



Environmental Analysis Program

Pomona College
Biogeochemistry Laboratory

SOP-L204

Shimadzu TOC-L Analyzer with TNM-L and SSM-5000A

Standard Operating Procedure

SOP Number	SOP-L204
Revision	2.1
Author(s)	Marc Los Huertos
Date	2026-06-26
Organization	EA Program, Pomona College

FOR INTERNAL USE ONLY

This document is the property of the Environmental Analysis Program at Pomona College.
Unauthorized distribution is not permitted.

Table of contents

1 Purpose	2
2 Scope	3
3 Responsibilities	3
4 Definitions	3
5 Safety and Hazards	4
5.1 Required PPE	4
5.2 Emergency Procedures	4
6 Instrument Description	4
6.1 System Overview	4
6.2 Measurement Modes (Liquid Samples)	5
6.3 Detection Principles	5
7 Required Materials	6
7.1 Instrument Components	6
7.2 Consumables	6
7.3 Calibration Standards	6
8 Procedure	7
8.1 Instrument Startup (Cold Start)	7
8.2 Instrument Startup (From Standby/Sleep)	7
8.3 Standby and Sleep Modes	8
8.3.1 Entering Active Standby	8
8.3.2 Entering Reduced Standby	8
8.3.3 Entering Sleep Mode	8
8.3.4 Full Shutdown	9
8.4 Software Navigation	9
8.5 Calibration	9
8.5.1 Carbon Calibration	9
8.5.2 Nitrogen Calibration (TNM-L)	10
8.6 Setting Up an Analytical Run (Liquid Samples)	10

8.7	Setting Up a Solid Sample Run (SSM-5000A)	10
8.8	Monitoring a Run in Progress	11
8.9	Data Export	11
9	Quality Control	11
10	Maintenance	12
10.1	Routine Maintenance Schedule	12
10.2	Catalyst Replacement Procedure	12
10.3	Combustion Tube Inspection and Replacement	13
10.4	Syringe and Needle Maintenance	13
11	Troubleshooting	13
12	Waste Disposal	14
13	References	14
14	Related SOPs	15
15	Training Record	15

1. Purpose

This SOP describes the general operation, calibration, maintenance, and shutdown of the Shimadzu TOC-L total organic carbon analyzer and its companion modules: the TNM-L Total Nitrogen Module and the SSM-5000A Solid Sample Module. The TOC-L uses a 680 C combustion catalytic oxidation method, which is one of two principal approaches to TOC analysis (the other being wet chemical oxidation with persulfate); the high-temperature combustion method is preferred for environmental samples because it more completely oxidizes refractory organic matter such as humic substances (Sharp, 1993; Sugimura & Suzuki, 1988).

All instrument control is performed through the LabSolutions TOC software on the dedicated computer.

This SOP covers the instrument itself. For application-specific sample preparation, measurement modes, and calculations, refer to the relevant analytical SOPs:

- [SOP-L311 Dissolved Organic Carbon in Water](#) (NPOC)
- [SOP-L407 Total Carbon \(Soils\)](#) (SSM)
- [SOP-L409 Microbial Biomass Carbon](#) (CFE extracts)
- [SOP-L418 Water-Extractable DOC](#) (soil extracts)

2. Scope

- **Applies to:** All use of the Shimadzu TOC-L analyzer, TNM-L module, SSM-5000A module, and ASI-L autosampler in the EA Program laboratory, operated through LabSolutions TOC software
- **Does not apply to:** Manual or standalone operation without computer control; instruments at other institutions; application- specific sample preparation or data interpretation (see analytical method SOPs)
- **Location:** EA Program analytical laboratory, SGM 133

3. Responsibilities

Role	Responsibility
Lab PI	Authorize instrument access, approve maintenance
Instrument Manager	Perform preventive maintenance, replace consumables, manage software updates
Trained Personnel	Operate the instrument per this SOP, log all runs
New Students	Complete training before independent use

