

# **450TOC Portable TOC Analyzer Standard Operating Procedure for**

- **TOC Calibration**
- **Conductivity Calibration**
- **Temperature Calibration**
- **Flow Rate Calibration**

**METTLER TOLEDO**

A graphic element consisting of a series of parallel diagonal lines that form a triangular shape pointing downwards, positioned behind the Mettler Toledo logo text.

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# IMPORTANT SAFETY INFORMATION

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Please read thoroughly before operating the TOC System Suitability Test Kit and the TOC Calibration Kit -

- Follow all warnings, cautions, and instructions indicated on and supplied with this product.
- Install equipment as specified in this instruction manual. Follow appropriate local and national codes.
- Use only factory documented components for repair. Tampering or unauthorized substitution of parts and procedures can affect the performance and cause unsafe operation of your process as well as void factory warranties.
- Protective covers must be in place unless qualified personnel are performing maintenance.
- If this equipment is used in a manner not specified by the manufacturer, the protection provided by it against hazards may be impaired.
- Prior to shipping the sensor back to the factory for repair or re-calibration, water **MUST** be drained from sensor to avoid damage due to freezing.

## WARNINGS:

- Installation of cable connections and servicing of this product require access to shock hazard voltage levels.
- Main power must employ a switch or circuit breaker as the disconnecting device for the equipment.
- Electrical installation must be in accordance with the National Electrical Code and/or any other applicable national or local codes.
- Safety and performance require that this Analyzer be connected and properly grounded through a three-wire power source.

This manual includes safety information with the following designations and formats:

WARNING: POTENTIAL FOR PERSONAL INJURY.

CAUTION: possible Analyzer damage or malfunction.

NOTE: important operating information.

## Definition of Equipment Symbols



On the Analyzer indicates: Warning, risk of electric shock.



On the Analyzer indicates: Caution (refer to accompanying documents).



On the Analyzer indicates: There is alternating current present.

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# 1. Introduction

The Mettler-Toledo Thornton 450TOC Portable TOC Analyzer measures the amount of organic carbon in high purity waters by oxidizing organic carbon to CO<sub>2</sub> with appropriate UV radiation. The resulting change between two temperature-compensated conductivity measurements of the sample flow stream at points before and after oxidation is used to calculate the amount of total organic carbon present.

# 2. Scope

This document provides procedures to calibrate the Thornton 450TOC Portable TOC Analyzer (PN 58 036 041). This document provides strategies for the frequency and content of "As Found" data and the calibration process with respect to lamp changes and performing System Suitability Tests (SST) to meet specific pharmacopeia requirements.

# 3. Calibration Process

The complete calibration of the 450TOC Portable TOC Analyzer consists of calibration of the following:

- Flow rate sensor measurement
- Two temperature sensor measurements
- Two conductivity sensor measurements
- TOC measurement

## 3.1. Sequence of Operations for Calibration, SST and Lamp Changes

When performing a calibration, the sequence of operations depends on other process decisions. For example:

- If a lamp change is intended, does "As Found" data need to be collected?
- Is an SST scheduled during this time?
- If an SST is required, does "As Found SST" data need to be collected?
- Is a TOC calibration required, or is a full calibration for TOC, conductivity, and temperature required?

Determination of answers for these questions, as shown in the flowchart, will determine the sequence of operations. The answers are determined by the users of this Analyzer, but Table 1 lists Thornton's minimum recommended service cycle.

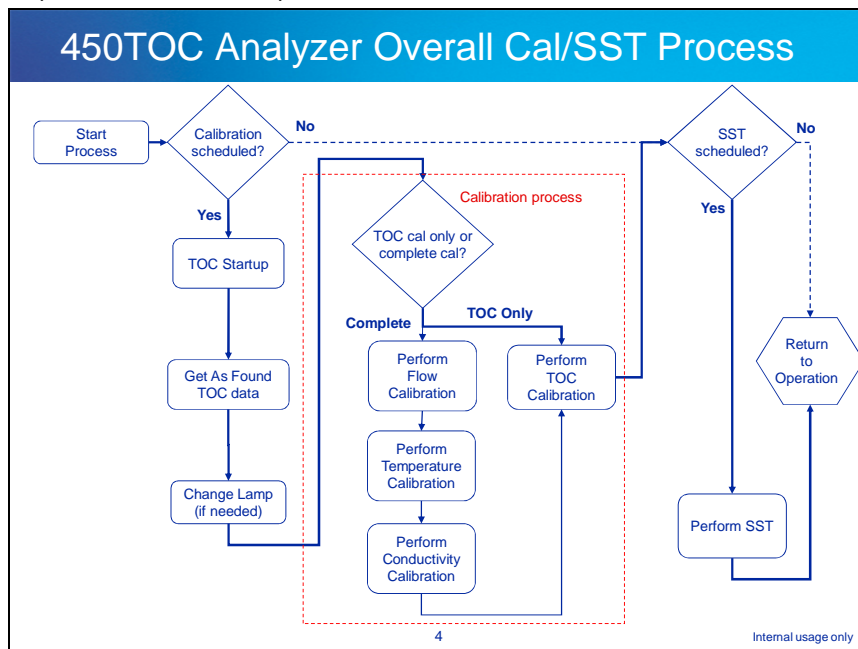


Table 1. Scheduled Calibration, Lamp change, and SST Service

Maintenance	Frequency/Schedule	Recommendations/Comments
Replace lamp	4500 hours of lamp operation or 1 calendar year, whichever is shorter.	<ul style="list-style-type: none"> <li>• TOC Calibration recommended after lamp change.</li> <li>• SST recommended after lamp change (where SST is required*). Refer to SST Standard Operating Procedure.</li> </ul>
TOC Calibration	After UV lamp replacement, not to exceed one calendar year.	<ul style="list-style-type: none"> <li>• Recommended after lamp change, not to exceed 1 calendar year.</li> </ul>
Complete Calibration (includes flow, TOC, conductivity, temperature)	As determined by user.	<ul style="list-style-type: none"> <li>• Recommended not to exceed 1 calendar year.</li> </ul>
System Suitability Test (SST)	As determined by user.	<ul style="list-style-type: none"> <li>• Recommended after UV lamp change.</li> </ul>

