5000TOCe Sensor
Standard Operating Procedure for

- TOC Calibration
- Conductivity Calibration
- Temperature Calibration
- Flow Rate Calibration
IMPORTANT SAFETY INFORMATION

Please read thoroughly before operating the 5000TOCe System Suitability Test Kit and the 5000TOCe 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 instrument 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 instrument damage or malfunction.

NOTE: important operating information.

Definition of Equipment Symbols

⚠️ On the instrument indicates: Warning, risk of electric shock.

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

∼ On the instrument indicates: There is alternating current present.
This page is intentionally blank.
## TABLE OF CONTENTS

1. Introduction ................................................................................................................................. 1
2. Scope ........................................................................................................................................ 1
3. Equipment Required .................................................................................................................... 1
   3.1. Full Calibration .................................................................................................................... 1
   3.2. TOC Calibration ................................................................................................................... 1
4. Reference and UUT Equipment Information ................................................................................ 1
   4.1. Record System Information .................................................................................................. 1
   4.2. 770MAX Configuration ....................................................................................................... 1
5. Calibration Equipment Preparation ............................................................................................... 2
   5.1. Install CAL/SST Module (part no. 58 091 559) .................................................................... 2
   5.2. TOC Calibration and CAL/SST Module Setup ...................................................................... 2
   5.3. Final Installation of the 5000TOCe Sensor Calibration Kit .................................................... 4
6. Record "As Found" Information ..................................................................................................... 4
   6.1. T1 Temperature As Found ...................................................................................................... 4
   6.2. C1 Conductivity As Found .................................................................................................... 4
   6.3. TOC As Found ..................................................................................................................... 5
7. Flow Rate Calibration .................................................................................................................... 6
   7.1. Introduction to Flow Rate Calibration Process ......................................................................... 6
   7.2. Flow Rate Verification Before Calibration ............................................................................. 6
   7.3. Flow Rate Calibration ........................................................................................................... 6
   7.4. Flow Rate Verification After Calibration ................................................................................ 8
8. Temperature Calibration ................................................................................................................. 8
   8.1. T1 Temperature Calibration .................................................................................................. 8
   8.2. T2 Temperature Calibration .................................................................................................. 9
9. Conductivity Calibration ............................................................................................................... 9
   9.1. C1 Conductivity Calibration ................................................................................................ 9
   9.2. C2 Conductivity Calibration ................................................................................................ 10
10. TOC Calibration .......................................................................................................................... 11
    10.1. Set Up TOC Calibration Kit ................................................................................................ 11
    10.2. TOC Calibration Procedure ............................................................................................... 11
11. Restore to Operation ...................................................................................................................... 13
12. Flush and Drain Reference Conductivity Senor and Housing ....................................................... 14
    Worksheet 1: Unit Under Test Information .................................................................................. 15
    Worksheet 2: Reference Equipment Information ......................................................................... 16
    Worksheet 3: As Found Data for T1 and C1 .............................................................................. 17
    Worksheet 4: As Found Data for TOC ...................................................................................... 18
    Worksheet 5: Flow Rate Verification and Calibration ................................................................. 19
    Worksheet 6: Temperature Sensor Verification and Calibration .................................................. 20
    Worksheet 7: Conductivity Sensor Verification and Calibration ................................................. 21
    Worksheet 8: TOC Calibration and Verification ........................................................................... 22
13. Appendix 1: Calibration Process ................................................................................................ 23
   13.1. Scope .................................................................................................................................. 23
   13.2. Sequence of Operations for Calibration, SST and Lamp Changes ..................................... 23
   13.3. Summary of Calibration Process ......................................................................................... 23
14. Appendix 2 - Setting Sample Flow Rate .................................................................................... 24
   14.1. Flow Rate Adjustment Procedure ....................................................................................... 24
1. Introduction

The Mettler-Toledo Thornton 5000TOCe Total Organic Carbon Sensor and 770MAX meter measures the amount of organic carbon in high purity waters by oxidizing organic carbon to CO₂ with appropriate UV radiation. The resulting increase 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 5000TOCe or 5000TOC Total Organic Carbon Sensor (PN 58 036 001, PN 58 036 002, 58 036 003, 58 036 004). The 5000TOCe Sensor is used with the Thornton 770MAX transmitter. Calibration of the 770MAX transmitter is covered in separate documentation. Throughout this manual, references will be made to the 5000TOCe sensor model. In all cases, this can be interpreted to include the 5000TOCe and 5000TOC sensor models.

3. Equipment Required

3.1. Full Calibration

For a complete calibration (flow, temperature, conductivity, and TOC), the following equipment is required.
• 5000TOCe CAL/SST Module Kit (Thornton PN 58 091 559). This 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.
• 5000TOCe Calibration Solution Set (Thornton PN 58 091 529).
• 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% tolerance for flow calibration process.
• Latex/nitrile gloves are recommended during the procedure when wetted components such as tubing are handled.

Note: Instructions for use of the previous version of the 5000TOCe Calibration and SST Kits (Thornton PN 58 091 534, or PN 58 091 525 with 58 091 528) can be found in Appendix 3 – Using SST and Calibration kit 58 0791 534.

3.2. TOC Calibration

For a TOC calibration (TOC measurement only), the following equipment is required.
• 5000TOCe Calibration Test Kit (Thornton PN 58 091 534) or System Suitability Test Kit (Thornton PN 58 091 525) is required. The 5000TOCe Calibration Test Kit is described above. The System Suitability Test Kit consists of a holding brackets, pump, power supply, and miscellaneous connectors and tubing, all stored in a case.
• 5000TOCe Calibration Solution Set (Thornton PN 58 091 529).

4. Reference and UUT Equipment Information

4.1. Record System Information

1. Record all relevant information regarding the unit under test (UUT) in Worksheet 1: Unit Under Test Information.
2. Record all relevant information regarding the reference system in Worksheet 2: Reference Equipment Information.

4.2. 770MAX Configuration

The 770MAX accepts input from multiple sensors from which it computes values in many different units. This extensive variety is supported by a multi-line scrolling display and the instrument's "custom names" feature, which allows labeling of each measurement. To simplify the calibration process, the 770MAX can be configured to display the following measurements and units (see, 770MAX Multiparameter Analyzer/Transmitter Instruction Manual, part number 84372) during the calibration process. However, this configuration is not required.

Measurement A: TOC displayed in ppb (parts per billion)
Measurement B: Conductivity displayed in µS/cm (resolution: 3 decimal places)
Measurement C: Temperature displayed in degrees Celsius (°C) (resolution: 2 decimal places)
Measurement D: Reference Conductivity displayed in µS/cm (resolution: 3 decimal places)
Measurement E: Reference temperature displayed in degrees Celsius (°C) (resolution: 2 decimal places)

To simplify the calibration process, this procedure will use conductivity in units of µS/cm and temperature in degrees Celsius. Alternative measurement units may be used.

5. **Calibration Equipment Preparation**

Note: Use a hold time to freeze the relay and analog outputs so that alarms are not triggered during any service procedures. Normal relay and analog output operation resumes when the set period has elapsed or when the hold time is reset to zero. Set hold time by pressing “Menu” from the 770MAX main display. Scroll to “Set Hold time” (v5.1 or earlier) or “Hold Outputs” (v5.2 or later) by using the up/down arrows. Press “Enter”, then enter a time between 1 to 99 minutes, and press “Menu”. The hold time is now set.

Caution: Be sure to adjust needle valve to less than 3 turns from closed position before re-installing into process line!

5.1. **Install CAL/SST Module (part no. 58 091 559)**

This section discusses the installation process for CAL/SST Module kit P/N 58 091 559. For use of the previous version of the 5000TOCe Calibration and SST Kits (Thornton PN 58 091 534, or PN 58 091 525 with 58 091 528), refer to Appendix 3 – Using SST and Calibration kit 58 0791 534.