4. Definitions

- **Catalytic combustion:** Oxidation of organic carbon in the sample at 680 °C over a platinum catalyst in the presence of oxygen; the standard method used by the Shimadzu TOC-L for liquid samples.
- **Dissolved organic carbon (DOC):** Organic carbon in a water or soil extract sample that passes through a 0.45 µm filter; the NPOC method is the standard approach for DOC measurement.
- **IC (inorganic carbon):** Carbon present as carbonate, bicarbonate, and dissolved CO₂; removed by acidification and sparging before NPOC measurement.
- **NDIR detector (non-dispersive infrared detector):** A detector that measures CO₂ concentration by its absorption of infrared light at ~4.3 µm; the detection element in the Shimadzu TOC-L.
- **NPOC (non-purgeable organic carbon):** The organic carbon fraction remaining in a sample after acidification and sparging to remove inorganic carbon and purgeable volatile organic carbon; the operationally defined DOC measured by the Shimadzu TOC-L.
- **SSM-5000A:** The Shimadzu Solid Sample Module that combusts dried, ground solid materials (soils, sediments, plant tissue) directly in ceramic boats at up to 900 °C for total carbon analysis.
- **TC (total carbon):** The sum of all carbon forms (organic and inorganic) in a sample; measured by direct combustion without acidification.
- **TOC (total organic carbon):** TC minus IC; in practice, usually determined as NPOC for environmental samples.

- **TON (total organic nitrogen):** Total nitrogen minus inorganic nitrogen; measured simultaneously by the TNM-L module using chemiluminescence detection of NO produced during combustion.

5. Safety and Hazards

Hazards

- **Thermal:** The combustion furnace operates at 680 C in liquid analysis mode and up to 900 C in SSM mode. Ceramic boats, combustion tubes, and furnace surfaces can cause severe burns.
- **Chemical:** Hydrochloric acid (2 M HCl) is used for acidification and inorganic carbon sparging. Phosphoric acid (25%) is used as the IC reagent. Avoid skin and eye contact with both.
- **Compressed gas:** The instrument requires high-purity carrier gas (air or oxygen) supplied from a pressurized cylinder at approximately 200 kPa. Follow all compressed gas safety procedures (National Research Council, 2011).
- **Electrical:** The instrument operates on high-voltage mains power. Do not open instrument panels or attempt internal repairs.
- **Ozone:** The TNM-L chemiluminescence detector uses an ozone generator. The instrument vents ozone through an internal decomposer, but ensure that the exhaust line is connected and unobstructed. If an ozone odor is detected, shut down the TNM-L and ventilate the area.

5.1. Required PPE

- ☐ Safety glasses
- ☐ Lab coat
- ☐ Nitrile gloves (when handling acid, standards, or samples)
- ☐ Heat-resistant gloves (when handling ceramic boats from the SSM)

5.2. Emergency Procedures

For acid splashes, flush the affected area with water for at least 15 minutes. For burns from hot surfaces, apply cool running water and seek first aid. If ozone is detected, shut down the TNM-L, open windows, and leave the room until the odor dissipates. Refer to [SOP-L002 Laboratory Safety](#) and [SOP-L003 Hazardous Materials Handling](#).

6. Instrument Description

6.1. System Overview

The Shimadzu TOC-L is a combustion catalytic oxidation analyzer that quantifies carbon (and, with the TNM-L, nitrogen) in liquid samples (Shimadzu Corporation, 2019b). The system in the

EA Program laboratory consists of four main components:

- **TOC-L analyzer:** Combusts liquid samples at 680 C over a platinum catalyst. The resulting CO₂ is measured by a non-dispersive infrared (NDIR) detector. The analyzer supports multiple measurement modes (TC, IC, TOC, NPOC, POC).
- **TNM-L Total Nitrogen Module:** Measures total nitrogen (TN) simultaneously with carbon analysis. Nitrogen compounds in the combustion gas are converted to nitric oxide (NO), which is detected by a chemiluminescence detector after reaction with ozone (Shimadzu Corporation, 2019a). The TNM-L operates in parallel with the NDIR carbon detector, so carbon and nitrogen data are acquired from the same injection.
- **SSM-5000A Solid Sample Module:** Accepts solid samples in ceramic boats for direct combustion at up to 900 C. Connects to the TOC-L for CO₂ detection. The TNM-L is not used during SSM operation.
- **ASI-L Autosampler:** Holds up to 93 sample vials (8 mL or 40 mL) for unattended sequential analysis of liquid samples. Includes an automated rinse function between injections.