\*See USP <643> and EP 2.2.44

### 3.2. Summary of Calibration Process

1. Determine if the TOC measurement or all measurements will be calibrated.
2. Collect all equipment.
3. Install equipment and tubing.
4. If required, record As Found TOC Calibrations Factors and collect As Found TOC data as described in Section 7 *Record TOC As-Found Readings*.
5. If required, change UV lamp.
6. To perform a TOC calibration only, complete the following steps:
  - a. Complete sections A and E of the Calibration Record Sheet.
  - b. Refer to Section 7 Record TOC As-Found Readings to collect as-found TOC data.
  - c. Perform Section 11 TOC Calibration to complete the calibration process.
  - d. Perform Section 12 Record As-Left TOC Readings to collect as-left TOC readings.
7. To perform a complete system calibration, complete all sections of this procedure. If as-found data is not required, the following sections may be omitted:
  - a. Section 7 Record TOC As-Found Readings.
  - b. Section 13 Record As-Found Temperature Data.
  - c. Section 14 Record As-Found Conductivity Data.
8. Restore unit to operation or perform SST, as required.

## 4. Equipment Required

### 4.1. Full Calibration

For a complete calibration (flow, temperature, conductivity, and TOC), the following equipment is required.

- CAL/SST Module Kit (Thornton PN 58 091 566). This kit consists of a calibrated conductivity sensor, patch cable, 316L SS flow housing, holding brackets, SOP for Calibration, pump, power supply, and miscellaneous connectors and tubing, all stored in a case.
- An appropriate TOC Calibration Solution Set:  
For a Standard Calibration (Up to 1000 ppb)
  - 58 091 529: Calibration Standards solution kit for Standard TOC calibration
  - 58 091 537: Combination Calibration Standards solution kit for Standard TOC calibration plus System Suitability Standards
- Stop watch or other timing device with 1 second resolution for flow calibration process.



- 100 mL or larger volumetric container such as graduated cylinder with <3% accuracy tolerance for flow calibration process.
- Latex/nitrile gloves are recommended during the procedure when wetted components such as tubing are handled.

## 4.2. TOC Calibration

For a TOC calibration (TOC measurement only), the following equipment is required.

- TOC Cal/SST Module Kit (Thornton P/N 58 091 566 or equivalent) is required. The Cal/SST Module Kit is described above.
- TOC Calibration Solution Set (Thornton PN 58 091 529).

# 5. Reference and UUT Equipment Information

## 5.1. Record System Information

1. Record all relevant information regarding the unit under test (UUT) on a copy of the Calibration Worksheet found at the end of this document (or equivalent) in Section A: Equipment Information.
2. Record all relevant information regarding the reference system on a copy of the Calibration Worksheet found at the end of this document (or equivalent) in Section A: Equipment Information.

## 5.2. 450TOC Configuration for TOC Sensor Calibration

The 450TOC is capable of displaying values for a variety of different measurements and units. This extensive variety is supported by a four-line display and the Analyzer's "custom names" feature, which allows labeling of each measurement. To simplify the calibration process, the 450TOC can be configured to display the following measurements and units (see, 450TOC Portable TOC Analyzer Operation Manual for details on how to configure the transmitter display setup) during the calibration process. However, this configuration is not required.

Line 1: TOC displayed in ppb (parts per billion)

Line 2: TOC sensor Conductivity displayed in  $\mu\text{S}/\text{cm}$  (resolution: 3 decimal places)

Line 4: TOC sensor Temperature displayed in degrees Celsius ( $^{\circ}\text{C}$ ) (resolution: 1 decimal place).

This procedure will use conductivity in units of  $\mu\text{S}/\text{cm}$  and temperature in degrees Celsius. Alternative measurement units may be used if desired.

For a full system calibration, a separate reference conductivity sensor and transmitter must be used. Please refer to the appropriate documentation for configuration instructions. It is recommended that the reference transmitter be configured to display conductivity in  $\mu\text{S}/\text{cm}$  (resolution: 3 decimal places), and temperature in degrees Celsius ( $^{\circ}\text{C}$ , resolution: 1 decimal place).

# 6. Calibration Equipment Preparation

CAL/SST Module 58 091 566 with the appropriate transmitter provides the apparatus needed to perform a complete calibration on the 450TOC Portable TOC Analyzer (PN 58 036 041). These kits each consist of a calibrated conductivity sensor, patch cable, 316L SS flow housing, holding bracket, Operating Procedure for Calibration and SST, pump, power supply, and miscellaneous connectors and tubing, all stored in a case. This kit is designed for use with Thornton Standard Solution bottles included in the calibration Solutions Kit listed in Section 4.1 Full Calibration. In addition, the CAL/SST Pump Stand (58 091 586) is required.

## 6.1. TOC CAL/SST Module Setup for Temperature and Conductivity

1. Set up the CAL/SST Pump Stand on a clean, flat work surface by placing the four rubber feet on the work surface, and inserting the tabs at the bottom of the rear panel into the slots on the base.
2. Hang the CAL/SST Module on the mounting hook provided on the stand. Refer to figure 1.



