

Standard Operating Procedure

Soil Total Carbon and Nitrogen by Dry Combustion

Scope

This document outlines the measurement of total carbon (C) and nitrogen (N) concentrations in mineral soil samples by dry combustion. The measurement procedure is described in general terms, as the operation of individual dry combustion instruments will vary by brand, model, and sensor technology. This SOP is intended to be used in conjunction with an instrument-specific SOP. Results are reported as percent C and N 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

Consult instrument-specific SOP for additional materials

Optional:

180- μ m (80 mesh) sieve

Fine-grinding apparatus (e.g., ball mill, agate mortar and pestle)

Reagents

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

Procedure

1. Air-dry the bulk soil sample at 30-40 °C for a minimum of 48 hours. 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. Any soil aggregates retained on top of the sieve must be further ground to avoid fractionating the sample. Excluding aggregated soil from the analysis may bias the results. 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. Prepare a subsample as follows to determine a correction factor for residual moisture present in the sample after air-drying. Weigh and record the mass of an empty, labeled drying tin. Add 10.0 ± 1.0 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. (Note: residual moisture will fluctuate with changes in room temperature and relative humidity. Dry combustion analysis must be conducted within 48 hours of moisture correction determination; if more than 48 hours elapse prior to measurement, a new moisture correction should be determined).
4. Optional: if a fine-grind is requested prior to analysis, 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 instrument-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. If the sample submitter requests that this value be included in their data report, round to a minimum of three decimal places (e.g., 1.028).
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 the tenth previous sample (e.g., rerun sample #10 after sample #20, sample #30 after sample #40, etc.). 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
2. 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. 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