CAL/SST Module Kit 58 091 559 provides the apparatus needed to perform a system suitability test on the 5000TOCe Sensor (PN's 58 036 001, 58 036 002, 58 036 003 and 58 036 004). Components contained in this kit include the pump module, a mounting bracket, connecting tubing equipped with quick-disconnect fittings, a universal power supply and assorted tools. This kit is designed for use with Thornton Standard Solution bottles included in the calibration Solutions Kit (p/n 58 091 529).

5.2. **TOC Calibration and CAL/SST Module Setup**

1. Remove the mounting bracket from the carrying case and place on top of the 5000TOCe Sensor to be tested. See Figure 1.
2. Connect the CAL/SST Module to the mounting bracket. Ensure that the bracket and module are aligned to the left side of the sensor so that UV Lamp power button is accessible and the indicating LED’s are visible.
3. Remove the 60 micron filter assembly from the 5000TOCe inlet.
4. Attach the threaded end of connection tube equipped with the male quick-disconnect fitting to the 5000TOCe 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. Ensure proper connection is made.
5. Attach the threaded end of connection tube equipped with the female quick-disconnect fitting to the 5000TOCe 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. Ensure proper connection is made.
6. Set selector valve on CAL/SST pump module to “Cond/Res CAL and Temp CAL” position. Figure 1 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 right side of the module.
8. 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.
9. Remove the threaded grey, protective cap from the bottom of the CAL/SST Pump Module.

10. Wearing clean protective gloves to avoid contamination, insert a silicone suction tube onto the suction fitting located in the bottle receptacle as shown in Figure 2. A fresh suction tube should be used for each SST test. Protective gloves and a replacement suction tube are provided with each SST solution kit.

11. Break the protective seal on the SST solution bottle and remove cap. Set cap aside for re-use if any SST 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. Screw SST solution bottle directly into the solution bottle receptacle at the base of the CAL/SST pump module.

13. Install a bottle of blank water onto the CAL/SST Module now, and proceed to the next section.
5.3. Final Installation of the 5000TOCe Sensor Calibration Kit

1. Connect the Reference Conductivity sensor smart connector to the 770MAX patch cable.
2. Connect the other end of the 770MAX patch cable to an unused calibrated channel on the 770MAX transmitter. If no channel is available, then another calibrated 770MAX transmitter should be utilized.
3. Set up a measurement to display the Reference Sensor temperature and temperature compensated conductivity. For example, Measurement B could be set to display conductivity and Measurement C could be set to display temperature (see suggested 770MAX configuration in Section 4.2 770MAX Configuration).
4. Turn on the pump. The setup should now be such that water continuously circulates through the Blank Water bottle and the 5000TOCe Sensor.
5. Adjust the flow rate to its maximum rate. This may be done by adjusting the needle valve knob fully counterclockwise until it stops.
6. The system is now ready for temperature calibration. The pump may be left on if you are proceeding to the next section.

6. Record “As Found” Information

Prior to the calibration/adjustment process or prior to a lamp change, “As Found” data may be required prior to any changes. This section describes the procedure to acquire the “As Found” data. This is recommended for displayed measurements.

If a calibration of only the TOC measurement is scheduled, go to Section 6.3. TOC As Found. If the temperature and conductivity are to be calibrated, then continue to Section 6.1. T1 Temperature As Found.

6.1. T1 Temperature As Found

This procedure is to be performed on T1, which corresponds to the temperature sensor embedded in the inlet conductivity sensor.

1. Record all data in Worksheet 3: As Found Data for T1 and C1.
2. Turn off the UV lamp (if not already off). If necessary, press the Lamp button located on the front panel of the 5000TOCe Sensor. The lamp shall remain off throughout this procedure.
3. If the pump is off, turn on the pump. Water should now be continuously circulating through the pump, 5000TOCe Sensor, Reference Conductivity Sensor, the water container, and back through the pump.
4. Allow the pump to run until thermal equilibrium is achieved, typically 1/2 hour (less if the lamp has been off).
5. Record the As Found Temperature T1 Multiplier and Adder found in the Measurement Menu on the 770MAX.
6. Record the Reference Sensor temperature (to two decimal places) displayed on the 770MAX, T_ref.
7. Record the UUT temperature of T1 (to two decimal places) displayed on the 770MAX, T_before.
8. Compute the difference, T_ref - T_before.
9. If the absolute value of the difference is less than the limit, then a temperature calibration is not required.
10. Press Menu (exit) to return to measurement mode.

6.2. C1 Conductivity As Found

This procedure is to be performed on C1 which corresponds to the inlet conductivity sensors within the unit.

1. Record all data in Worksheet 3: As Found Data for T1 and C1.
2. Verify the UV lamp is off. If the lamp was on, wait until thermal equilibrium is achieved.
3. Continuing with the same Blank Water from the Temperature Calibration, verify that the water quality is <1.5 µS/cm (>0.67 MΩ·cm) as reported by Reference Conductivity sensor. If the water conductivity has increased above 1.5 µS/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 µS/cm with no adverse measurement impact, but we recommend a lower conductivity calibration to emulate process water.
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 As Found Conductivity C1 Multiplier and Adder found in the Measurement Menu on the 770MAX.
6. Record the conductivity (at least three significant figures) of the Reference Conductivity Sensor displayed on the 770MAX, \( C_{\text{ref}} \).
7. Record the UUT conductivity of C1 (at least three significant figures) displayed on the 770MAX, \( C_{\text{before}} \).
8. Compute the % difference according to eq 1.

\[
\frac{100 \times (C_{\text{before}} - C_{\text{ref}})}{C_{\text{ref}}} \quad \text{eq 1}
\]

9. If the absolute value of the % difference is less than the limit, then a C1 conductivity calibration is not required.
10. Press Menu (exit) to return to measurement mode.

6.3. TOC As Found

Record all data in the following worksheet: Worksheet 4: As Found Data for TOC

1. Disconnect tube from the TOC sample outlet and remove it completely from the test assembly.
2. At the CAL/SST Module, disconnect the tube attached to the TOC Sample Inlet, and re-connect it to the mating connector found on the top right-side of the CAL/SST Module.
3. Re-position the selector valve on the CAL/SST pump module to “SST/TOC CAL” position.
4. Record the As Found TOC Multiplier(s) and Adder(s) found in the Measurement Menu on the 770MAX.
5. Access the 770MAX TOC menus and record Lamp Lifetime (hrs).
6. Record the TOC of the Reference Solution, \( \text{TOC}_{\text{ref}} \).
7. Remove the Blank Water bottle from the CAL/SST Moduel and replace it with a 500 ppb TOC (sucrose) solution bottle.
8. Re-connect the original drain tube to the Sample Outlet of the 5000TOCe Sensor and run it to atmospheric drain. It is critical not to re-circulate this solution. Tubing should now appear as in Figure 3.

Figure 3

9. Turn on the pump. Water should now be visible at the drain line at the atmospheric drain. Note: the ~500 mL volume of the calibration solution will provide more than 20 minutes of operation at the nominal flow rate of 20 mL/min.
10. Verify that UV Lamp is on. The lamp must be on to measure and display TOC.
11. Adjust the flow rate to 20 mL/min per Section 14.
12. Allow the 500 ppb TOC (sucrose) solution to flow through the 5000TOCe Sensor until the readings stabilize, approximately 5 minutes.
13. Record the TOC of the 5000TOCe Sensor for the 500 ppb Solution, TOCbefore.
14. Compute the difference, TOCref - TOCbefore.
15. If the absolute value of the difference for both the 500 ppb is less than the limit, then a TOC calibration is not required.

“As Found” is complete.