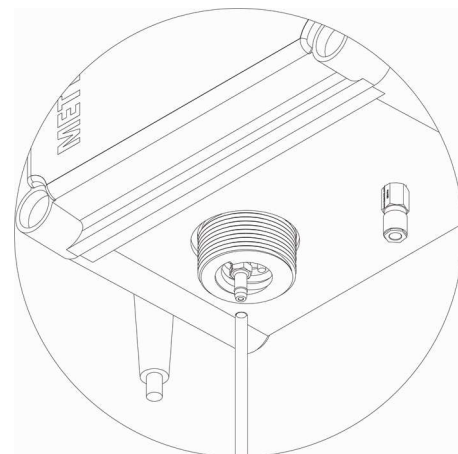
Figure 1. CAL/SST Pump Stand Setup

3. Remove the 60 micron filter assembly from the 450TOC inlet.
4. Attach the threaded end of connection tube equipped with the male quick-disconnect fitting to the 450TOC Sample Inlet connection. After the threaded connection is made, push the quick-disconnect fitting into the mating connector found on the lower right side of the CAL/SST pump module marked 'to TOC sample inlet'. Ensure proper connection is made.
5. Attach the threaded end of connection tube equipped with the female quick-disconnect fitting to the 450TOC Sample Outlet connection. After the threaded connection is made, push the quick-disconnect fitting into mating connector found on the lower right side of the CAL/SST pump module marked 'from TOC sample outlet'. Ensure proper connection is made.
6. Set the selector valve on the CAL/SST pump module to "Cond/Res CAL and Temp CAL" position. Figure 2 shows the proper installation of the CAL/SST pump module.
7. Verify the pump's power switch is in the off position. The switch is located on the left side of the module.
8. Connect the Universal Power Supply to the CAL/SST pump and to 100–240 VAC 50-60 Hz power. The kit includes a universal AC adaptor with assorted international standard connectors. Select the appropriate AC connector, and install it onto the AC adaptor and connect to the AC supply.
9. Remove the grey, threaded, protective cap from the bottom of the CAL/SST Pump Module.



**Figure 2. CAL/SST Pump Module Setup for Cond/Temp**

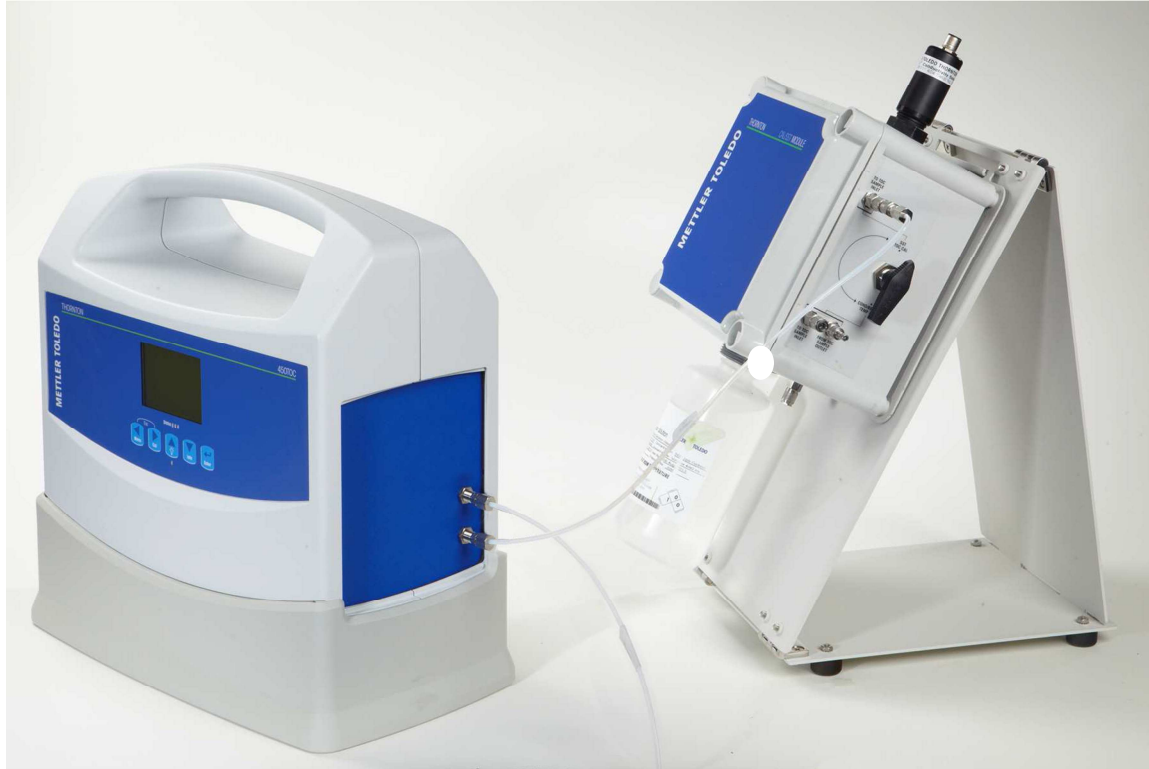
10. Wearing clean protective gloves to avoid contamination, insert a silicone suction tube onto the stainless steel suction fitting located in the bottle receptacle as shown in Figure 3. A fresh suction tube should be used for each calibration. Protective gloves and a replacement suction tube are provided with each solution kit.
11. Break the protective seal on a blank water bottle and remove cap. Set cap aside for re-use if any solutions remain after completion of the test.
12. Insert free end of the suction tube into the mouth of the bottle while raising the bottle into place. Attach the solution bottle directly into the solution bottle receptacle at the base of the CAL/SST pump module and proceed to 6.3 Final Installation of the TOC CAL/SST Module for Conductivity and Temperature.