6.2. Measurement Modes (Liquid Samples)

Mode	Full Name	What It Measures
TC	Total Carbon	All carbon (organic + inorganic) by direct combustion
IC	Inorganic Carbon	Carbonate/bicarbonate carbon by acidification and sparging only
TOC	Total Organic Carbon (by difference)	TOC = TC - IC; requires two sequential measurements
NPOC	Non-Purgeable Organic Carbon	Sample is acidified and sparged to remove IC, then combusted; the most common mode for routine analysis (APHA et al., 2012, Section 5310B)
POC	Purgeable Organic Carbon	Volatile organic carbon recovered during the sparging step; POC = TC - (NPOC + IC)
TN	Total Nitrogen (TNM-L)	All nitrogen species measured simultaneously with any carbon mode

6.3. Detection Principles

The TOC-L and TNM-L rely on two detectors operating in series on the same combustion gas stream:

- **NDIR detector (carbon):** Infrared absorption by CO₂ is proportional to the mass of carbon injected. The detector is selective for CO₂ and insensitive to other combustion gases.
- **Chemiluminescence detector (nitrogen, TNM-L only):** NO in the combustion gas reacts with ozone (O₃) to produce excited NO₂^{*}, which emits light as it returns to the ground state. The emitted light intensity is proportional to the mass of nitrogen injected (Walsh, 1989).

7. Required Materials

7.1. Instrument Components

Component	Notes
Shimadzu TOC-L analyzer	With combustion furnace and NDIR detector
TNM-L Total Nitrogen Module	Chemiluminescence detector for TN
SSM-5000A Solid Sample Module	For direct combustion of solid samples
ASI-L Autosampler	93-position; 8 mL or 40 mL vials
Dedicated computer	Running LabSolutions TOC software
Carrier gas (high-purity air)	Regulated to 200 kPa (or per manual)
Exhaust tubing	Connected to fume hood or building exhaust

7.2. Consumables

Item	Notes
TC catalyst (Pt on alumina beads)	Replace when blanks rise or sensitivity drops; typical life 3,000–5,000 injections
IC reagent (25% phosphoric acid)	For IC/NPOC acidification; check level weekly
Combustion tubes (quartz)	Inspect monthly; replace if cracked or discolored
Ceramic sample boats	For SSM; pre-combust at 900 C before first use
Halogen scrubber	Replace every 6–12 months or per manufacturer schedule
Dehumidifier / moisture trap	Drain weekly; more often in humid weather
Autosampler vials and septa	Acid-washed for trace-level work; use pre-combusted glass vials for NPOC
Syringe needle (instrument syringe)	Inspect for bends or clogs; replace as needed

7.3. Calibration Standards

Standard	Use	Preparation
Potassium hydrogen phthalate (KHP)	Primary TC/NPOC calibration	2.125 g/L = 1,000 mg C/L stock (APHA et al., 2012)
Sodium bicarbonate / sodium carbonate	Primary IC calibration	See Shimadzu manual for concentrations
Potassium nitrate (KNO ₃)	Primary TN calibration (TNM-L)	7.218 g/L = 1,000 mg N/L stock
Deionized water (18.2 MOhm)	Blanks, dilutions, and standard preparation	From Milli-Q; verify TOC less than 0.05 mg/L

8. Procedure

! Computer Control Only

All operations described below are performed through LabSolutions TOC. Do not adjust furnace temperature, gas flow, or other parameters using the instrument front panel unless directed by the instrument manager during troubleshooting.

8.1. Instrument Startup (Cold Start)

A cold start applies when the instrument has been fully powered down (furnace off, gas closed). Allow approximately 60 minutes from power-on to running samples.

1. Open the carrier gas cylinder valve. Verify the regulator output pressure is set to 200 kPa at the instrument inlet. Check for leaks at all fittings (listen for hissing; apply leak-detection solution if uncertain).
2. Verify that the exhaust line from the TNM-L ozone decomposer is connected and routed to the fume hood or building exhaust
3. Check consumable levels:
 - IC reagent bottle (25% phosphoric acid): refill if below the minimum line
 - Dehumidifier / moisture trap: drain if water is visible
 - Rinse water reservoir on the ASI-L: fill with fresh deionized water
4. Power on the TOC-L main unit using the switch on the right side panel
5. If the TNM-L is needed, power on the TNM-L using the switch on its rear panel
6. Power on the ASI-L autosampler
7. Turn on the dedicated computer and launch LabSolutions TOC
8. In LabSolutions TOC, verify that communication with all modules is established. The status bar should show “Warming Up”
9. **Wait for the furnace** to reach 680 C (approximately 20–30 minutes). The software status changes from “Warming Up” to “Ready”
10. **Wait for the TNM-L** ozone generator and chemiluminescence detector to stabilize (approximately 30–45 minutes after power-on; the TNM-L status indicator changes to “Ready”)
11. Run 3–5 injections of deionized water as system conditioning blanks to flush the injection port, syringe, and combustion tube. Discard these results.
12. Run 3 additional deionized water blanks and verify baseline stability: TC blank values should be below 0.1 mg/L C; TN blank values should be below 0.1 mg/L N