7. Flow Rate Calibration

7.1. 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 10. TOC Calibration. If the flow, temperature, and conductivity are to be calibrated, then proceed. Since flow rate is not a reported measurement, “As Found” data is not required. This procedure uses a “volume and time” method for calibrating the flow sensor. Process water is sufficient to use 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.

7.2. Flow Rate Verification Before Calibration
1. Record all data in Worksheet 5: Flow Rate Verification and Calibration.
2. Set up equipment as shown in Figure 3 using Blank Water. Optionally, the flow rate verification and calibration may be done using process water, with the TOC sensor 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. Adjust the 5000TOCe Sensor flow rate to 20 mL/min (4 LEDs) as described in Section 14. Leave “Set Flow Rate” set to “Yes”.
5. Arrange the drain tube at the atmospheric drain such that the water dripping from it may be collected into the volumetric container.
6. Place a dry volumetric container under the drain line to capture the water, and begin the time measurement using the stopwatch. It is important that the water collection and time start be closely matched.
7. Collect a known volume of water with the volumetric container for a specific time, at least 3 minutes.
8. In Section A of Worksheet 5, record the volume of water in mL and the elapsed time in seconds. These two values determine the reference flow rate, Fref.
9. Calculate the reference flow rate and record as Fref 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).

\[
Flow \ Rate \ (\text{mL/min}) = F_{\text{ref}} = \frac{60 \text{ sec/min} \times \text{volume collected (mL)}}{\text{Elapsed Time (sec)}} \quad \text{eq 2}
\]
10. Record the number of LEDs on the front of the 5000TOCe as the Set Flow Rate.
11. If (Fref - 20) is within the limit, then flow rate adjustment is not required. Change “Set Flow Rate” back to “No”. Press Menu to exit and proceed to Section 8. Temperature Calibration. Otherwise, leave the 770MAX in its current state and proceed to the next section.

7.3. Flow Rate Calibration
NOTE: All flow adjustments are to be made using the needle valve located near the inlet of the 5000TOCe Sensor.

1. On the 770MAX, press Menu (exit).
2. Press ▲ or ▼ to select Calibrate. Press Enter (or Page Down).
3. Press ▲ or ▼ to select Sensor. Press Enter (or Page Down).
4. Enter Output Hold Time. Press Enter (or Page Down). “Output Hold Time” is the amount of time that you want the present relay status and analog output value to be held (unchanged) while calibration is performed.

5. Press ▲ or ▼ to select the letter for the measurement of the 5000TOCe Sensor being calibrated. Any of the various sub-sensors within the 5000TOCe Sensor may be calibrated from any other measurement letter. For example, if Measurement C is the TOC from the unit being calibrated, and Measurement D is its conductivity, then any of the sub-sensors within the unit may be calibrated from either Measurement C or D. Press Enter.

6. Press ▲ or ▼ to select 2-point. Press Enter or ▶.

7. Press ▲ or ▼ to select Flow. Press Page Down. The 770MAX should now be displaying the 5000TOCe Sensor flow rate as shown in Figure 4.

8. Adjust the flow rate through the 5000TOCe Sensor to about 24 ± 1 mL/min as indicated on the 770MAX display.

9. Place a dry volumetric container under the drain line to capture the water, and begin the time measurement using the stopwatch. It is important that the water collection and time start be closely matched.

10. Collect a known volume of water with the volumetric container for a measured time, at least 3 minutes.

11. In Section B, record the volume of water in mL and the elapsed time in seconds.

12. Using eq 2, calculate the water flow rate and record as $F_{\text{ref1}}$.

13. Enter the reference flow rate into the 770MAX and press Page-Down.

14. Wait while the first part of the calibration is being performed. 770MAX display will appear as shown in Figure 5.

15. After the first calibration point is complete adjust the flow rate to about 16 ± 1 mL/min.

16. Empty the volumetric container. Repeat steps 9-11.

17. Using eq 2, calculate the water flow rate and record data as $F_{\text{ref2}}$.

18. Enter the reference flow rate into the 770MAX and press Page-Down.

19. Wait while the last part of the calibration is being performed.
20. After the flow sensor calibration is completed, enter the correct date. Press Page-Down.
21. Select YES to save the new calibration data within the 5000TOCe Sensor.

Note: if NO is selected, the calibration data will only be temporarily saved, and the calibration factors will revert back to the previous values should power be lost or the 5000TOCe Sensor patch cable is unplugged.

7.4. Flow Rate Verification After Calibration
1. Repeat steps 1-10 in Section 7.2 to verify the flow sensor is in calibration.
2. Record data in section C instead of section A.

Flow Calibration is complete.

8. Temperature Calibration
This procedure is to be performed on T1 and T2, which correspond to the temperature sensors embedded in the two conductivity sensors.

8.1. T1 Temperature Calibration
2. Turn off the UV lamp (if not already off) by pressing the lamp button so that it’s green LED is off. The lamp shall remain off throughout this procedure.
3. Set up the equipment as shown in Figure 1 using Blank Water.
4. Ensure that selector valve is set to “Cond/Res and Temp Cal’ position.
5. Turn on the pump. Water should now be continuously circulating through the pump, 5000TOCe Sensor, Reference Conductivity Sensor, the water container, and back through the pump.
6. Allow the pump to run until thermal equilibrium is achieved, typically 1/2 hour (less if the lamp has been off).
7. If T1 does not require adjustment, proceed to Section 8.2 T2 Temperature Calibration.
8. If T1 requires adjustment, press Menu (exit).
9. Press ▲ or ▼ to select Calibrate. Press Enter (or Page Down).
10. Press ▲ or ▼ to select Sensor. Press Enter (or Page Down).
11. Enter Output Hold Time. Press Enter (or Page Down). “Output Hold Time” is the amount of time that you want the present relay status and analog output value to be held (unchanged) while calibration is performed.
12. Press ▲ or ▼ to select the letter for the measurement of the 5000TOCe Sensor displaying TOC. Press Enter.
13. Press ▲ or ▼ to select 1-point. Press Enter or ►.
15. Enter the temperature of the Reference Sensor into the 770MAX and press Page-Down. To avoid inaccuracy due to changes in ambient temperature, this value should be entered as soon as practical after reading the Reference Sensor.
16. Wait for calibration to complete.
17. Enter the correct date (if needed). Press Page-Down.
18. Select YES to save the new calibration data within the 5000TOCe Sensor. Press Enter.

Note: if NO is selected, the calibration data will only be temporarily saved, and the calibration factors will revert back to the previous values should power be lost or the 5000TOCe Sensor patch cable is unplugged.

19. Press Menu (exit) to return to measurement mode.
20. Record the temperature of T_{ref} and T_{after} (for T1).
21. Verify that T_{ref} – T_{after} is within limits for T1.
22. Verify that the new computed temperature factor for T1 is within acceptable limits by performing the following steps:
23. Press Menu (exit) to go to MAIN MENU.
24. Press ▲ or ▼ to select Measurement. Press Enter (or Page Down).
25. Press ▲ or ▼ to select the letter for the measurement of the 5000TOCe Sensor displaying temperature.
27. Record the value of Multiplier1 from the 770MAX display.
28. Record the value of Adder1 from the 770MAX display.
29. Verify Multiplier1 and Adder1 are within limits. If they exceed limits, repeat Section 8.1 T1 Temperature Calibration.
30. Return the 770MAX to measurement mode by pressing the Menu (exit) key.