**Figure 3. Suction Tube Installation**

## 6.2. TOC CAL/SST Module Setup for TOC and Flow

1. Set up the CAL/SST Pump Stand on a clean, flat work surface by placing the four rubber feet on the work surface, and inserting the tabs at the bottom of the rear panel into the slots on the base.
2. Hang the CAL/SST Module on the mounting hook provided on the stand. Refer to figure 4.
3. Remove the 60 micron filter assembly from the 450TOC inlet.
4. Attach the threaded end of the connection tube equipped with the male quick-disconnect fitting to the 450TOC Sample Inlet connection. After the threaded connection is made, push the quick-disconnect fitting into mating connector found on the lower right side of the CAL/SST pump module marked 'to TOC sample inlet'. Ensure proper connection is made.



**Figure 4. CAL/SST Pump Module Setup for TOC and Flow**

5. Set selector valve on CAL/SST pump module to “SST, TOC CAL” position. Figure 4 shows the proper installation of the CAL/SST pump module.
6. Verify the CAL/SST module power switch is in the off position. The switch is located on the right side of the module.
7. Connect the Universal Power Supply to the pump and to 100–240 VAC 50-60 Hz power. The kit includes a universal AC adaptor with assorted international standard connectors. Select the appropriate AC connector, and install it onto the AC adaptor and connect to the AC supply.
8. Remove the grey, threaded, protective cap from the bottom of the CAL/SST Pump Module.

### 6.3. Final Installation of the TOC CAL/SST Module for Conductivity and Temperature Calibration

1. If calibrating conductivity or temperature, connect the reference conductivity/temperature sensor to an appropriate transmitter or display.
2. Configure the reference transmitter readout to display the reference sensor temperature and temperature compensated conductivity.
3. Set the selector valve on the CAL/SST pump module to “Cond/Res CAL and Temp CAL” position. Figure 2 shows the proper installation of the CAL/SST pump module.
4. Energize the CAL/SST module pump. The setup should now be such that water continuously circulates through the Blank Water bottle and the 450TOC Analyzer.
5. The system is now ready for temperature or conductivity calibration. The pump may be left on if you are proceeding to the Section 9 Temperature Calibration.

## 7. Record TOC As-Found Readings

**Note:** If desired, as-found readings for TOC should be collected prior to beginning the maintenance and calibration process. It is not necessary to collect as-found temperature and conductivity readings prior to Analyzer calibration. As-found readings for flow will be collected as part of the flow calibration procedure.

Prior to performing any maintenance or calibration steps, collection of as-found TOC readings should be performed if desired. Perform these steps prior to replacement of the UV Lamp. Record this data in the As-Found portion of Section E of the Calibration Test Report.

1. Record Lamp Lifetime (hrs) as follows:
  - a. Press the Info (▼) key on the 450TOC.
  - b. Press ▲ or ▼ to select UV Lamp Time Remaining and press Enter.
  - c. Record the lamp life data displayed.
  - d. Simultaneously press Menu and Cal (◀ and ▶) to return to Measurement Mode.
2. Record the As Found TOC Multiplier(s) and Adder(s) as follows:
  - a. Press the Info (▼) key on the 450TOC.
  - b. Press ▲ or ▼ to select Calibration Data and press Enter.
  - c. Press ▲ or ▼ to select TOC and press Enter.
  - d. Record the calibration data displayed.
  - e. Simultaneously press Menu and Cal (◀ and ▶) to return to Measurement Mode.
3. Set up the CAL/SST kit as described in Section 6.2 TOC CAL/SST Module Setup for TOC and Flow and attach the 500 ppb calibration solution bottle to the CAL/SST module.
4. Ensure that the CAL/SST module selector valve is set to SST/TOC CAL.
5. Configure the 450TOC to display TOC readings.
6. Set the CAL/SST Module pump switch to on.
7. Allow the 500 ppb solution to flow through the TOC sensor for a minimum of 3 minutes to allow readings to stabilize.
8. Note the indicated TOC reading on the 450TOC, and record the value under TOC<sub>before</sub> in Section E: TOC Measurement Calibration Data of the calibration record found at the end of this manual.
9. Set the CAL/SST Module pump switch to off.
10. If a full calibration is to be performed, proceed to Section 8 Flow Rate Calibration. If only a TOC calibration is being performed, proceed to Section 11 TOC Calibration.

## 8. Flow Rate Calibration

### 8.1. Introduction to Flow Rate Calibration Process

This section describes the calibration/adjustment process for the flow rate. If a calibration of only the TOC measurement is scheduled, proceed directly to Section 11 TOC Calibration. If flow, temperature, and conductivity are to be calibrated, then continue.

This procedure uses a “volume and time” method for calibrating the flow sensor. Process water may be used for this calibration. The end of the drain tube at the sample outlet is the measurement point.

NOTE: Since it is possible for the person performing this calibration to come into contact with the process water, use of protective gloves, or other suitable safety equipment is recommended.

**Caution:** If the process water is hot, protection from direct contact should be used, or an alternate source of water must be provided.

Record this data in the As-Found portion of Section B of the Calibration Test Report.

### 8.2. Flow Rate Verification before Calibration

1. Record the as-found flow calibration factors:
  - a. Press the Info (▼) key on the 450TOC.
  - b. Press ▲ or ▼ to select Calibration Data and press Enter.