8.2. Instrument Startup (From Standby/Sleep)

When the instrument is in standby mode (furnace at temperature, gas flowing, software idle), startup is faster:

1. Launch LabSolutions TOC if not already open
2. Verify that the status bar shows “Ready” for the TOC-L and (if applicable) the TNM-L

3. Check the IC reagent level and drain the moisture trap
4. Fill the ASI-L rinse reservoir with fresh deionized water
5. Run 3 deionized water conditioning blanks. If blanks are below 0.1 mg/L C and 0.1 mg/L N, proceed to calibration.

8.3. Standby and Sleep Modes

Choose the appropriate idle mode based on how soon the instrument will be used again:

Mode	Furnace	Carrier Gas	TNM-L	When to Use
Active standby	680 C (on)	Flowing	On	Next run within 24 hours
Reduced standby	680 C (on)	Flowing	Off	Next run within 2–3 days; no TN needed
Sleep	Off (cooling)	Closed	Off	No runs planned for 3+ days
Full shutdown	Off (cold)	Closed	Off	Extended break, maintenance, or relocation

8.3.1. Entering Active Standby

1. After the final sample, run 3–5 deionized water blanks to flush the system
2. Leave the furnace at 680 C and the carrier gas flowing
3. Leave the TNM-L powered on
4. Empty the autosampler rack and cover with a dust cover
5. The instrument manager may set the software to a low carrier gas flow rate overnight to conserve gas (consult the manual for the minimum flow that maintains a positive purge of the combustion tube)

8.3.2. Entering Reduced Standby

1. Flush the system with deionized water blanks as above
2. Power off the TNM-L (rear panel switch). This extends the ozone decomposer lifetime.
3. Leave the TOC-L furnace at 680 C and gas flowing
4. Cover the autosampler

8.3.3. Entering Sleep Mode

1. Flush the system with deionized water blanks
2. Power off the TNM-L
3. In LabSolutions TOC, initiate a furnace cooldown (or simply power off the TOC-L)
4. Once the furnace temperature drops below 200 C (approximately 90 minutes), close the carrier gas cylinder valve
5. Power off the ASI-L
6. Close LabSolutions TOC and shut down the computer

8.3.4. Full Shutdown

Follow the Sleep Mode steps, then:

1. Allow the furnace to cool to room temperature
2. Disconnect the carrier gas line if the instrument will be moved or serviced
3. Empty the IC reagent bottle if the shutdown will exceed 30 days (phosphoric acid can corrode the reagent lines over long idle periods)
4. Empty the ASI-L rinse reservoir to prevent microbial growth
5. Cover the instrument with a dust cover

8.4. Software Navigation

LabSolutions TOC organizes work into the following areas:

- **Instrument Monitor:** Real-time display of furnace temperature, carrier gas flow, detector signal (NDIR and chemiluminescence), and system status
- **Method Editor:** Define measurement mode (TC, IC, NPOC, TN, etc.), injection volume, number of replicate injections, sparge time (for NPOC), acidification volume, and calibration curve parameters
- **Sample Table:** Enter sample IDs, vial positions, dilution factors, and measurement method for each sample
- **Calibration:** Build, view, and edit calibration curves. The software fits curves by least-squares regression and reports r^2 and residuals.
- **Data Browser:** Review completed runs, view peak integrations, export results to CSV, and reprocess data

8.5. Calibration

Calibrate at the start of each analytical session or whenever the method, measurement mode, or concentration range changes. Carbon and nitrogen calibrations are built independently but can be run simultaneously if both detectors are active.

