Total Organic Carbon (TOC / Foc) in Soil/Sediment by Combustion (PBM)

Parameters

Total Organic Carbon (TOC)
Fraction Organic Carbon (Foc)

Analytical Method

Total Organic Carbon (Automated Combustion Method)

Introduction

Organic Carbon is formed in soil by the decomposition of plant and animal residue, microorganisms and soil biota. Organic carbon has many functions that are vital to soil health such as improving the structural stability of clay soils, supporting the microorganisms that make nutrients available to plants and increasing water holding capacity.

The Organic Carbon content of a soil governs the adsorption of organic compounds to the soil, and is directly related to the mobility and retardation of organic contaminants in groundwaters moving through a soil. Organic Carbon content (TOC or Foc) can be used to predict the partitioning and bioavailability of organic contaminants when they interact with a soil or sediment.

Foc is the Fraction of Organic Carbon in a soil, which is simply its Total Organic Carbon content expressed as a decimal fraction (e.g. 1.0% TOC = 0.010 Foc).

Method Summary

A small sub-sample of a dried, ground soil sample is mixed with catalyst or accelerators and heated to high temperature (generally ≥ 1000°C) within a resistance or induction furnace in a stream of oxygen to convert all forms of Carbon into CO_2. Evolved CO_2 is most commonly detected and quantified using infrared or thermal conductivity detection.

Total Organic Carbon may be determined from the difference of Total Carbon minus Total Inorganic Carbon, or else Inorganic Carbon may be physically removed from the sample by acid treatment prior to direct measurement for TOC.

For soils, TOC / Foc is measured on the < 2 mm fraction of dried, disaggregated, and sieved soil or sediment materials.

This method is performance-based. Laboratories may adopt alternative options to improve performance or efficiency provided that all stated performance requirements and prescribed (mandatory) elements are met.

MDL and EMS Codes

Analyte	MDL (units)	EMS Code
Total Organic Carbon (TOC)	0.050% wt/wt	N/A
Fraction Organic Carbon (Foc)	0.00050	N/A

Matrix

Soil, Sediment.

Interferences and Precautions

High levels of inorganic carbon (e.g. when fragments of mollusc shells are present in a sample) can cause problems when using the TC minus IC subtraction method, because when the majority of the carbon in a sample is inorganic, the uncertainty associated with the difference of TC minus IC becomes too great to give a useful measure for TOC. In such cases, direct measurement of TOC should be used after treatment with sulfurous acid.

High levels of inorganic carbon in a sample can be detected by adding a few drops of 3M HCl to 4-6 grams of wetted sample. Effervescence indicates the presence of inorganic carbon.

Sample Handling and Preservation

Collect samples in a clean polyethylene or glass container. Glass soil jars or polyethylene bags are both appropriate container types.

Stability

Holding Time: Analyze within 28 days of collection (Reference: EPA SW-846 Chapter 3, Feb 2007).

Storage: Store at ≤6°C (≤10°C during shipment to the laboratory). Hold time can be extended indefinitely by drying the sample at <60ºC to less than ~3% moisture content.

Procedure

Soils should be dried, disaggregated, and sieved to produce a < 2 mm fraction, prior to grinding. Exclude foreign materials (such as twigs or rocks) with dimensions exceeding 2 mm that cannot be disaggregated to pass through the 2 mm sieve. Soils with high peat content should be manually ground to pass through the 2 mm sieve, or directly transferred to the grinding step.

After drying and disaggregation, all soils must be ground or pulverized to a particle size appropriate for the size of sub-sample to be used, to ensure complete combustion and to ensure representative sampling. All of the < 2 mm fraction (or a representative portion of this fraction) must be finely ground and analyzed. Only the > 2 mm fraction is to be discarded. For small sub-samples, a manual or automated mortar and pestle is recommended.

Sample Amount Combusted:	**Grinding or Sieving Requirement:**
< 0.01 grams	minimum 100 mesh or 0.15 mm sieve
≥ 0.01 grams – 0.4 grams	minimum 60 mesh or 0.25 mm sieve
> 0.4 grams	minimum 30 mesh or 0.50 mm sieve

Inorganic carbon, typically in the forms of calcite or dolomite, must be removed prior to analysis or accounted for. Prior to instrumental analysis, inorganic carbon may be removed by treatment with 6% Sulfurous Acid (H_2SO_3) as described by the Canadian Society of Soil Science and Soil Science Society of America methods manuals.

