



Environmental Analysis Program

Pomona College
Biogeochemistry Laboratory

SOP-L407

Total Carbon – Shimadzu TOC-L with SSM-5000A

Standard Operating Procedure

SOP Number	SOP-L407
Revision	1.1
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Date	2026-06-26
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1. Purpose

This SOP describes the operation of the Shimadzu TOC-L total organic carbon analyzer and the SSM-5000A Solid Sample Module for quantifying dissolved organic carbon (DOC) in soil extracts and total carbon (TC) or total organic carbon (TOC) in solid soil samples (Shimadzu Corporation, 2016, 2019).

2. Scope

- **Applies to:** K_2SO_4 extracts from the chloroform fumigation-extraction method ([SOP-L409](#)); water samples; and dried, ground solid soil samples.
- **Does not apply to:** Samples with extremely high salt concentrations that exceed the instrument's tolerance (consult the instrument manual); sea water or marine sediments with high salt content.
- **Location:** EA Program analytical laboratory, SGM 133.

3. Responsibilities

Role	Responsibility
Lab PI	Approve protocol, authorize instrument access
Instrument Manager	Maintain instrument, replace catalyst and consumables
Trained Personnel	Operate instrument, prepare standards and samples
New Students	Complete training before independent use

4. Definitions

- **Calibration verification standard (CVS):** A known-concentration standard analyzed at regular intervals during a run to confirm that instrument response has not drifted since initial calibration; acceptance criterion is within 10% of the certified value (Shimadzu Corporation, 2019).
- **Dissolved organic carbon (DOC):** Organic carbon in a liquid sample (water or soil extract) that passes through a 0.45 μm filter; quantified by the NPOC method on the Shimadzu TOC-L.
- **Inorganic carbon (IC):** Carbon present as carbonate (CO_3^{2-}), bicarbonate (HCO_3^-), and dissolved CO_2 ; removed from liquid samples by acidification and sparging; measured directly in soils at higher temperatures on the SSM-5000A.
- **NDIR detector:** Non-dispersive infrared detector; measures CO_2 evolved from sample combustion by absorption at 4.26 μm ; the primary detection element of the TOC-L and SSM-5000A (Shimadzu Corporation, 2019).
- **NPOC (non-purgeable organic carbon):** The organic carbon fraction remaining after acidification and sparging to remove IC and volatile organic carbon; the operationally defined DOC fraction measured by the TOC-L for liquid samples.

- **SSM-5000A:** The Shimadzu Solid Sample Module that accepts dried, ground solid samples loaded in ceramic boats and combusts them at up to 900 °C to determine total carbon directly (Shimadzu Corporation, 2016).
- **TC (total carbon):** Sum of all carbon forms (organic and inorganic) in a sample; measured by direct high-temperature combustion of solid samples or by TC mode for liquid samples (Nelson & Sommers, 1996).
- **TOC (total organic carbon):** TC minus IC; in liquid samples, determined as NPOC; in solid soil samples, determined as TC minus IC or after acid treatment to remove carbonates before combustion (Nelson & Sommers, 1996).

5. Safety and Hazards

Hazards

- **Chemical:** Hydrochloric acid (HCl) is used to acidify liquid samples for IC removal; handle in a fume hood with appropriate PPE.
- **Thermal:** The combustion furnace operates at 680 °C (liquid mode) or up to 900 °C (SSM mode); do not contact the furnace chamber.
- **Pressurized gas:** Carrier gas (high-purity air or O₂) is supplied under pressure; inspect fittings for leaks.

5.1. Required PPE

- ☐ Safety glasses
- ☐ Laboratory coat
- ☐ Nitrile gloves

5.2. Emergency Procedures

Refer to [SOP-L002 Laboratory Safety](#) and the Shimadzu TOC-L instrument manual for instrument-specific emergency shutdowns.

6. Equipment and Materials

6.1. Equipment

Item	Notes
Shimadzu TOC-L	With ASI-L autosampler; SGM 133
SSM-5000A	Solid Sample Module; SGM 133
Analytical balance	0.01 mg resolution for solid sample weighing
0.45 µm syringe filters	For liquid sample pre-filtration
Autosampler vials	Acid-washed glass or certified TOC-clean

6.2. Reagents and Consumables

Item	Notes
Potassium hydrogen phthalate (KHP)	Primary standard for C calibration
Sodium bicarbonate / carbonate	IC calibration standards
2 M HCl	For carbonate removal from solid samples
High-purity air or O ₂	Carrier gas for SSM-5000A combustion
Pre-combusted ceramic boats	900 °C prior to use; one per sample
0.5 M K ₂ SO ₄	Method blank for extract analyses

7. Procedure

7.1. Startup

1. Verify carrier gas supply pressure (typically 200 kPa or per instrument manual (Shimadzu Corporation, 2019)).
2. Turn on the TOC-L main unit and allow the furnace to reach operating temperature (680 °C for liquid mode).
3. Launch LabSolutions TOC software on the connected computer.
4. Run a system blank check using deionized water to verify baseline stability; the blank value should be below 0.1 mg/L C.

7.2. Calibration

1. Prepare a multi-point calibration curve using KHP dissolved in deionized water.
2. Typical calibration range: 0, 1, 5, 10, 25, 50, 100 mg/L C (adjust to expected sample range).
3. For IC calibration (if measuring TC - IC = TOC), prepare sodium bicarbonate/carbonate standards.
4. Run calibration standards and verify that the correlation coefficient (r^2) is at least 0.999 (Shimadzu Corporation, 2019).
5. Save the calibration curve in the software.