- c. Press ▲ or ▼ to select Flow and press Enter.
  - d. Record the calibration data displayed.
  - e. Simultaneously press Menu and Cal (◀ and ▶) to return to Measurement Mode.
2. Set up equipment as described in section 6.2 TOC CAL/SST Module Setup for TOC and Flow using Blank Water. Optionally, the flow rate verification and calibration may be done using process water, with the TOC Analyzer connected to the water system as normal.
  3. If using the CAL/SST Module, ensure the selector valve is set to the 'SST/TOC Cal' position.
  4. Arrange the drain tube at the atmospheric drain such that the water dripping from it may be collected into the volumetric container.
  5. Energize the pump on the CAL/SST module, or restore flow to the TOC Analyzer as necessary. If using the CAL/SST module, allow 30 seconds for the flow to stabilize. Place a dry volumetric container under the drain line to capture the water, and begin the time measurement using the stop watch. It is important that the water collection and start time be closely matched.
  6. Collect a known volume of water with the volumetric container for a specific time, at least 2 minutes.
  7. Turn off the CAL/SST pump.
  8. In Section B of the calibration sheet under Flow As-Found Data, record the volume of water in mL and the elapsed time in seconds. These two values determine the reference flow rate,  $F_{ref}$ .
  9. Calculate the reference flow rate and record as  $F_{ref}$  according to the equation below. The volume may also be determined by weight. Measure the mass increase of the container in grams, and divide the mass by the density of the water (typically, 1.00 g/mL at room temperature).

$$\text{Flow Rate (mL/min)} = F_{ref} = \frac{60 \text{ sec/min} \times \text{volume collected (mL)}}{\text{Elapsed Time (sec)}} \quad \text{Eq. 1}$$

10. If ( $F_{ref} - 20$ ) is within the limit, then flow rate adjustment is not required. If no flow adjustment is required, proceed to Section 9. Temperature Calibration. Otherwise, proceed to the next section to perform a flow rate calibration.

### 8.3. Flow Rate Calibration

1. On the 450TOC, press 'Cal' (▶) to enter calibration mode.
2. Press ▲ or ▼ to select Flow and press Enter.
3. Energize the CAL/SST pump to initiate flow to the 450TOC, and follow the on-screen prompts.
4. The 450TOC Analyzer will automatically adjust the flow rate to the first test measurement value.
5. Place a dry volumetric container under the drain line to capture the water, and begin the time measurement using the stop watch. It is important that the water collection and start time be closely matched.
6. Collect a known volume of water with the volumetric container for a measured time, at least 2 minutes.
7. In Section B of the calibration worksheet under Flow Sensor Calibration, record the volume of water in mL and the elapsed time in seconds.
8. Using eq 1 calculate the water flow rate and record as  $F_{ref1}$ .
9. Enter the reference flow rate into the 450TOC and press Enter.
10. The 450TOC will automatically adjust the flow rate to the second test measurement value.
11. Empty the volumetric container, and repeat steps 6-8.
12. Using eq 1 calculate the water flow rate and record data as  $F_{ref2}$ .
13. Enter the reference flow rate into the 450TOC and press Enter. Record the calibration factors in Section B of the calibration worksheet under Flow Sensor Verification after Calibration.
14. Press Enter to save the new calibration data within the 450TOC. If 'No' is selected, the previous calibration will be restored.

## 8.4. Flow Rate Verification After Calibration

1. Repeat steps described in Section 8.2 Flow Rate Verification before Calibration to verify the flow sensor is in calibration.
2. Record data in section B of the calibration worksheet under Flow Sensor Verification After Calibration.

Flow Calibration is complete.

## 9. Temperature Calibration

This procedure is to be performed on T1 and T2, which correspond to the temperature sensors embedded in the two conductivity sensors within the 450TOC. In order to record as-left readings, configure the 450TOC to display the temperature reading.

1. Record data in section C of the calibration worksheet under Temperature Sensor Calibration and Verification.
2. Turn off the UV lamp (if not already off) by pressing the lamp button so that the green UV Lamp On LED is off. The lamp shall remain off throughout this procedure.
3. Set up the Cal/SST Module using Blank Water as described in Section 6.1.
4. Ensure the selector valve on the CAL/SST module is set to "Cond/Res and Temp Cal" position.
5. On the 450TOC, press 'Cal' (▶) to enter calibration mode.
6. Press ▲ or ▼ to select Temperature1 and press Enter.
7. Press enter. A 1-Point Offset calibration is the only method available for calibration of the TOC temperature measurement.
8. Turn on the pump. Water should now be continuously circulating through the pump, 450TOC Analyzer, Reference Conductivity Sensor, the bottle containing the blank water, and back through the pump.
9. Press Enter.
10. Allow the pump to run until thermal equilibrium is achieved, typically 1/2 hour (less if the lamp has been off).
11. Enter the reference value into the 450TOC once the reference reading has stabilized. To avoid inaccuracy due to changes in ambient temperature, this value should be entered as soon as possible after reading the Reference Sensor.
12. Record the multiplier (M) and adder (A) for the calibrated temperature measurement in section C of the calibration worksheet under Temperature Sensor Calibration and Verification.
13. Press Enter to save the new calibration data within the 450TOC. If 'No' is selected, the previous calibration will be restored.
14. After returning to measurement mode on the 450TOC, record the temperature of  $T_{ref}$  and  $T_{offer}$  for T1 on the calibration worksheet. Under Temperature Sensor Calibration and Verification.
  - a. Record the temperature reading of the reference temperature sensor as  $T_{ref}$ .
  - b. Record the temperature reading as indicated by the TOC Analyzer as  $T_{offer}$ .
15. Verify that  $T_{ref} - T_{offer}$  is within limits for T1. If they exceed limits, repeat the temperature calibration procedure.
16. Repeat steps 5-13, selecting Temperature2 at step 6.

## 10. Conductivity Calibration

This procedure is to be performed on C1 and C2 which correspond to the two conductivity sensors within the Analyzer. In order to record as-left readings, configure the 450TOC to display the conductivity reading.