8.5.1. Carbon Calibration

1. Prepare a multi-point calibration series from KHP dissolved in deionized water. Choose concentrations that bracket the expected sample range (e.g., 0, 1, 5, 10, 25, 50, 100 mg/L C). Prepare fresh daily or from a refrigerated stock per [SOP-L020](#) (APHA et al., 2012).
2. For IC calibration, prepare standards from sodium bicarbonate/sodium carbonate in deionized water
3. In LabSolutions TOC, open the Method Editor and select the measurement mode (e.g., NPOC, TC, or TC-IC)
4. Enter calibration standard concentrations in the Calibration window
5. Load standards into the autosampler in order of increasing concentration
6. Run the calibration sequence. The software will perform replicate injections (typically 3) of each standard and fit a regression

7. Verify that $r^2 \geq 0.999$ and that each standard back-calculates to within 10% of its nominal value (15% at the lowest standard is acceptable)
8. Save the calibration curve

8.5.2. Nitrogen Calibration (TNM-L)

1. Prepare a multi-point calibration series from KNO_3 dissolved in deionized water (e.g., 0, 0.5, 1, 5, 10, 25, 50 mg/L N)
2. In the Method Editor, confirm that TN measurement is enabled alongside the carbon mode
3. Enter TN calibration concentrations in the Calibration window
4. The TN calibration runs simultaneously with the carbon calibration if both are loaded in the same sample table; otherwise, run the TN standards as a separate sequence
5. Verify that $r^2 \geq 0.999$ for the nitrogen curve
6. Save the calibration curve

8.6. Setting Up an Analytical Run (Liquid Samples)

1. In the Method Editor, confirm the measurement mode, injection volume, number of replicate injections, and (for NPOC mode) sparge time and acid volume
2. In the Sample Table, enter the run sequence:
 - a. Calibration verification standard (mid-range) as the first sample
 - b. Instrument blanks (deionized water) at the beginning, every 10–15 samples, and the end of the run
 - c. Method blanks appropriate to the application (e.g., 0.5 M K_2SO_4 for soil extracts; consult the analytical method SOP)
 - d. Sample vials in the order they are loaded in the autosampler
 - e. A final calibration verification standard
3. Set the dilution factor for each sample if applicable
4. Verify that autosampler vial positions in the Sample Table match the physical rack positions
5. Confirm that the rinse reservoir on the autosampler is filled with deionized water
6. Click **Start** to begin the sequence

Replicate Injection Settings

The software default is 3 replicate injections per sample. If the coefficient of variation (CV) among replicates exceeds 2%, the software can be configured to add up to 2 additional injections automatically (set in Method Editor under “Maximum Injections”). The outlier replicate is excluded from the reported mean.

8.7. Setting Up a Solid Sample Run (SSM-5000A)

1. In LabSolutions TOC, switch to SSM mode
2. Pre-combust ceramic boats at 900 C in a muffle furnace ([SOP-L111](#)) to remove residual carbon. Allow boats to cool in a desiccator.

3. Weigh the solid sample into a clean ceramic boat. Record the mass to 0.01 mg.
4. In the Sample Table, enter the sample ID, boat number, and sample mass
5. Place the ceramic boat on the SSM sample port and push it into the furnace when prompted by the software
6. The SSM combusts the sample; evolved CO₂ flows to the TOC-L NDIR detector
7. The software reports carbon content in mg C or as a percentage of sample mass

TNM-L and SSM

The TNM-L chemiluminescence detector is not used during SSM solid sample analysis. Total nitrogen in solid samples requires a separate instrument (e.g., the Flash EA; see [SOP-L208](#)).

8.8. Monitoring a Run in Progress

1. Use the Instrument Monitor to watch real-time peak shapes and detector baselines
2. If a peak appears saturated (flat top), the sample exceeds the calibration range. Pause the run, dilute the sample, and re-queue.
3. If the CV among replicate injections exceeds 2%, investigate (air bubble, particulates, insufficient sample volume) and consider re-running.
4. If the system blank rises above 0.5 mg/L C or 0.5 mg/L N during a run, the combustion tube or catalyst may need replacement. Complete the current run, note affected samples, and consult Section 9.