8.2. T2 Temperature Calibration
1. Repeat steps 1-18 in Section 8.1 T1 Temperature Calibration, except select T2 when entering the Sensor Calibration menu.
2. Record the temperature of $T_{ref}$ and $T_{after}$ (for T2).
3. Verify that $T_{ref} – T_{after}$ is within limits for T2.
4. Verify that the new computed temperature factor for T2 is within acceptable limits by performing the following steps:
   5. Press Menu (exit) to go to MAIN MENU.
   6. Press ▲ or ▼ to select Measurement. Press Enter (or Page Down).
   7. Press ▲ or ▼ to select the letter for the measurement of the 5000TOCe Sensor displaying temperature.
9. Press Enter until cursor moves to Multiplier1.
10. Press ▲ or ▼ change value to Multiplier2.
11. Record the value of Multiplier2.
12. Press Enter until cursor moves to Adder1.
13. Press ▲ or ▼ change value to Adder2.
14. Record the value of Adder2 from the 770MAX display.
15. Verify Multiplier2 and Adder2 are within limits. If they exceed limits, repeat Section 8.2 T2 Temperature Calibration.
16. Return the 770MAX to measurement mode by pressing the Menu (exit) key.

Temperature Calibration is complete.

9. Conductivity Calibration
This procedure is to be performed on C1 and C2 which correspond to the two conductivity sensors within the unit.

9.1. C1 Conductivity Calibration
1. Record data in Worksheet 7: Conductivity Sensor Verification and Calibration.
2. Verify the UV lamp is off. Turn off the UV lamp (if not already off) by pressing the lamp button so that it’s green LED is off. The lamp shall remain off throughout this procedure. Wait until stable readings are achieved.
3. Set up the equipment as shown in Figure 1.
4. Ensure that selector valve is set to “Cond/Res and Temp Cal’ position.
5. Continuing with the same Blank Water from the Temperature Calibration, verify that the water quality is <1.5 $\mu$S/cm (>0.67 MΩ-cm) as reported by Reference Conductivity sensor. If the water conductivity has increased above 1.5 $\mu$S/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 $\mu$S/cm with no adverse measurement impact, but we recommend a lower conductivity calibration to emulate process water.
6. If the same bottle of Blank Water is still being used, then continue to the next step. Otherwise, allow the pump to run until complete mixing and thermal equilibrium is achieved, typically 30 minutes.
7. If C1 does not require adjustment, proceed to Section 9.2 C2 Conductivity Calibration.
8. If C1 requires adjustment, press Menu (exit).
9. Press ▲ or ▼ to select Calibrate. Press Enter (or Page Down).
10. Press ▲ or ▼ to select Sensor. Press Enter (or Page Down).
11. Enter Output Hold Time. Press Enter (or Page Down). “Output Hold Time” is the amount of time that you want the present relay status and analog output value to be held (unchanged) while calibration is performed.

12. Press ▲ or ▼ to select the letter for the measurement of the 5000TOCe Sensor displaying TOC. Press Enter.

13. Press ▲ or ▼ to select 1-point. Press Enter or ▶.


15. Enter the conductivity of the Reference Sensor into the 770MAX and press Page-Down. To avoid inaccuracy due to changes in ambient temperature, this value should be entered as soon as practical after reading the Reference Sensor.

16. Wait for calibration to complete.

17. Enter the correct date (if needed). Press Page-Down.

18. Select YES to save the new calibration data within the 5000TOCe Sensor. Press Enter.

Note: if NO is selected, the calibration data will only be temporarily saved, and the calibration factors will revert back to the previous values should power be lost or the 5000TOCe Sensor patch cable is unplugged.

19. Press Menu (exit) to return to measurement mode.

20. Record the conductivity of the C1, Cafter.

21. Compute the % difference according to eq 2.

\[
\text{eq 2} \quad \frac{100 \times (C_{\text{after}} - C_{\text{ref}})}{C_{\text{ref}}}
\]

22. Verify that the % difference is within limits.

23. Verify that the new computed conductivity factor is within acceptable limits by performing the following steps:

24. Press Menu (exit) to go to MAIN MENU.

25. Press ▲ or ▼ to select Measurement. Press Enter (or Page Down).

26. Press ▲ or ▼ to select the letter for the measurement of the 5000TOCe Sensor displaying conductivity.


28. Record the value of Multiplier1 from the 770MAX display.

29. Record the value of Adder1 from the 770MAX display.

30. Verify Multiplier1 and Adder1 are within limits. If they exceed limits, repeat Section 9.1 C1 Conductivity Calibration.

31. Return the 770MAX to measurement mode by pressing the Menu (exit) key.

9.2. C2 Conductivity Calibration

1. Repeat steps 1-18 in Section 9.1 C1 Conductivity Calibration except on Step 13 select C2.

2. Record the conductivity of the C2, Cafter.

3. Compute the % difference according to eq 2.

4. Verify that the % difference is within limits.

5. Verify that the new computed conductivity factor is within acceptable limits by performing the following steps:

6. Press Menu (exit) to go to MAIN MENU.

7. Press ▲ or ▼ to select Measurement. Press Enter (or Page Down).

8. Press ▲ or ▼ to select the letter for the measurement of the 5000TOCe Sensor displaying TOC. Press Enter.


10. Press Enter until cursor moves to Multiplier1.

11. Press ▲ or ▼ change value to Multiplier2.

12. Record the value of Multiplier2.

13. Press Enter until cursor moves to Adder1.

14. Press ▲ or ▼ change value to Adder2.
15. Record the value of Adder2.
16. Verify Multiplier2 and Adder2 are within limits. If they exceed limits, repeat Section 9.2 C2 Conductivity Calibration.
17. Return the 770MAX to measurement mode by pressing the Menu (exit) key.
18. Turn off the pump.

**Conductivity Calibration is now complete.**

10. **TOC Calibration**

Calibration is performed using a solution(s) of known TOC concentration(s). Mettler-Toledo Thornton provides the 5000TOCe Calibration Solution Set (P/N 58 091 529) with a bottle of 500 ppb TOC (sucrose) and 250 ppb TOC (sucrose) calibration solutions for this purpose. If using 770MAX software revision earlier than 5.2 or performing a 1-point slope calibration, then only use the bottle of 500 ppb TOC (sucrose) calibration solution.

10.1. **Set Up TOC Calibration Kit**

1. Record data in Worksheet 8: TOC Calibration and Verification.
2. Set up the equipment as shown in Figure 3.
3. Ensure that selector valve is repositioned to SST/TOC Cal position.
4. Verify the UV lamp is off.
5. Attach the Transfer Cap to the 500 ppb TOC (sucrose) solution bottle.
6. Install the 500 ppb TOC (sucrose) solution bottle on the Bottle Holder bracket.
7. Connect the original drain tube to the Sample Outlet of the 5000TOCe Sensor and run it to atmospheric drain. It is critical not to re-circulate this solution.
8. Turn on the pump. Solution should now be visible at the drain line at the atmospheric drain. Note: the 500 mL volume of the calibration solution will provide more than 20 minutes of operation at the nominal flow rate of 20 mL/min.
9. Verify that UV Lamp is on. The lamp must be on to measure and display TOC.
10. Adjust the flow rate to 20 mL/min per Section 14, if necessary.
11. Allow the 500 ppb TOC (sucrose) solution to flow through the 5000TOCe Sensor until the readings stabilize, approximately 5-10 minutes.