1. Record data in section D of the calibration worksheet under Conductivity Sensor Calibration and Verification.
2. Turn off the UV lamp (if not already off) by pressing the lamp button so that the green UV Lamp On LED is off. The lamp shall remain off throughout this procedure.
3. If not already done, set up the CAL/SST Module using Blank Water as described in section 6.1.
4. Ensure the selector valve on the CAL/SST module is set to "Cond/Res Cal and Temp Cal" position.
5. If a Temperature calibration has just been performed, continue using the same Blank Water bottle. Verify that the water conductivity is  $<1.5 \mu\text{S}/\text{cm}$  ( $>0.67 \text{ M}\Omega\text{-cm}$ ) as reported by the Reference Conductivity sensor. If the water conductivity

has increased above 1.5  $\mu\text{S}/\text{cm}$ , then turn off the pump, install a fresh bottle of Blank Water, and restart the pump. Note that the conductivity sensors can be calibrated above 1.5  $\mu\text{S}/\text{cm}$  with no adverse measurement impact, but it is recommended a calibration value that closely matches values found in the process water being monitored by the Analyzer.

6. If the Blank Water bottle has been changed, allow the pump to run until complete mixing and thermal equilibrium is achieved, typically 30 minutes. Otherwise, continue to the next step
7. On the 450TOC, press 'Cal' (▶) to enter calibration mode.
8. Press ▲ or ▼ to select Conductivity1 and press Enter.
9. Press ▲ or ▼ to select the conductivity compensation method to be used during calibration. 'Standard' compensation is recommended.
10. Press Enter. A 1-Point calibration is the only method available for calibration of the TOC conductivity measurement.
11. Turn on the pump. Water should now be continuously circulating through the pump, 450TOC Analyzer, Reference Conductivity Sensor, the bottle containing the blank water, and back through the pump.
12. Press Enter.
13. Once the readings from the reference sensor and C1 have stabilized, enter the conductivity reading of the Reference Sensor into the 450TOC using the arrow keys and press Enter. To avoid inaccuracy due to changes in ambient temperature, this value should be entered as soon as practical after reading the Reference Sensor.
14. Record the multiplier (M) and adder (A) for the calibrated conductivity measurement in section D of the calibration worksheet under Conductivity Sensor Calibration and Verification.
15. Press Enter to save the new calibration data within the 450TOC. If 'No' is selected, the previous calibration will be restored.
16. After returning to measurement mode on the 450TOC, record the indicated conductivity for C1 and  $C_{\text{ref}}$  on the calibration worksheet under Conductivity Sensor Calibration and Verification.
  - a. Record the conductivity reading of the reference sensor as  $C_{\text{ref}}$ .
  - b. Record the conductivity reading as indicated by the TOC Analyzer as  $C_{\text{after}}$ .
17. Compute the % difference according to eq 2.

$$\frac{100 \times (C_{\text{after}} - C_{\text{ref}})}{C_{\text{ref}}} \quad \text{Eq. 2}$$

18. Verify that the % difference is within limits. If not, repeat the Conductivity Calibration process.
19. Repeat steps 7-15, selecting Conductivity2 at step 8.



## 11. TOC Calibration

Calibration is performed using TOC Standard Solutions of known concentration. Mettler-Toledo Thornton provides Calibration Solution Sets for this purpose. Refer to Section 4.1 for more detail on which kits should be used for calibration. If performing a 1-point slope calibration, then use only the bottle of 500 ppb TOC (sucrose) calibration solution.

1. Record data in section E of the calibration worksheet under TOC Measurement Calibration and Verification.
2. If not already completed, set up the CAL/SST kit as described in Section 6.2 TOC CAL/SST Module Setup for TOC and Flow and attach the 250 ppb calibration solution bottle to the CAL/SST module.
3. On the 450TOC, press 'Cal' (▶) to enter calibration mode.
4. Press ▲ or ▼ to select TOC and press Enter.
5. Select the calibration method to be performed:
  - a. Normally, the Standard calibration method is performed, and is ideal for most applications monitoring TOC levels below 1000 ppbC. Solution Kit PNs 58 091 529 or 58 091 537 should be used for this calibration.
  - b. 1-point Slope calibration is a special purpose calibration which should only be used in very specific situations. Please refer to the 450TOC instruction manual for guidance.
6. Turn on the pump.
7. Press Enter.
8. Allow the 250 ppb TOC solution to flow the 450TOC Analyzer until the readings stabilize, approximately 5 minutes.
9. Use the arrow keys on the 450TOC to enter the TOC value of the Reference Solution as written on the label or its certificate into the 450TOC for Cal point 1 and press Enter.
10. Turn off the pump.
11. Remove the 250 ppb TOC solution bottle from the calibration apparatus and replace it with the 500 ppb TOC solution.
12. Turn on the pump.
13. Verify the UV lamp is on. The lamp must be on to measure and display TOC. If the 450TOC is not set to automatically restart, the lamp will need to be energized manually.
14. Allow the 500 ppb TOC solution to flow the 450TOC Analyzer until the readings stabilize, approximately 5 minutes.
15. Use the arrow keys on the 450TOC to enter the TOC value of the Reference Solution as written on the label or its certificate into the 450TOC for Cal point 2 and press Enter.
16. Record the multiplier(s) (M) and adder(s) (A) for the calibrated TOC measurement in section E of the calibration worksheet under TOC Measurement Calibration and Verification.
17. Press Enter to save the new calibration data within the 450TOC. If 'No' is selected, the previous calibration will be restored.

## 12. Record As-Left TOC Readings

1. If not already completed, set up the CAL/SST kit as described in Section 6.2. If a calibration standard is not already installed on the CAL/SST kit, attach the first calibration standard bottle to the CAL/SST module.
2. Ensure that the CAL/SST module selector valve is set to SST/TOC CAL.
3. Set the CAL/SST Module pump switch to on.
4. Allow the installed solution to flow through the TOC analyzer for a minimum of 5 minutes to allow readings to stabilize.
5. Note the indicated TOC reading on the 450TOC, and record the value under TOC<sub>after</sub> in Section E of the calibration worksheet under TOC Measurement Calibration and Verification.
6. Set the CAL/SST Module pump switch to off.
7. Repeat steps 1 to 8 for each calibration standard used.