7.3. Liquid Sample Analysis (DOC in Soil Extracts)

1. Filter all liquid samples through 0.45 µm syringe filters or pre-combusted glass fiber filters into clean autosampler vials.
2. For K₂SO₄ extracts, include 0.5 M K₂SO₄ blanks in the run.
3. Select the appropriate measurement mode in the software:
 - **NPOC mode** (recommended for most soil extracts): The instrument acidifies the sample with HCl, sparges with carrier gas to remove IC as CO₂, then combusts the remaining sample at 680 °C. The evolved CO₂ is measured by the NDIR detector.
 - **TC-IC mode** (if both total and inorganic carbon are needed): TC is measured by direct combustion; IC is measured by acidification only. TOC = TC - IC.

4. Load vials into the autosampler and start the run.
5. The instrument performs replicate injections (typically 3) and reports the mean and coefficient of variation.

High-Salt Extracts

If K₂SO₄ extracts have high salt content, use the instrument's automatic dilution function to extend catalyst and combustion tube life. Dilution factors are applied automatically to the reported concentrations (Shimadzu Corporation, 2019).

7.4. Solid Sample Analysis (SSM-5000A)

1. Switch the instrument to SSM mode in the LabSolutions software (Shimadzu Corporation, 2016).
2. Pre-combust ceramic boats at 900 °C to remove any residual carbon.
3. Weigh 10–50 mg of dried, finely ground soil into a clean ceramic boat using the analytical balance (record mass to 0.01 mg).
4. For TOC measurement (inorganic carbon removal): pre-treat the sample by adding a few drops of 2 M HCl to the boat, allow to react until effervescence stops, and dry at 105 °C before analysis (Nelson & Sommers, 1996).
5. For TC measurement (no acid pre-treatment): proceed directly.
6. Place the ceramic boat into the SSM sample port.
7. The SSM combusts the sample in an oxygen-rich environment at high temperature; the evolved CO₂ is carried to the NDIR detector.
8. The software reports total carbon content in mg C or as a percentage of sample mass.

7.5. Instrument Shutdown

1. After the final sample, run 3–5 deionized water blanks to flush the system.
2. Review all data in the software for outliers or failed injections.
3. Export data and save the run file.
4. If the instrument will not be used for several days, turn off the furnace and carrier gas per the instrument manual (Shimadzu Corporation, 2019).

8. Quality Control

- Run a calibration verification standard (mid-range) every 10–15 samples; acceptance criterion is within 10% of the known value.
- Include instrument blanks (deionized water) at the beginning, middle, and end of each run.
- Include method blanks (0.5 M K₂SO₄ for extract analyses) in each batch.
- Run sample duplicates at a rate of at least 1 per 10 samples.
- Monitor the coefficient of variation among replicate injections; re-run if CV exceeds 2% for liquid samples.
- Log catalyst replacement, combustion tube changes, and calibration dates in the instrument logbook (Shimadzu Corporation, 2019).

9. Maintenance

Task	Frequency
System blank check	Before each analytical run
Calibration verification	Each run (mid-range check standard)
Combustion tube inspection	Monthly or after approximately 500 injections
Catalyst replacement	As needed (when blank values increase or sensitivity decreases)
Drain trap and humidifier check	Weekly
Carrier gas cylinder replacement	As needed (monitor pressure gauge)

10. Waste Disposal

Waste Type	Disposal Method
Acidified liquid waste	Neutralize and dispose per EH&S guidelines
Spent catalyst	Collect for EH&S hazardous waste pickup
Used ceramic boats	Clean and reuse, or dispose in standard trash
K ₂ SO ₄ extract waste	Aqueous waste; drain if approved, or collect

11. References

- Shimadzu Corporation (2019)
- Shimadzu Corporation (2016)
- Nelson & Sommers (1996)
- [SOP-L002 Laboratory Safety](#)
- [SOP-L204 Shimadzu TOC-L \(Instrument\)](#)
- [SOP-L409 Microbial Biomass Carbon](#)

Nelson, D. W., & Sommers, L. E. (1996). Total carbon, organic carbon, and organic matter. In D. L. Sparks (Ed.), *Methods of soil analysis, part 3: Chemical methods* (pp. 961–1010). Soil Science Society of America. <https://doi.org/10.2136/sssabookser5.3.c34>

Shimadzu Corporation. (2016). *SSM-5000A solid sample combustion unit: User manual*.

Shimadzu Corporation. (2019). *TOC-L series total organic carbon analyzer: User manual*.

12. Related SOPs

SOP	Title	Relationship
L002	Laboratory Safety	General safety framework
L204	Shimadzu TOC-L Instrument	Instrument startup/shutdown SOP

SOP	Title	Relationship
L406	Loss on Ignition	Complementary SOM method
L409	Microbial Biomass C	Source of K ₂ SO ₄ extracts
L418	Water-Extractable DOC	Water extraction extracts

13. Training Record

Before operating this instrument independently, personnel must:

1. Read this SOP and the relevant sections of the Shimadzu TOC-L and SSM-5000A user manuals (Shimadzu Corporation, 2016, 2019)
2. Complete [SOP-L003 Hazardous Materials Handling](#) training
3. Observe a trained operator perform startup, calibration, sample analysis, and shutdown
4. Demonstrate competence with calibration standard preparation
5. Perform a supervised analytical run with acceptable QC results
6. Be signed off by the instrument manager or PI