10.2. **TOC Calibration Procedure**

**SECTION A**

For 770MAX software revision 5.2 proceed as follows (for previous software revision go to section B):

1. Press Menu (exit) to go to MAIN MENU.
2. Press ▲ or ▼ to select Calibrate. Press Enter (or Page Down).
3. Press ▲ or ▼ to select Sensor. Press Enter (or Page Down).
4. Enter Output Hold Time. Press Enter (or Page Down). Outputs will reactivate when the timer expires or you may return to this menu at the end of the calibration and set timer to 000.
5. Press ▲ or ▼ to select the letter for the measurement of the 5000TOCe Sensor displaying TOC. Press Enter.
6. Press ▲ or ▼ to select “multi” to use the enhanced calibration features that are resident in the 770MAX software revision v5.2. Press Enter or ▶.
7. Press ▲ or ▼ to select TOC. Press Page Down.
8. Press ▲ or ▼ to set the flow rate to YES.
9. The LED lights will now indicate flow. Adjust the flow rate to 20 mL/min per Section 13, if necessary.
10. Press ▲ or ▼ to set the flow rate to NO. Press Page Down.
11. Allow the 500 ppb TOC solution to flow through the 5000TOCe Sensor until the readings stabilize, approximately 5-10 minutes.
12. Enter the TOC of the Reference Solution, as written on the label or its certificate, into the 770MAX for Cal pt 1.
13. Press page down.
14. Wait for calibration to complete.
15. Turn off pump.
16. Remove the transfer cap from the 500 ppb TOC solution, and install the cap onto the 250 ppb TOC solution and place into the bracket.
17. Turn on the pump.
18. Verify that UV Lamp is on. The lamp must be on to measure and display TOC.
19. Press ▲ or ▼ to set the flow rate to YES.
20. The LED lights will now indicate flow.
21. Adjust the flow rate to 20 mL/min per Section 13, if necessary.
22. Press ▲ or ▼ to set the flow rate to NO. Press Page Down.
23. Allow the 250 ppb TOC solution to flow through the 5000TOC e Sensor until the readings stabilize, approximately 5-10 minutes.
24. Enter the TOC of the Reference Solution, as written on the label or its certificate, into the 770MAX for Cal pt 2.
25. Press page down.
26. Wait for calibration to complete.
27. Enter the correct date (if needed). Press Page-Down.
28. Select YES to save the new calibration data within the 5000TOCe Sensor.

**Note:** if NO is selected, the calibration data will only be temporarily saved, and the calibration factors will revert back to the previous values should power be lost or the 5000TOCe Sensor patch cable is unplugged.

29. Return the 770MAX to measurement mode by pressing the Menu (exit) key twice.
30. Record the TOC as written on the label of the 250 ppb TOC solution or its certificate. This is TOC_{\text{ref}}.
31. Record the measured TOC displayed on the 770MAX, TOC_{\text{after}}.
32. Verify that difference is within acceptable limits in this worksheet.
33. Turn off pump.
34. Remove the transfer cap from the 250 ppb TOC solution, and install the cap onto the 500 ppb TOC solution and place in the bracket.
35. Turn on the pump.
36. Verify that UV Lamp is on. The lamp must be on to measure and display TOC.
37. Adjust the flow rate to 20 mL/min per Section 13, if necessary.
38. Allow the 500 ppb TOC solution to flow through the 5000TOC e Sensor until the readings stabilize, approximately 5-10 minutes.
39. Return the 770MAX to measurement mode by pressing the Menu (exit) key twice.
40. Record the TOC as written on the label of the 500 ppb TOC solution or its certificate. This is TOC_{\text{ref}}.
41. Record the measured TOC displayed on the 770MAX, TOC_{\text{after}}.
42. Verify that difference is within acceptable limits in this worksheet.
43. Enter the lamp life.
44. Press Menu (exit) to go to MAIN MENU.
45. Press ▲ or ▼ to select Measurement. Press Enter (or Page Down).
46. Press ▲ or ▼ to select the letter for the measurement of the 5000TOCe Sensor displaying TOC.
47. Press Page Down.
48. Record the value of TOC Multiplier(s) from the 770MAX display.
49. Record the value of TOC Adder(s) from the 770MAX display.
50. If the difference is not within acceptable limits, repeat Section TOC Calibration Procedure for 770MAX Software revision 5.2.

TOC Calibration is complete. Proceed to section 11.
SECTION B
For 770MAX software revision 5.1 or earlier proceed as follows:

1. Press Menu (exit) to go to MAIN MENU.
2. Press ▲ or ▼ to select Calibrate. Press Enter (or Page Down).
3. Press ▲ or ▼ to select Sensor. Press Enter (or Page Down).
4. Enter Output Hold Time. Press Enter (or Page Down). Outputs will reactivate when the time expires or you may return to this menu at the end of the calibration and set timer to 000.
5. Press ▲ or ▼ to select the letter for the measurement of the 5000TOCe Sensor displaying TOC. Press Enter.
6. Press ▲ or ▼ to select 1-point. Press Enter or ▶.
7. Press ▲ or ▼ to select TOC. Press Page Down.
8. Allow the 500 ppb TOC solution to flow through the 5000TOCe Sensor until the readings stabilize, approximately 5-10 minutes.
9. Enter the TOC of the Reference Solution, as written on the label or its certificate, into the 770MAX for Cal pt 1.
11. Wait for calibration to complete.
12. Record the TOC as written on the label of the 500 ppb TOC solution or its certificate. This is TOC_ref.
13. Record the measured TOC displayed on the 770MAX, TOC_after.
14. Verify that difference is within acceptable limits.
15. Return the 770MAX to measurement mode by pressing the Menu (exit) key twice.
16. Enter the lamp life.
17. Press Menu (exit) to go to MAIN MENU.
18. Press ▲ or ▼ to select Measurement. Press Enter (or Page Down).
19. Press ▲ or ▼ to select the letter for the measurement of the 5000TOCe Sensor displaying TOC.
21. Record the value of TOC Multiplier(s) from the 770MAX display.
22. Record the value of TOC Adder(s) from the 770MAX display.
23. If the difference is not within acceptable limits, repeat Section TOC Calibration Procedure for 770MAX Software revision 5.1

TOC Calibration is complete. Proceed to section 11.

11. Restore to Operation
Caution: Be sure to adjust needle valve to less than 3 turns from closed position before re-installing into process line!
1. Restore the connections to the unit in the same operational configuration prior to calibration.
2. Connect the process water line to the 5000TOCe Sensor sample inlet.
3. Turn on the process water supply to the 5000TOCe Sensor using the shut off valve (recommended in the 5000TOCe Sensor Instruction Manual). Insure the process water supply is turned on by viewing the discharge line at the atmospheric drain.
4. Adjust the flow rate to 20 mL/min per Section 14. Appendix 2 - Setting Sample Flow Rate.
5. Turn on the UV Lamp.
6. 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.
7. 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 Sensor.

12. 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.

1. Connect both provided tubes to the quick-disconnect fittings on the lower right side of the CAL/SST Module.
2. Set the selector valve to the “Cond/Res and Temp Cal” position.
3. 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.
4. 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.
5. 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.
6. Remove and discard the silicon suction tube from the solution bottle interface, and replace the protective cap.
7. 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.
8. Remove the CAL/SST module and mounting bracket from the TOC Sensor. Remove all connected tubing from the CAL/SST module and return all components to the supplied storage case.
Worksheet 1: Unit Under Test Information

Use the table below to record relevant information for the unit under test (UUT).

Note: The reference 770MAX can be the same as the UUT 770MAX if the transmitter has been calibrated according to the 770MAX Multiparameter Analyzer/Transmitter Instruction Manual, part number 84372.

<table>
<thead>
<tr>
<th>770MAX and 5000TOCe Sensor Unit Under Test (UUT) Data</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>770MAX UUT</strong></td>
</tr>
<tr>
<td>Part Number</td>
</tr>
<tr>
<td>Serial Number</td>
</tr>
<tr>
<td>ID Tag (if available)</td>
</tr>
<tr>
<td>Date Last Calibrated</td>
</tr>
<tr>
<td>Date Calibration Due</td>
</tr>
</tbody>
</table>

Name (printed)  
Signature  
Date  
Comments
Worksheet 2: Reference Equipment Information

Use the table below to record relevant information for reference equipment.

Note: The reference 770MAX transmitter can be the same as the UUT 770MAX transmitter, if the transmitter has been calibrated according to the 770MAX Multiparameter Analyzer/Transmitter Instruction Manual, part number 84372.