## 13. Record As-Found Temperature Data

If required for retention, collect as-found temperature calibration data using the following procedure.

1. Record the As Found Temperature T1 data in Section C of the calibration worksheet under Temperature As-Found Data.
2. Record the as-found T1 calibration factors:
  - a. Press the Info (▼) key on the 450TOC.
  - b. Press ▲ or ▼ to select Calibration Data and press Enter.
  - c. Press ▲ or ▼ to select Temperature and press Enter.
  - d. Record the calibration data displayed.
  - e. Simultaneously press Menu and Cal (◀ and ▶) to return to Measurement Mode.
3. Turn off the UV lamp (if not already off) by pressing the lamp button so that the green UV Lamp On LED is off. The lamp shall remain off throughout this procedure.
4. If not already done, set up the Cal/SST Module using Blank Water as described in section 6.1.
5. Ensure the selector valve on the CAL/SST module is set to "Cond/Res Cal and Temp Cal" position.
6. Turn on the pump. Water should now be continuously circulating through the pump, 450TOC Analyzer, Reference Conductivity Sensor, the bottle containing the blank water, and back through the pump.
7. In Section C of the calibration sheet under Temperature As-Found data, record the Reference Sensor temperature,  $T_{ref}$
8. Record the T1 temperature displayed on the 450TOC,  $T_{before}$ .
9. Compute the difference,  $T_{before}-T_{ref}$ .
10. If the absolute value of the difference is less than the limit, then a temperature calibration is not required.

## 14. Record As-Found Conductivity Data

If required for retention, collect as-found conductivity calibration data using the following procedure.

1. Record the As Found Conductivity C1 reading in Section D of the calibration worksheet under Conductivity As-Found Data.
2. Record the as-found C1 calibration factors:
  - a. Press the Info (▼) key on the 450TOC.
  - b. Press ▲ or ▼ to select Calibration Data and press Enter.
  - c. Press ▲ or ▼ to select Conductivity and press Enter.
  - d. Record the calibration data displayed.
  - e. Simultaneously press Menu and Cal (◀ and ▶) to return to Measurement Mode.
3. If a Temperature calibration has just been performed, continue using the same Blank Water bottle. Verify that the water conductivity is  $<1.5 \mu\text{S}/\text{cm}$  ( $>0.67 \text{ M}\Omega\text{-cm}$ ) as reported by the Reference Conductivity sensor. If the water conductivity has increased above  $1.5 \mu\text{S}/\text{cm}$ , then turn off the pump, install a fresh bottle of Blank Water, and restart the pump. Note that the conductivity sensors can be calibrated above  $1.5 \mu\text{S}/\text{cm}$  with no adverse measurement impact, but it is recommended a calibration value that closely matches values found in the process water being monitored by the Analyzer.
4. If the same bottle of Blank Water is still being used from the previous section, then continue to the next step. Otherwise, allow the pump to run until complete mixing is achieved, typically 30 minutes.
5. Record the conductivity of the Reference Conductivity Sensor,  $C_{ref}$ .
6. Record the C1 conductivity displayed on the 450TOC,  $C_{before}$ .
7. Compute the difference according to the equation 3:

$$\frac{100 \times (C_{before} - C_{ref})}{C_{ref}}$$

Eq. 3

8. If the absolute value of the % difference is less than the limit, then a C1 conductivity calibration is not required.

## 15. Restore to Operation

1. Restore the drain and supply connections to the 450TOC to the same operational configuration prior to calibration.
2. Connect the process water line to the 450TOC Analyzer sample inlet.
3. Turn on the process water supply to the 450TOC Analyzer using the shut off valve (recommended in the 450TOC Portable TOC Analyzer Operation Manual). Ensure the process water supply is turned on by viewing the discharge line from the TOC analyzer at the atmospheric drain.
4. Turn on the UV Lamp.
5. Flush the CAL/SST Module and connected tubing with blank water for 3 to 5 minutes. If there is not adequate blank water remaining, a blank water bottle may be filled with RO quality water or better. Flush the CAL/SST Module. Ensure that the drain tube is directed to an appropriate drain. If an adequate drain is not available, use an empty test solution bottle to collect the flushing water.
6. If a temperature and/or conductivity calibration was performed, proceed to the next section to flush and drain the reference conductivity sensor and housing. Otherwise, remove the blank water bottle from the CAL/SST module, remove and discard the silicon suction tube, and replace the protective cap for the solution bottle interface. Remove the CAL/SST module and mounting bracket from the TOC Analyzer.

## 16. Flush and Drain Reference Conductivity Sensor and Housing

During conductivity and temperature calibration, the flow housing containing the reference conductivity sensor will become filled with test solution. It is necessary to flush and drain the conductivity sensor flow housing prior to storage of the CAL/SST Module.

9. Connect both provided tubes to the quick-disconnect fittings on the lower right side of the CAL/SST Module.
10. Set the selector valve to the "Cond/Res and Temp Cal" position.
11. Connect a bottle of the supplied blank water to the CAL/SST Module. If the remaining blank water is not sufficient, a blank water bottle may be filled with process water of RO quality or better to flush the CAL/SST Module.
12. Turn the pump on and flush the CAL/SST Module for a minimum of 5 minutes. Ensure that the discharge tube from the CAL/SST module (marked "To TOC Sample Inlet") is directed to an appropriate drain. If an adequate drain is not available, use an empty test solution bottle to collect the flushing water.
13. Remove the blank water bottle from the CAL/SST module. Allow the CAL/SST Module pump to run until all solution has been discharged from the CAL/SST module.
14. Remove and discard the silicon suction tube from the solution bottle interface, and replace the protective cap.
15. With both lines attached to the CAL/SST module and the selector valve in the Con/Res and Temp Cal position, connect the provided syringe and quick-disconnect assembly to the quick-disconnect fitting located at the bottom of the CAL/SST module. Pull the plunger of the syringe back to draw the residual water out of the Conductivity sensor flow housing. Remove the syringe from the silicone tubing and empty the syringe into an appropriate drain or container. If necessary, repeat this process until all solution has been removed.  
Remove the CAL/SST module and mounting bracket from the TOC Analyzer. Remove all connected tubing from the CAL/SST module and return all components to the supplied storage case.



