

TOC Advance User Guide

Total Organic Carbon Analyzer Advanced User Guide (from TOC_AUG_20100429)

Manual Information

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Introduction

TOC Flow Paths

The liquid sample is delivered by syringe pump through the teflon tubing to the 4-port valve, which rotates to four fixed positions (Fig 1 and Fig 2):

- SAMPLE: syringe pulls in the sample liquid
- TC PORT: syringe is connected to TC lines (Fig 3)
- IC PORT: syringe is connected to IC lines (Fig 4)

Depending on the 4-port valve position and autosampler needle position over the vial containing zero water or sample, the syringe pumps sample or water to the TC or IC port, performing necessary rinsing or sample injection.

Both IC and TC ports have ability to slide into either position over the port itself for the sample injection or over the waste valve for the flow line rinsing operation.

The choice flow path, TC or IC, can be selected either from TOC-5000A instrument front panel or from a software dialog window.

Figure 1-1. TOC-5000A Flow Schematic.

Figure 1-2. Flow Schematic Key to Symbols.

Figure 1-3. Total Carbon Flow Path.

Figure 1-4. Inorganic Carbon Flow Path.

TOC System Installation

Connections

The TOC should be connected on the back as follows:

- PC through RS232 port by 9-pin cable
- Autosampler by two cables mounted in the TOC

- Gas Generator by one tubing connection

Power cables are provided for TOC, PC, and Gas Generator. The Autosampler is powered by the TOC analyzer.

Figure 1-5. TOC System Connections.

- Connect the Gas Generator to a compressed air source with a pressure regulator in the range of 65–125 psi (450–850 kPa). If the system is connected to an ultra-high purity air tank instead of a Gas Generator, set the maximum tank regulator delivery pressure to 80 psi.
- Connect drain lines from the TOC and IC slidable ports and IC solenoid with a Y connector followed by a T-connector to add the dehumidifier drain valve line to the general drain line. Plug the Humidifier into an outlet after filling the Humidifier with distilled water.
- Fit the Autosampler arm with a needle and align over the rinse hole position inside the Autosampler body.
- Load software onto the PC from two sets of floppy disks entitled "TOC PC Control/Version 1.05."

General Startup Procedure

This general startup procedure is usually performed after the instrument has been powered down.

Start Up Hardware

| Step | Action |
|------|---|
| 1 | Make sure there are no obvious malfunctions in the TOC system (e.g., tubing and cables are connected properly). |
| 2 | Turn on the compressed air feed line (65–80 psi) for the Gas Generator (for a high purity air tank the delivery pressure should not exceed 100 psi and normally is set to 80 psi). |
| 3 | Turn on the Gas Generator power. The start up yellow light should come on. Wait 40–60 min for the Gas Generator to get in ready condition. THC light should be green and there should be no CHECK SYSTEM/ OVERFLOW lights on. <ul style="list-style-type: none"> • If the Gas Generator was down for more than a few weeks, it is a good idea to let the generator run at least 12 hr prior to an analysis. |
| 4 | Fill Autosampler Rinse Bottle with Zero-water (i.e., distilled water). |
| 5 | Fill the Humidifier with distilled water until the water level is between the white lines. Fully plug in the rubber stopper. |
| 6 | Fill IC vessel with a 25% phosphoric acid solution up to 75–100 mL. |
| 7 | Make sure all drain lines have a negative slope alignment. Empty the drain container, if required. |
| 8 | Check position of three-way cock arm (see chart on TOC door inner side). The cock arm should be in the left position. |
| 9 | Turn on TOC main power switch. Make sure black switch on front top right side (behind instrument door) is in PC CONTROL position. |

Start Up Software

| Step | Action |
|------|--|
| | Turn on PC. If computer was logged off, use DAQ for username and password. |
| | Click on the TOC_CONTROL icon on the computer desktop. Type DAQ for username and password again. |

Start Up Gas Generator

| Step | Action |
|------|---|
| 1 | Open the Gas Generator inlet line coming from the compressed air generator. |

| | |
|---|--|
| 2 | Ensure the feeding pressure is in the range of 65–80 psi (450–550 kPa). |
| 3 | Turn on the Gas Generator (power switch is above the power cable). The yellow start up light should turn on. |
| 4 | After 10-15 min the sequence of lights below the THC sign on the front panel will start with a yellow light at the top and gradually will go through the lights. It will take another 30-40 min to get a green light on for THC parameter, which means the catalyst unit is heated to the appropriate temperature. |
| 5 | After the green light is on, it is OK to use the outlet air for TOC analysis. <ul style="list-style-type: none"> • If the Gas Generator has been shut down for a long time, it's better to let the instrument run for another 12 hr, so it will be conditioned before performing any analyses. |

Shutting Down the TOC Analyzer

| Step | Action |
|------|---|
| 1 | Click on the toolbar Measure –Standby button and make sure the oven is in the switched off position. |
| 2 | After 30 min, click on the toolbar <i>Measure –Disconnect</i> line. |
| 3 | Switch off the main power button on the TOC instrument body. |
| 4 | Shut down the air lines. |