<table>
<thead>
<tr>
<th>Reference 770MAX Transmitter Data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Part Number</td>
</tr>
<tr>
<td>Serial Number</td>
</tr>
<tr>
<td>Notes</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Reference Conductivity/Temperature Sensor Data (If needed)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Part Number</td>
</tr>
<tr>
<td>Serial Number</td>
</tr>
<tr>
<td>Notes</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Reference TOC Solution Manufacturing Dates/Lot #/Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>500 ppb TOC Solution</td>
</tr>
<tr>
<td>250 ppb TOC Solution</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Other Reference Information and Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glassware</td>
</tr>
<tr>
<td>Other</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Name (printed)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Signature</td>
</tr>
<tr>
<td>Date</td>
</tr>
<tr>
<td>Comments</td>
</tr>
</tbody>
</table>
# Worksheet 3: As Found Data for T1 and C1

- Check if not applicable

## Temperature T1

<table>
<thead>
<tr>
<th>Measurement Type and Sensor #</th>
<th>Reference Temperature $T_{ref}$ (°C)</th>
<th>As Found Temperature $T_{before}$ (°C)</th>
<th>$T_{ref} - T_{before}$ (°C)</th>
<th>Limit (°C)</th>
<th>Adjustment Required (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature T1</td>
<td></td>
<td></td>
<td>±0.5</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

## Temperature Calibration Factors

- Multiplier
- Adder

## Conductivity C1

<table>
<thead>
<tr>
<th>Measurement Type and Sensor #</th>
<th>Reference Conductivity $C_{ref}$ (µS/cm)</th>
<th>As Found Conductivity $C_{before}$ (µS/cm)</th>
<th>$\frac{100 \times (C_{before} - C_{ref})}{C_{ref}}$</th>
<th>Limit (%)</th>
<th>Adjustment Required (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conductivity C1</td>
<td></td>
<td></td>
<td>$\pm 2.0$</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

## Conductivity Calibration Factors

- Multiplier
- Adder

---

Name (printed)  
Signature  
Date  
Comments

---
### Worksheet 4: As Found Data for TOC

☐ Check if not applicable

<table>
<thead>
<tr>
<th>Nominal</th>
<th>Reference TOC&lt;sub&gt;ref&lt;/sub&gt; (ppb)</th>
<th>As Found TOC&lt;sub&gt;before&lt;/sub&gt; (ppb)</th>
<th>TOC&lt;sub&gt;ref&lt;/sub&gt; - TOC&lt;sub&gt;before&lt;/sub&gt; (ppb)</th>
<th>Limit (ppb)</th>
<th>Adjustment Required (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TOC, 500 ppb</td>
<td></td>
<td></td>
<td></td>
<td>±50</td>
<td></td>
</tr>
</tbody>
</table>

**TOC Calibration Factors**

<table>
<thead>
<tr>
<th>Multiplier1</th>
<th>Multiplier2*</th>
<th>Adder1</th>
<th>Adder2*</th>
</tr>
</thead>
</table>

**Lamp Life (hrs)**

<table>
<thead>
<tr>
<th>Name (printed)</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Signature</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Date</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Comments</th>
</tr>
</thead>
</table>

*Note: If software revision 5.2 or higher is being used with "multi" calibration type, a second multiplier/adder will appear.*
### Worksheet 5: Flow Rate Verification and Calibration

- **Check if not applicable**

---

#### Section A: Flow Sensor Verification Before Calibration

<table>
<thead>
<tr>
<th>Calibration point and conditions</th>
<th>Volume (mL)</th>
<th>Elapsed Time (sec)</th>
<th>Reference Flow Rate $F_{ref}$ (mL/min)</th>
<th>Set Flow Rate # LEDs</th>
<th>Limit (mL/min)</th>
<th>Adjustment Required (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As found ~20 mL/min</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>±1.5</td>
<td></td>
</tr>
</tbody>
</table>

#### Section B: Flow Sensor Calibration

<table>
<thead>
<tr>
<th>Calibration point and conditions</th>
<th>Volume (mL)</th>
<th>Elapsed Time (sec)</th>
<th>Reference Flow Rate $F_{ref}$ (mL/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1 ~24 mL/min</td>
<td></td>
<td></td>
<td>$F_{ref1}$</td>
</tr>
<tr>
<td>#2 ~16 mL/min</td>
<td></td>
<td></td>
<td>$F_{ref2}$</td>
</tr>
</tbody>
</table>

#### Section C: Flow Sensor Verification After Calibration

<table>
<thead>
<tr>
<th>Calibration point and conditions</th>
<th>Volume (mL)</th>
<th>Elapsed Time (sec)</th>
<th>Reference Flow Rate $F_{ref}$ (mL/min)</th>
<th>Set Flow Rate # LEDs</th>
<th>Limit (mL/min)</th>
<th>Within Limits (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>~20 mL/min</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>±1.5</td>
<td></td>
</tr>
</tbody>
</table>

---

Name (printed)  
Signature  
Date  
Comments
# Worksheet 6: Temperature Sensor Verification and Calibration

☐ Check if not applicable

<table>
<thead>
<tr>
<th>Measurement Type and Sensor #</th>
<th>Reference Temperature $T_{ref}$ (°C)</th>
<th>Temperature After $T_{after}$ (°C)</th>
<th>$T_{ref} - T_{after}$ (°C)</th>
<th>Limit (°C)</th>
<th>Within Limits (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature T1</td>
<td></td>
<td></td>
<td></td>
<td>±0.5</td>
<td></td>
</tr>
<tr>
<td>Temperature T2</td>
<td></td>
<td></td>
<td></td>
<td>±1.0</td>
<td></td>
</tr>
</tbody>
</table>

## Temperature Calibration Factor Limits

<table>
<thead>
<tr>
<th>Calibration Factors</th>
<th>Factor</th>
<th>Limit</th>
<th>Within Limits (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1 Multiplier1</td>
<td>0.9900 to 1.0100*</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T1 Adder1</td>
<td>-1.000 to +2.500</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T2 Multiplier2</td>
<td>0.9900 to 1.0100*</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T2 Adder2</td>
<td>-1.000 to +2.500</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* The Temperature Multiplier factor typically set to 1.0000 and it is the Temperature Adder that is adjusted during calibration.
# Worksheet 7: Conductivity Sensor Verification and Calibration

☐ Check if not applicable

<table>
<thead>
<tr>
<th>Measurement Type and Sensor #</th>
<th>Reference Conductivity $C_{\text{ref}}$ ($\mu S/cm$)</th>
<th>As Found Conductivity $C_{\text{after}}$ ($\mu S/cm$)</th>
<th>$100 \times \frac{(C_{\text{after}} - C_{\text{ref}})}{C_{\text{ref}}}$</th>
<th>Limit (%)</th>
<th>Within Limits (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conductivity C1</td>
<td></td>
<td></td>
<td></td>
<td>±2.0</td>
<td></td>
</tr>
<tr>
<td>Conductivity C2</td>
<td></td>
<td></td>
<td></td>
<td>±5.0</td>
<td></td>
</tr>
</tbody>
</table>

## Conductivity Calibration Factor Limits

<table>
<thead>
<tr>
<th>Calibration Factors</th>
<th>Factor</th>
<th>Limit</th>
<th>Within Limits (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1 Multiplier1</td>
<td>0.097 – 0.109</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C1 Adder1</td>
<td>-5.000 – 5.000*</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C2 Multiplier2</td>
<td>0.097 – 0.109</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C2 Adder2</td>
<td>-5.000 – 5.000*</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* The Conductivity Adder factor typically set to 0.000 and it is the Conductivity Multiplier that is adjusted during calibration.

