soil Environmental Chemistry Program, The Ohio State University; Method 4H2a1-3a1 of the Kellogg Soil Survey Laboratory Methods Manual (v5, 2014). For any specific questions, contact Dr. Liz Rieke erieke@soilhealthinstitute.org.