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SECTION A: Equipment Information					
450TOC Portable TOC Analyzer Unit Under Test (UUT) Data					
Part Number		Control FW Version			
Serial Number		Comm FW Version			
ID Tag (if available)		TOC FW Version			
Date Last Calibrated		TOC HW Version			
Date Calibration Due		DCS FW Version			
		USB FW Version			
Reference Transmitter Data					
Model:					
Part Number:		Date Last Calibrated:			
Serial Number:		Date Calibration Due:			
Reference Conductivity/Temperature Sensor Data (if needed)					
Model:					
Part Number:		Date Last Calibrated:			
Serial Number:		Date Calibration Due:			
<b>Reference TOC Solution</b>	<b>Manufacturing Date</b>		<b>Lot #</b>		
250 ppb TOC Solution:*					
500 ppb TOC Solution:*					
Other Reference Information and Notes					
Graduated Cylinder:					
Other:					
SECTION B: Flow Sensor Calibration Data					
Flow As-Found Data					
Calibration point and conditions	Volume (mL)	Elapsed Time (sec)	Reference Flow Rate $F_{ref}$ (mL/min)	Limit (mL/min)	Within Limits (Y/N)
20 mL/min	/	* 60 =		±1.5	
Flow Calibration Factors		F Multiplier (M)		F Adder (A)	
Flow Sensor Calibration					
Calibration point and conditions	Volume (mL)	Elapsed Time (sec)		Reference Flow Rate (mL/min)	
#1 ~18 mL/min				$F_{ref1}$	
#2 ~22 mL/min				$F_{ref2}$	
Flow Sensor Verification After Calibration					
Calibration point and conditions	Volume (mL)	Elapsed Time (sec)	Reference Flow Rate $F_{ref}$ (mL/min)	Limit (mL/min)	Within Limits (Y/N)
20 mL/min	/	* 60 =		±1.5	
Flow Calibration Factors		F Multiplier (M)		F Adder (A)	

\*Mark N/A if not needed for current calibration method

Performed By: \_\_\_\_\_  
 Name (Print) Signature Date

SECTION C: Temperature Sensor Calibration Data					
Temperature As-Found Data					
Measurement Type and Sensor #	T1 Standard T <sub>ref</sub> (°C)	T1 As Found T <sub>before</sub> (°C)	T <sub>before</sub> - T <sub>ref</sub> (°C)	Limit (°C)	Within Limits (Y/N)
Temperature T1				±0.5	
Temperature Calibration Factors		T1 Multiplier (M)		T1 Adder (A)	
Temperature Sensor Calibration and Verification					
Measurement Type and Sensor #	Reference Temperature T <sub>ref</sub> (°C)	Temperature After T <sub>after</sub> (°C)	T <sub>after</sub> - T <sub>ref</sub> (°C)	Limit (°C)	Within Limits (Y/N)
Temperature T1				±0.5	
Calibration Factors	Factor			Factor	
T1 Multiplier (M)			T1 Adder (A)		
T2 Multiplier (M)			T2 Adder (A)		
SECTION D: Conductivity Sensor Calibration Data					
Conductivity As-Found Data					
Measurement Type and Sensor #	C1 Standard C <sub>ref</sub> (µS/cm)	C1 As Found C <sub>before</sub> (µS/cm)	$\frac{100 \times (C_{\text{before}} - C_{\text{ref}})}{C_{\text{ref}}}$ (%)	Limit (%)	Within Limits (Y/N)
Conductivity C1				±2.0	
Conductivity Calibration Factors		Multiplier (M)		Adder (A)	
Conductivity Sensor Calibration and Verification					
Measurement Type and Sensor #	Reference Conductivity C <sub>ref</sub> (µS/cm)	As Left Conductivity C <sub>after</sub> (µS/cm)	$\frac{100 \times (C_{\text{after}} - C_{\text{ref}})}{C_{\text{ref}}}$ (%)	Limit (%)	Within Limits (Y/N)
Conductivity C1				±2.0	
Calibration Factors	Factor			Factor	
C1 Multiplier (M)			C1 Adder (A)		
C2 Multiplier (M)			C2 Adder (A)		
SECTION E: TOC Measurement Calibration Data					
TOC As-Found Data					
TOC Lamp Hours Remaining					
Nominal	Reference TOC <sub>ref</sub> (ppb)	As Found TOC <sub>before</sub> (ppb)	TOC <sub>ref</sub> - TOC <sub>before</sub> (ppb)	Limit (ppb)	
TOC, 500 ppb				±50	
TOC Calibration Factors	Multiplier (M1)		Adder (A1)		
	Multiplier (M2)*		Adder (A2)*		
TOC Measurement Calibration and Verification					
Nominal	TOC <sub>ref</sub> (ppb)	TOC <sub>after</sub> (ppb)	TOC <sub>after</sub> - TOC <sub>ref</sub> (ppb)	Limit (ppb)	Within Limits (Y or N)
TOC, 250 ppb				±25	
TOC, 500 ppb				±50	
TOC Calibration Factors	Multiplier (M1)		Adder (A1)		
	Multiplier (M2)*		Adder (A2)*		

\*Required for Standard only. Mark N/A if not needed for current calibration method

Performed By: \_\_\_\_\_  
 Name (Print) Signature Date

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