<table>
<thead>
<tr>
<th>Name (printed)</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Signature</td>
<td></td>
</tr>
<tr>
<td>Date</td>
<td></td>
</tr>
<tr>
<td>Comments</td>
<td></td>
</tr>
</tbody>
</table>

---

Page 21
Worksheet 8: TOC Calibration and Verification

☐ Check if not applicable

<table>
<thead>
<tr>
<th>Reference TOC&lt;sub&gt;ref&lt;/sub&gt; (ppb)</th>
<th>TOC&lt;sub&gt;after&lt;/sub&gt; (ppb)</th>
<th>TOC&lt;sub&gt;ref&lt;/sub&gt; – TOC&lt;sub&gt;after&lt;/sub&gt; (ppb)</th>
<th>Limit (ppb)</th>
<th>Within Limits? (Y or N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TOC, 500 ppb</td>
<td></td>
<td></td>
<td>±50</td>
<td></td>
</tr>
<tr>
<td>TOC, 250 ppb*</td>
<td></td>
<td></td>
<td>±25</td>
<td></td>
</tr>
</tbody>
</table>

TOC Calibration Factors

<table>
<thead>
<tr>
<th>Multiplier1</th>
<th>Multiplier2*</th>
<th>Adder1</th>
<th>Adder2*</th>
</tr>
</thead>
</table>

Lamp Life (hrs)

Name (printed)

Signature

Date

Comments

*Note: If software revision 5.2 or later is used with "multi" calibration type, a second multiplier/adder will appear.
13. Appendix 1: Calibration Process

The Thornton 5000TOCe Total Organic Carbon Sensor and 770MAX transmitter measure the amount of organic carbon in high purity waters by oxidizing organic carbon to CO₂ with appropriate UV radiation. The resulting increase between two temperature-compensated conductivity measurements of the sample flow stream at points before and after the oxidation process is used to calculate the amount of organic carbon present. The complete calibration of the 5000TOCe Sensor consists of calibration of the following.

- flow rate sensor measurement
- two temperature sensor measurements
- two conductivity sensor measurements
- TOC measurement

13.1. Scope

This document provides procedures to calibrate the Thornton 5000TOCe Total Organic Carbon Sensor (PN's 58 036 001, 58 036 002, 58 036 003, 58 036 004 and related part numbers). The 5000TOCe Sensor is used with the Thornton 770MAX transmitter. Calibration of the 770MAX transmitter is described in separate documentation.

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 pharmacopeial requirements.

13.2. 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 instrumentation, but the following is recommended by Thornton as a minimum service cycle.

<table>
<thead>
<tr>
<th>Table 1. Scheduled Calibration, Lamp change, and SST Service</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Maintenance</strong></td>
</tr>
<tr>
<td>Replace lamp</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>TOC Calibration</td>
</tr>
<tr>
<td>Complete Calibration (includes flow, TOC, conductivity, temperature)</td>
</tr>
<tr>
<td>System Suitability Test (SST)</td>
</tr>
</tbody>
</table>

*See USP <643> and EP 2.2.44

13.3. 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. Fill out Worksheet 1: Unit Under Test Information.
5. Fill out Worksheet 2: Reference Equipment Information.
6. If TOC only calibration is to be performed, go to step 8 if as found data is required, or step 16 if as found data is not required.

7. Fill out Worksheet 3: As Found Data for T1 and C1. If required, get As Found T1 and C1 data. Get As Found T1 and C1 Calibrations Factors.

8. Fill out Worksheet 4: As Found Data for TOC. If required, get As Found TOC data. Get As Found TOC Calibrations Factors.

9. If required, change lamp.

10. If TOC only calibration is to be performed, go to step 16.

11. Fill out Worksheet 5: Flow Rate Verification and Calibration. Perform Flow Rate Verification. If acceptable, to go to step 12. Otherwise, perform Flow Rate Calibration, then perform Flow Rate Verification.

12. Fill out Worksheet 6: Temperature Sensor Verification and Calibration. Perform T1 Calibration. This step may be skipped if the T1 As Found was acceptable. Verify T1 Calibration factors are acceptable.


14. Fill out Worksheet 7: Conductivity Sensor Verification and Calibration. Perform C1 Calibration. This step may be skipped if the C1 As Found was acceptable. Verify C1 Calibration factors are acceptable.


16. Fill out Worksheet 8: TOC Calibration and Verification. If 770MAX software is v5.2 or later, perform TOC Calibration with 250 ppb and 500 ppb TOC solutions. Get As Found TOC Calibrations Factors. This step may be skipped if the TOC As Found was acceptable.

17. Restore unit to operation or perform SST, as required.

### 14. Appendix 2 - Setting Sample Flow Rate

At various times during the calibration of TOC, the flow rate needs to be set using this procedure. This is an informational section. This section is only required as needed.

#### 14.1. Flow Rate Adjustment Procedure

1. Press Menu (exit) to go to MAIN MENU.
2. Scroll to the Measurements menu and press Enter.
3. Select the measurement letter (A, B, C...) for the TOC measurement of interest.

4. Press Page Down (twice) until the “Press 5 for TOC menu” appears.

5. Press enter to activate Set Flow Rate option. Change this setting to Yes. This immediately initiates the Set Flow Rate mode at the sensor. It will stay in this mode until the user changes the “Set Flow Rate: Yes” back to “Set Flow Rate: No”. If the user does not set the flow rate once the Set Flow Rate mode has been initiated, the 770MAX will time out, at which time the mode automatically goes back to “Set Flow Rate: No” and the instrument returns to the measurement display screen.

6. See Figure 6. The flow rate is set by adjusting the needle on the 5000TOCe Sensor. NOTE: Clockwise (CW) to decrease flow, counterclockwise (CCW) to increase flow.

![Figure 6. Needle Valve Location](image)

7. In this mode, the LEDs on the front cover of the 5000TOCe Sensor act as a bar-graph indicator as described according to Figure 7.

8. Adjust the inlet Needle Valve until three or four LEDs light on the 770MAX for at least 30 seconds.

9. Go to the 770MAX and change “Set Flow Rate: Yes” to “Set Flow Rate: No”. The flow rate setting operation is now complete.

10. Press Exit Menu twice to return to measurement mode.

**14.2. Flow Rate Indication**

The flow rate value is indicated by the four LEDs (lights) on the front of the 5000TOCe Sensor.

<table>
<thead>
<tr>
<th>LED Status</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fault</td>
<td>LED illuminates (●)</td>
</tr>
<tr>
<td>Error</td>
<td>LED illuminates (●)</td>
</tr>
<tr>
<td>Sensor Status</td>
<td>LED illuminates (●)</td>
</tr>
<tr>
<td>UV Lamp On</td>
<td>LED illuminates (●)</td>
</tr>
</tbody>
</table>

● = LED illuminated  ○ = LED not illuminated

When the flow rate is at 20 mL/min (±2.5%), all four LEDs will be on. This is acceptable. When the flow rate is at 20 mL/min (±7.5%), the top three LEDs OR the bottom three LEDs will be on. This is acceptable. If the flow rate is too high, the top two LEDs will be on. Turn the adjustment knob clockwise. If the flow rate is too low, the bottom two LEDs will be on. Turn the adjustment knob counterclockwise. If the flow rate is far from 20 mL/min, only the top (Fault) LED or the bottom (UV Lamp) LED will be on.
As the flow rate approaches the an acceptable value, more LEDs will turn on. For example, if the flow rate is too high, as the adjustment knob is turned clockwise, first the Fault LED will be on, then the Fault and Error LEDs will be on, then the Fault, Error and Sensor Status LEDs will be on and finally all four LEDs will be on when the flow rate is 20 mL/min (±2.5%).

