

Sievers^{*} CheckPoint Pharma and CheckPoint^e Portable/Online TOC Sensors

standard operating protocols: installation, operation and performance qualification

1.06 firmware or later



<u>evers CheckPoin</u>

Portable /Online TOC Senso

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For a copy of the Declaration of Conformity and ISO 9001 Registration: Access the Sievers* website (Library > Declarations of Conformity) and (Company > ISO 9001 Certification).

Electronic versions of the Sensor's *Operation and Maintenance Manual* and *Standard Operating Protocols* are available from the Sievers website (Library > Manuals).

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REVISION HISTORY

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TRADEMARKS AND PATENTS

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US 6723565 and any of its non-US counterparts US 8101417 and any of its non-US counterparts US 8003048 and any of its non-US counterparts US 9116099 and any of its non-US counterparts US 9074967 and any of its non-US counterparts and other patents pending

CHECKPOINT STANDARD OPERATING PROTOCOLS

OVERVIEW

The CheckPoint[®] Standard Operating Protocols (SOPs) package includes protocols, worksheets, and other information to allow users to easily qualify a CheckPoint Pharma or CheckPoint^e Portable/On-Line TOC Sensor for use in their processes. The package is intended to primarily assist customers in the pharmaceutical industry in developing the documents and testing to provide assurance that the Sensor operates to specification and meets installation, operational, and performance requirements of their applications. The customer may modify these protocols to conform to company-specific or other industry-specific requirements in accordance with the License Agreement. The components of the SOP are listed in the Table of Contents.

Throughout this document, references to CheckPoint apply to both the CheckPoint Pharma and CheckPoint^e Portable/On-Line TOC Sensors unless otherwise specified.

ISO 9001 REGISTRATION

Veolia is registered to ISO 9001. For a copy of the certificate, please visit our website.

LICENSE AGREEMENT

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Grant of License

Veolia grants the user the right to use this package to aid the user in verification of one CheckPoint Portable/On-Line TOC Sensor. This package is linked to that Sensor and may not be used to support additional instrumentation.

Use Permissions

User is permitted to:

- Copy worksheets and protocols as needed to qualify the Sensor for any application.
- Modify any documents. A PDF version of the SOP is available for download from our website to provide a vehicle for modification of protocols and worksheets. Any documents so modified must clearly state the Veolia document used as the source and clearly state that the original document has been modified.

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RECOMMENDED ORDER OF QUALIFICATION

- **1.** Installation Qualification
 - Sensor and Firmware Installation Protocol and Checklist
 - (Optional) 4-20 mA Output Operation Verification Protocol and Worksheet
- **2.** Operation Qualification
 - Flow Rate Calibration Protocol and Worksheet
 - Conductivity Calibration/Verification Protocol and Worksheet
 - TOC Calibration/Verification Protocol and Worksheet
 - System Suitability (Response Efficiency) Protocol and Worksheet
- 3. Performance Qualification

(Optional) Linearity Verification Protocol and Worksheet

Limit of Detection and Limit of Quantitation Extrapolation Verification Protocol and Worksheet (requires running the Linearity Verification protocol)

or

Limit of Detection and Limit of Quantitation Repetitive Measurement Verification Protocol and Worksheet

SOP Checklist for CheckPoint TOC Sensor

Company Name_____

Analyst Name _____

Sensor Serial Number_____

Date_____

Firmware Version_____

Protocol, Checklist, or Worksheet Name	Result: Pass/Fail, Complete/ Incomplete, or N/A	Date	Initial
Sensor and Firmware Installation Protocol and Checklist			
(Optional) 4-20 mA Output Operation Verification Protocol and Worksheet			
Flow Rate Calibration Protocol and Worksheet			
(Optional) Conductivity Calibration/Verification Protocol and Worksheet			
TOC Calibration/Verification Protocol and Worksheet			
System Suitability (Response Efficiency) Protocol and Worksheet			
(Optional) Linearity Verification Protocol and Worksheet			
LOD and LOQ Extrapolation Protocol and Worksheet (requires running the Linearity Verification protocol)			
or			
LOD and LOQ Repetitive Measurement Protocol and Worksheet			

Performed By:	Date:
Reviewed By:	Date:
Verified By:	Date:

INSTALLATION QUALIFICATION PROTOCOLS

- Sensor and Firmware Installation Protocol and Checklist
- (Optional) 4-20 mA Output Operation Verification Protocol and Worksheet

Sensor and Firmware Installation Protocol for CheckPoint Portable/On-Line TOC Sensor

- **1. Purpose:** To verify the installation of a CheckPoint Portable/On-Line TOC Sensor.
- 2. **Scope:** This protocol is intended to document installation of a CheckPoint Portable/On-Line TOC Sensor, for verification purposes. A checklist is provided to assist with installation. Specific installation instructions and illustrations are detailed in the Sensor's *Operation and Maintenance Manual*.

3. Materials:

- 3.1. CheckPoint Portable/On-Line TOC Sensor shipping box and contents
- 3.2. Sensor and Firmware Installation Checklist for CheckPoint Portable/On-Line TOC Sensor Worksheet (see page 14)
- 3.3. The Sensor's Operation and Maintenance Manual
- 3.4. CheckPoint Pharma and CheckPoint^e SOP Package
- 4. **Definitions:** None

5. Procedures:

- 5.1. Unpack the box and verify that all of the following items have been received. Enter Yes, No, or NA and Initial and Date in the corresponding column of the Installation Checklist.
 - 5.1.1. CheckPoint Portable/On-Line TOC Sensor.
 - 5.1.2. The Sensor's Operation and Maintenance Manual.
 - 5.1.3. CheckPoint Standard Operating Protocols.
 - 5.1.4. Certificate of Calibration.
 - 5.1.5. Accessories. Verify contents match list described in the "Installation" chapter in the Sensor's *Operation and Maintenance Manual*.
 - 5.1.6. (Optional) Vial Sampling Kit.
 - 5.1.7. (Optional) Low-pressure sampling kit.
 - 5.1.8. (Optional) Sample inline filter.
- 5.2. On the Worksheet, record the Sensor's serial number (found on the left side of the Sensor) and the date of receipt. If you have printed the Sensor's *Operation and Maintenance Manual*, complete the "Identification Records" section found on page 3.
- 5.3. Confirm that the installation site meets the Sensor's environmental requirements. If you will be mounting the Sensor, install the mounting bracket, as described in the "Installation" chapter of the Sensor's *Operation and Maintenance Manual* in the section called "Step 1: Install the Mounting Bracket."
- 5.4. Install the power option.