Troubleshooting

Hardware Troubleshooting

Unstable Baseline

- Leak in carrier gas
- TC catalyst needs to be replaced
- Dehumidifier not operating correctly
- Environmental: vibration, ambient temperature, power supply
- NDIR defective

Bubbles in Sample/Syringe Tubing

Bubbles in the sample tubing affect the accuracy of an analysis. If bubbles are noted in the sample tubing, they need to be removed before beginning an analysis.

| Step | Action |
|------|---|
| 1 | Select Options > Maintenance > Mechanical Check > TOC. |
| 2 | Move the syringe plunger up and down using the plunger height control on the left while changing the position of the 4-port valve to Sample, TC port, or IC port. |
| 3 | When the sample goes to the TC/IC waste line, do not bring the syringe plunger all the way up. |

Peaks Are Too Broad or Are Tailing

Tailing should be considered a significant issue. Tailing is identified by a **T** in the real-time window in the *Notes* column and means the integration was cut before the peaks curve reached the baseline. Tailing peaks can be caused by

- Contaminated tubing: check a blank analysis for a contamination peak
- Clogged or leaking tubing:
- Leaking and restricted tubing affect the outlet (CO₂ absorber L-line) flow rate. Check both the flowmeter inlet and outlet openings because the TOC and laboratory flow meters may be different. Lower flow at the outlet suggests a clog or leak that must be isolated and fixed.

- Connect both L and S tubes of the CO₂ absorber in one line. In this configuration, the operator should see immediate bubbling in the dehumidifier drain container, meaning the line has no major leak and all gas is escaping through the drain. If no bubbling occurs that means the system has a leak or is clogged. The most common places to check for the flow rate inside the instrument are the connections before the combustion tube and the cooling coil outlet.

No Peaks

- The most common reason for an absent peak is a TOC flow line leak. Check the air supply line/carrier gas flow reading and make sure the humidifier stopper is tightly plugged.

TOC/ASI Normal Analysis Routine Stopped in Middle of Run without Operator Command

This may happen because of a hardware malfunction (e.g., bent needle or 4-port valve sticking).

- If the needle is bent, unscrew it and remove it from the autosampler arm. Usually the needle can be straightened. If not, then replace the needle. When replacing the needle in the autosampler arm, ensure the needle height and alignment above the rinse/sample vials are positioned correctly before tightening down the arm.
- If the 4-port valve is stuck, take it apart and check for breakage or particle contamination, as those can cause rotation failure.

Autosampler Troubleshooting

Regenerating TC Catalyst

| Step | Action |
|------|--|
| 1 | Place a vial containing 2 mM HCl in position S1. |
| 2 | Open the autosampler Maintenance screen and select Regeneration of TC Catalyst . |
| 3 | The sampling needle moves to the S1 vial then to the rinse water receptacle and rinses the injection needle 3 times. |

Replacing Rinse Water Pump Head

| Step | Action |
|------|--|
| 1 | After ~300 hr running time, replace the pump head assembly (PN 042-00405-11). |
| 2 | Take off the small cover attached to the side of the autosampler. |
| 3 | Pull out the pump head (white plastic) from the pump body (black plastic) by picking the claws of both sides with fingers. |
| 4 | Replace the pump head by disconnecting the elastic tubes from the Teflon tubes and connecting new ones. |
| 5 | Remount the pump head and cover. |

Removing the Syringe

| Step | Action |
|------|---|
| 1 | Unscrew the plastic plunger holder on the bottom of the syringe (may use pliers). |
| 2 | Unscrew the syringe body from the 4-port valve stand. |

Shimadzu Technical Support

- 1-800-477-1227, ext 1442
- Leonardo Ponds

Software Troubleshooting

Toolbar options don't respond properly

- Make sure previous windows are closed; software version is old and can operate only with one window open in the TOC Control screen
- Close/reopen TOC Control window
- Restart software/computer

Gas Generator Troubleshooting

- Normally the Gas Generator should be connected to the TOC through blue plastic tubing. In this case, the outlet flow rate of the Gas Generator is controlled by the TOC flow controller (150 mL/min). If the Gas Generator outlet flow line is disconnected and open to the atmosphere, the flow rate will vary, depending on the inlet pressure. When outlet flow is higher than 1250 mL/min, the Gas Generator front panel overflow indication light will turn yellow. In this situation, the technician should correct the flow rate, because overflow conditions will cause the Gas Generator catalyst module to cool and will not produce appropriate quality air.
- The outlet flow reading may drop below 150 mL/min or have no flow at all. This will significantly affect the TOC flow rate. The most common reason for such a malfunction is the final filter or other filters are clogged. Replace the filters to fix this problem.
- If the **CHECK SYSTEM** light located on the bottom of the front panel starts flashing, and especially when it turns to a permanent yellow light, the catalyst unit needs to be exchanged for a new one. Normally happens once every 2–3 yr of constant Gas Generator use.
- Every 4–5 min a pneumatic switch triggers audibly between the dryer and co-remover towers on the back of the Gas Generator. If the interval time between switches is ~10 min, there may be problems with the Gas Generator.