8.9. Data Export

1. After the run, open the Data Browser and review all results
2. Check that calibration verification standards recovered within 90–110% of their nominal values
3. Export results to CSV: **File > Export**, including sample ID, mode, mean concentration, SD, CV, and number of valid injections
4. Save the LabSolutions TOC run file (.toc) to the instrument computer and back up to the lab network drive
5. Record the run date, analyst, method, and file name in the instrument logbook

9. Quality Control

QC procedures are tailored to each analytical method and are specified in the relevant method SOP. The following instrument-level QC applies to all runs (US Environmental Protection Agency, 2009):

- System blanks (deionized water) at the start of every session must read below 0.1 mg/L C and 0.1 mg/L N
- A calibration verification standard (mid-range) must be run at the beginning and end of every sample sequence, with recovery within 90–110%
- Replicate injection CV must be below 2% for liquid samples

- All QC results are recorded in the instrument logbook and in the exported data file
- For additional QC requirements (method blanks, duplicates, matrix spikes, continuing calibration verification frequency), refer to [SOP-L802 Quality Control](#)

10. Maintenance

10.1. Routine Maintenance Schedule

Task	Frequency	Notes
System blank check	Before each analytical session	
Drain moisture trap / dehumidifier	Weekly (more often in humid conditions)	Prevents water from reaching the NDIR detector
Check IC reagent level	Weekly	Refill with fresh 25% H_3PO_4 when below the minimum line
Inspect injection syringe needle	Weekly	Replace if bent, corroded, or clogged
Check autosampler rinse reservoir	Before each session	Refill with fresh DI water
Inspect combustion tube	Monthly or every 500 injections	Look for discoloration, cracks, or catalyst settling
Replace TC catalyst	Every 3,000–5,000 injections or when blanks rise	Pt on alumina; record replacement date
Replace halogen scrubber	Every 6–12 months	Prevents halide damage to the NDIR detector
Replace TNM-L ozone decomposer	Annually	Degraded decomposer leaks ozone into the room
Replace SSM combustion tube	As needed	Inspect after each batch of solids
Carrier gas cylinder replacement	As needed	Monitor regulator pressure; order when below 500 psi
Software and firmware updates	As released by Shimadzu	Coordinate with instrument manager

10.2. Catalyst Replacement Procedure

1. Allow the furnace to cool to room temperature (power off the TOC-L and wait at least 3 hours, or use the forced-air cooling function if available)
2. Wearing heat-resistant gloves and safety glasses, remove the combustion tube from the furnace per the manufacturer's procedure
3. Dispose of the spent catalyst (Pt on alumina) as hazardous waste (precious metal content; collect for EH&S pickup per [SOP-L006](#))

4. Fill the combustion tube with fresh catalyst to the manufacturer's specified depth (approximately 12 cm)
5. Add a thin layer of quartz wool above and below the catalyst bed
6. Reinstall the combustion tube. Reconnect all tubing and fittings.
7. Restart the furnace and allow it to reach 680 C
8. Run 20–30 deionized water conditioning injections to stabilize the new catalyst. Blanks should decrease progressively to below 0.1 mg/L C.
9. Run a calibration verification standard to confirm sensitivity before resuming sample analysis
10. Record the replacement date, catalyst lot number, and conditioning results in the instrument logbook

10.3. Combustion Tube Inspection and Replacement

1. Remove the combustion tube as described in Section 9.2, step 2
2. Inspect for cracks, discoloration, or accumulation of non-volatile residue on the inner walls
3. If the tube is damaged, replace with a new quartz combustion tube
4. Repack with catalyst and condition as in Section 9.2

10.4. Syringe and Needle Maintenance

1. Remove the syringe from the autosampler or manual injection port
2. Inspect the needle for bends, burrs, or corrosion
3. Flush the syringe barrel with deionized water, then with dilute HCl (0.1 M), then with deionized water again
4. If the syringe plunger binds or the needle is damaged, replace the entire syringe assembly
5. Reinstall and run 3 deionized water blanks to verify proper function

11. Troubleshooting

Symptom	Probable Cause	Corrective Action
Blank values increasing over time	Catalyst degradation; contaminated combustion tube	Replace catalyst; inspect and replace combustion tube
Poor replicate precision (CV > 2%)	Air bubble in syringe; particulates in sample; insufficient volume	Check syringe for air leaks; re-filter sample; verify vial volume
NDIR signal saturated (flat-top peaks)	Sample exceeds calibration range	Dilute sample and re-run
TNM-L "Not Ready" status persists	Ozone generator not warmed up; decomposer exhausted; exhaust blocked	Allow more warm-up; replace decomposer; check exhaust tubing
No communication with instrument	Cable disconnected; software crash; instrument not powered on	Check cables; restart software; verify power