Inorganic carbon content can also be determined separately, then subtracted from the total carbon result using the acetic acid, gravimetric or pressure calcimeter methods described by the Canadian Society of Soil Science and Soil Science Society of America methods manuals (see References).

Detailed instrumental procedures are not provided in this method, since they are specific to each instrument. Refer to the instrument operating manual provided by the manufacturer.

Performance Requirements

Any analytical method options selected for this analysis must meet or exceed the performance requirements specified below.

Accuracy and Precision requirements apply to measures of long term method performance (averages and standard deviations). Achievement of these requirements is to be demonstrated during initial and ongoing method re-validation studies. They do not constitute acceptance criteria or Data Quality Objectives for individual Quality Control samples. For Initial Validations, averages of at least 8 replicates of one or more soil CRMs must be assessed (preferably taken from multiple analytical batches). Ongoing Re-validations (performance reviews) should assess QC data encompassing longer timeframes (e.g. 6 months to 1 year). A minimum frequency of 2 years is recommended for Ongoing Re-validations.

Accuracy Requirement: Laboratories must demonstrate method accuracy (measured as average recovery) of 90-110% or better for clean matrix spikes or certified reference materials at concentrations above ten times the MDL.

Precision Requirement: Laboratories must demonstrate method precision equal to or better than 10% relative standard deviation for soils or sediments at concentrations above ten times the MDL.

Sensitivity Requirement: A reported detection limit of 0.05% or lower must be met for this method in order to meet BC MOE requirements for Protocol 13. Qualify test results if this DL cannot be met due to analytical difficulties.

Quality Control

Summary of QC Requirements		
QC Component	**Minimum Frequency**	**Minimum Data Quality Objectives***
Method Blank	One per batch of 20	Less than reported DL
Reference Material or Laboratory Control Sample	One per batch of 20	80 – 120%
Lab Duplicates	One per batch of 20	20% RPD
* Minimum DQOs apply at levels above 10x MDL. Report qualified data if DQOs are not met.		

Method Blank: Use a carbon free <60 mesh sand matrix, e.g. silica sand muffled at > 400ºC overnight, then cooled.

Reference Material: Soil Certified Reference Materials are recommended (e.g. NIST 8704 Buffalo River Sediment, NRC Stream Sediment STSD-4). In-house Reference Materials may also be used if the target value can be demonstrated to be scientifically defensible by calibration against a soil CRM.

Laboratory Control Sample: A recommended LCS option is <60 mesh silica sand muffled at > 400ºC overnight, cooled, then spiked with known quantity of ACS grade sodium carbonate.

Prescribed Elements

The following components of this method are mandatory:

1. The < 2mm fraction of all samples must be ground to an appropriate particle size to obtain a representative, finely ground sample for analysis. Particle size requirements are dependent on size of sub-sample combusted, as defined in the procedure. (Reference: WREP 125, LECO application note 203-821-437).

2. Inorganic carbon may only be removed from the sample prior to analysis using the H_2SO_3 method. Use of other acids may result in oxidation of organic carbon (Reference: Carter).

3. For purposes of BC MOE Protocol 13, a maximum reported detection limit of 0.05% TOC (Foc 0.0005) is normally required. Data should be qualified if technical difficulties prevent this detection limit from being achieved.

4. QC requirements must be met as stated.

5. Loss on Ignition testing is not equivalent to TOC or Foc, and may not be used for purposes of BC MOE Protocol 13.

References

1. Methods of Soil Analysis: Part 3, Chemical methods, 3rd ed., ASA and SSSA, Madison, WI. Book series no. 5, pages 973-974.

2. Carter, Martin. Soil Sampling and Methods of Analysis, Ch 21, Total and Inorganic Carbon, Canadian Society of Soil Scientists (2008), pages 225-237.

3. Plant, Soil and Water Reference Methods for the Western Region, R.G. Gavlak, D.A. Hornacek and R.O. Miller, Total Organic Carbon, Combustion, Western Regional Extension Publication WREP 125, University of Alaska, Fairbanks (1994).

4. Carbon/Nitrogen in Soil and Plant Tissue, Leco Corporation application note 203-821-437, 2012.

Revision History

Aug 15, 2014 New method for BC Lab Manual in support of Protocol 13 and to improve interlaboratory consistency. Effective date of this method is Nov 1, 2014.