<table>
<thead>
<tr>
<th></th>
<th>Very LOW</th>
<th>LOW</th>
<th>NEAR SET</th>
<th>SET</th>
<th>NEAR SET</th>
<th>HIGH</th>
<th>Very HIGH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fault</td>
<td>○</td>
<td>○</td>
<td>○</td>
<td>●</td>
<td>●</td>
<td>●</td>
<td>●</td>
</tr>
<tr>
<td>Error</td>
<td>○</td>
<td>○</td>
<td>●</td>
<td>●</td>
<td>●</td>
<td>●</td>
<td>○</td>
</tr>
<tr>
<td>Sensor Status</td>
<td>○</td>
<td>●</td>
<td>●</td>
<td>●</td>
<td>●</td>
<td>○</td>
<td>○</td>
</tr>
<tr>
<td>UV Lamp On</td>
<td>●</td>
<td>●</td>
<td>●</td>
<td>●</td>
<td>●</td>
<td>○</td>
<td>○</td>
</tr>
</tbody>
</table>

● = LED illuminated  ○ = LED not illuminated

**Figure 7. LED Indicator for Flow rate**
15. Appendix 3 – Using SST and Calibration kit 58 0791 534

15.1. Installing the Thornton 5000TOCe Calibration Test Kit Brackets

1. Refer to Figure 8. Install Main Bracket A on top of the 5000TOCe Sensor.
2. For conductivity/resistivity calibration, install the Reference Sensor Holder over the Main Bracket A. If performing an SST or TOC calibration only, the Sensor Holder may be omitted.
3. Attach Main Bracket B onto the Reference Sensor Holder and Main Bracket A with 2 thumbscrews as shown.

![Figure 8](image_url)

4. Refer to Figure 9. Align the slotted hole in the pump assembly over the left thumb screw, and then slide the pump assembly to the left to hold in position.

![Figure 9](image_url)

5. Align the slotted hole in the Bottle Holder Bracket over the right thumb screw, and then slide the bracket to the left to hold in position.

6. For conductivity/resistivity calibration, install the Reference Sensor Assembly onto the Reference Sensor Holder. For conductivity/resistivity or temperature calibration, the Reference Sensor Assembly may be omitted.

7. Secure the entire assembly by tightening the thumb screws. The entire bracket assembly is now complete and should look like Figure 10.
15.2. Installing 5000TOCe Calibration Kit Tubing for Conductivity and Temperature Calibration

1. Refer to Figure 11. Clean the Transfer Cap assembly using de-ionized or process water (water to be tested).
2. Take one of the Blank Water bottles and shake it to homogenize the contents.
3. Remove the Blank Water bottle cap and replace it with the cleaned Transfer Cap.
4. Slide the Blank Water bottle and Transfer Cap onto the assembled Bottle Holder Bracket.

5. See Figure 12. Clean Tube A using de-ionized or process water (water to be tested).
6. Insert one end of Tube A into the Transfer Cap until it comes to approximately 1 cm from the bottom of the Blank Water bottle.
7. Place the other end of Tube A into the "IN" port of the Pump.
8. Connect Tube B from the "OUT" port of the Pump to the input of the Reference Sensor Assembly.
9. Connect one end of Tube C to the output of the Reference Sensor Assembly.
10. Connect the other end of Tube C to the inlet filter on the Sample Inlet of the 5000TOCe Sensor.
11. Clean Tube D using de-ionized or process water (water to be tested).
12. Connect one end of Tube D to the Sample Outlet of the 5000TOCe Sensor. Do not connect the other end of Tube D.
13. Place the other end of Tube D over a container that can hold at least 100 mL of water. Do not allow the end of the tube to become contaminated.
14. Verify the pump switch is in the OFF position.
15. Connect the Universal Power Supply to the pump and to 100–240 VAC 50-60 Hz power. The kit includes a North American AC power cord. Replace or adapt the power cord as necessary and in accordance with local regulations.

16. Turn the pump power switch to ON and operate pump for approximately 2 minutes to remove any residual water from the pump and the 5000TOCe Sensor.

17. Turn the pump OFF.

18. Once the residual water has been successfully purged, insert about 4 inches (10 cm) of the exposed end of Tube D into the Transfer Cap. The assembly is now complete and should now appear as Figure 12.

![Figure 12](image)

NOTE: When the pump is first put into service, or after extended storage, it may need to be primed by injecting reagent water into the inlet fitting with the provided syringe. Place the pump in its normal vertical position with the inlet tube from the bottle removed from the inlet fitting and the outlet tube placed to discharge to a nearby drain or into a container. Draw reagent or clean process water into the syringe, place the tip of the 30 mL syringe into the pump inlet fitting, turn on the pump and push about 10 mL of water into the fitting or until water appears at the outlet fitting. Excess water will run from the inlet fitting. Turn off the pump. Reattach the tube from the bottle to the pump inlet fitting, and proceed. Reagent water, known to be low TOC, is ideal to be utilized for flushing the 5000TOCe Sensor.

15.3. Final Installation of the 5000TOCe Sensor Calibration Kit

1. Connect the Reference Conductivity sensor smart connector to the 770MAX patch cable (1015-79).

2. Connect the other end of the 770MAX patch cable to an unused calibrated channel on the 770MAX transmitter. If no channel is available, then another calibrated 770MAX transmitter should be utilized.

3. Set up a measurement to display the Reference Sensor temperature and temperature compensated conductivity. For example, Measurement B could be set to display conductivity and Measurement C could be set to display temperature (see suggested 770MAX configuration in Section 4.2 770MAX Configuration).

4. Turn on the pump. The setup should now be such that water continuously circulates through the Blank Water bottle and the 5000TOCe Sensor.

5. Adjust the flow rate to its maximum rate. This may be done by adjusting the needle valve knob fully counterclockwise until it stops.

6. The system is now ready for temperature calibration. The pump may be left on if you are proceeding to the next section.
15.4. TOC Calibration Apparatus Installation

1. Carefully place the assembled bracket, with pump, over the 5000TOCe Sensor. Slide the assembly to the left slightly, leaving the ‘UV Lamp On’ key accessible. Figure 3 shows the proper location and orientation of the System Suitability Apparatus.

![Figure 13. TOC Calibration Apparatus (shown with solution bottle installed)](image)

2. Verify the pump switch is in the off position.
3. Connect the Universal Power Supply to the pump and to 100–240 VAC 50-60 Hz power. The kit includes a North American AC power cord. Replace or adapt the power cord as necessary and in accordance with local regulations.
4. Clean the Transfer Cap and the Open Top Cap using deionized or process water (water to be tested). These parts are identified in the parts description for the Pump Kit.
5. Assemble as shown in Figure 13. Without touching the lower 7 ½" (20 cm) of the tubing, carefully insert the bottle tube (plain tube without nut and ferrule) into the thumb fitting on the Transfer Cap fitting with 6 ¼" (16 cm) extending from bottom of Transfer Cap. Do not allow the lower end of the bottle tube to become contaminated. If the tube will not slide through the thumb fitting, inspect the ferrule immediately below it.
6. Connect the other end of the bottle tube to the “Inlet” fitting on the pump.
7. Connect the plain end of the Sensor Tube (tube with nut and ferrule) to the “Outlet” fitting on the pump.
8. Remove the solid cap from the Calibration Standard solution bottle and install the transfer cap. The end of the bottle tube should be near the bottom of the Reagent Water container. Place the appropriate Calibration Standard Solution bottle into the holder notch on the bracket.

**IMPORTANT NOTE:**
When the pump is first put into service, or after extended storage, it may need to be primed by injecting reagent water into the inlet fitting with the syringe provided. Place the pump in its normal vertical position with the inlet tube from the bottle removed from the inlet fitting and the outlet tube placed to discharge into a container. Draw reagent water into the syringe, place the tip of the 30cc syringe into the pump inlet fitting, turn on the pump and push about 10 ml of water into the fitting or until water appears at the outlet fitting. Excess water will run from the inlet fitting. Turn off the pump. Reattach the tube from the bottle to the pump inlet fitting and proceed.
This Page is intentionally Blank