Symptom	Probable Cause	Corrective Action
Autosampler skips vials	Position mismatch; septa not pierced	Verify positions; check that septa are intact
SSM signal noisy or irreproducible	Boat not fully inserted; sample mass too large; residual carbon on boat	Confirm position; reduce mass; pre-combust boats longer
Carrier gas pressure alarm	Cylinder empty or regulator set incorrectly	Replace cylinder; adjust regulator
Moisture trap fills rapidly	High humidity; furnace tube leak	Drain more frequently; inspect tube fittings for leaks
IC reagent not dispensing	Reagent bottle empty; tubing kinked or clogged	Refill bottle; inspect and replace tubing

12. Waste Disposal

Waste Type	Disposal Method
Acidified liquid waste (HCl/H ₃ PO ₄)	Neutralize and dispose per SOP-L006
Spent TC catalyst (Pt on alumina)	Collect for EH&S hazardous waste pickup (precious metal)
Used combustion tubes (quartz)	Standard glass waste (broken-glass box) if not contaminated
Used ceramic boats (SSM)	Clean and reuse, or standard trash
Autosampler rinse water	Drain
Calibration standard waste	Aqueous waste; drain if dilute

13. References

- APHA, AWWA, & WEF. (2012). *Standard methods for the examination of water and wastewater* (22nd ed., p. 1360). American Public Health Association.
- National Research Council. (2011). *Prudent practices in the laboratory: Handling and management of chemical hazards* (Updated). National Academies Press. <https://doi.org/10.17226/12654>
- Sharp, J. H. (1993). The dissolved organic carbon controversy: An update. *Oceanography*, 6(2), 45–50. <https://doi.org/10.5670/oceanog.1993.12>
- Shimadzu Corporation. (2019a). *TNM-L total nitrogen module: User manual*.
- Shimadzu Corporation. (2019b). *TOC-L series total organic carbon analyzer: User manual*.
- Sugimura, Y., & Suzuki, Y. (1988). A high-temperature catalytic oxidation method for the determination of non-volatile dissolved organic carbon in seawater by direct injection of a liquid sample. *Marine Chemistry*, 24(2), 105–131. [https://doi.org/10.1016/0304-4203\(88\)90043-6](https://doi.org/10.1016/0304-4203(88)90043-6)
- US Environmental Protection Agency. (2009). *Method 415.3, rev. 1.1: Determination of total organic carbon and specific UV absorbance at 254 nm in source water and drinking water* (EPA/600/R-09/122). Office of Research; Development, US EPA.

Walsh, T. W. (1989). Determination of dissolved nitrogen in estuarine waters using high-temperature combustion and chemiluminescence detection. *Marine Chemistry*, 26(4), 295–311. [https://doi.org/10.1016/0304-4203\(89\)90036-4](https://doi.org/10.1016/0304-4203(89)90036-4)

14. Related SOPs

SOP	Title	Relationship
L003	Hazardous Materials Handling	Chemical safety for acids
L006	Waste Disposal	Catalyst and chemical waste disposal
L020	Reagent and Standard Preparation	KHP and KNO ₃ standard prep
L111	Muffle Furnace	Pre-combustion of ceramic boats
L311	Dissolved Organic Carbon (Water)	NPOC application SOP
L407	Total Carbon (Soils)	SSM application SOP
L409	Microbial Biomass Carbon (CFE)	DOC extract application SOP
L418	Water-Extractable DOC	Soil extract application SOP
L802	Quality Control	QC requirements for all runs

15. Training Record

Before operating this instrument independently, personnel must:

1. Read this SOP in full and the relevant sections of the Shimadzu TOC-L, TNM-L, and SSM-5000A user manuals
2. Complete [SOP-L002 Laboratory Safety](#) and [SOP-L003 Hazardous Materials Handling](#) training
3. Observe a trained operator perform a complete session: cold start, calibration (carbon and nitrogen), sample analysis, data export, and transition to standby
4. Demonstrate competence preparing calibration standards (KHP and KNO₃) per [SOP-L020](#)
5. Perform a supervised analytical run with acceptable QC results (blanks below 0.1 mg/L, calibration verification recovery 90–110%, replicate CV below 2%)
6. Demonstrate entering and exiting standby and sleep modes
7. Be signed off by the instrument manager or PI