- 5.4.1. The Sensor can be used with a power cord or power conduit. Connect the power option, as described in the section called "Step 2: Install the Power Option" in the Sensor's *Operation and Maintenance Manual*.
- 5.5. Install the network and I/O cabling (optional)
 - 5.5.1. If you will be connecting CheckPoint to your network or directly to a computer, connect an Ethernet cable, as described in the section called "Step 3: Install the I/O Cabling" in the Sensor's *Operation and Maintenance Manual*.
 - 5.5.2. If the Sensor is configured with an I/O board, connect the 4-20 mA outputs, binary input, and alarm wiring as described in the section called "Step 3: Install the I/O Cabling" in the Sensor's *Operation and Maintenance Manual*.
- 5.6. Connect the sample inlet and waste outlet. Follow the instructions in the "Installation" chapter of the *Operation and Maintenance Manual* in the section called "Step 4: Install the Sample Inlet and Waste Outlet."
- 5.7. Enable the flow of sample water to the Sensor.
- 5.8. Power the Sensor on.
- 5.9. Set the date and time.
 - 5.9.1. Press the 🖶 button and press **F3** to select the **Setup** screen.
 - 5.9.2. Use the scroll bars to highlight **Clock** and press the *→* button.
 - 5.9.3. Press **F1** and use the scroll buttons to specify the date. Press to set the date.
 - 5.9.4. Press **F2** and use the scroll buttons to specify the time. Press to set the time.
 - 5.9.5. Press **F4** to return to the previous screen.
- 5.10. Record the firmware version number.
 - 5.10.1. Press the *L* button until the **System Info** screen displays.
 - 5.10.2. The **Ver** field lists the firmware version number. Record this number in the field at the top of the Worksheet.
- 5.11. Rinse the Sensor down. Press the button and let the Sensor run for 6 hours.

Sensor and Firmware Installation Checklist for CheckPoint Portable/On-Line TOC Sensor

Company Name	Date
Analyst Name	Firmware Version
Sensor Serial Number	Date of Receipt

Protocol Step No.	Description	Yes/No or N/A Initial & Date
5.1.1	CheckPoint Portable/On-Line TOC Sensor received.	
5.1.2	Sensor Operation and Maintenance Manual CD-ROM received.	
5.1.3	CheckPoint Standard Operating Protocols received.	
5.1.4	Certificate of Calibration received.	
5.1.5	(Optional) All accessories received.	
5.2	Sensor serial number and date of receipt recorded on Worksheet.	
5.3	Installation site satisfies Sensor environmental requirements, and Sensor mounted (optional).	
5.4.1	Power source installed.	
5.5.1	(Optional) Ethernet cable connected.	
5.5.2	(Optional) 4-20 mA outputs, binary input, and alarms connected.	
5.6	Sample inlet and waste outlet installed.	
5.7	Flow of sample water to Sensor enabled.	
5.8	Sensor powered on.	
5.9	Date and time set.	
5.10	Firmware version number recorded on Worksheet.	

Protocol Step No.	Description	Yes/No or N/A Initial & Date
5.11	Sensor has rinsed down for 6 hours.	

Performed By:	Date:
Reviewed By:	Date:
Verified By:	Date:

4-20 mA Output Operation Verification Protocol (Optional)

- **1. Purpose:** To verify the operation of 4-20 mA outputs on a CheckPoint Portable/On-Line TOC Sensor.
- 2. **Scope:** This protocol applies to CheckPoint Portable/On-Line TOC Sensors equipped with the optional I/O board. This protocol assumes familiarity with the functionality of 4-20 mA output and supplemental tools, such as a digital multimeter. Note that the accuracy of the multimeter can affect the results of this protocol. This protocol is an optional part of the Sensor's operation verification.

3. Materials:

3.1. 4-20 mA Output Verification Worksheet (see page 18)

3.2. Digital multimeter, or other device capable of measuring current from 0-20 mA

4. Definitions: None

5. Procedure:

- 5.1. Turn off power to the Sensor.
- 5.2. Open the I/O board cover by loosening the thumbscrew.
- 5.3. Connect wiring from a 4-20 mA output to the digital multimeter. For instructions on connecting wiring to the 4-20 mA outputs, consult the "Installation" chapter of the Sensor's *Operation and Maintenance Manual*. Run the multimeter wires through the pass-through port on the I/O cover, so that the cover can be closed during the procedure.
- 5.4. Restore power to the Sensor.
- 5.5. Press the 🖶 button.
- 5.6. Confirm the analog output is enabled.
 - 5.6.1. Press F2 to select the I/O screen.
 - 5.6.2. Use the button to highlight **Analog outputs** and press the *use* button.
 - 5.6.3. Depending on which output you wired in 5.3, press F1 to select Analog output 1, press F2 to select Analog output 2, or press F3 to select Analog output 3.
 - 5.6.4. Press the *L* button to toggle the Analog output on and off. Ensure that the Analog output is set to ON.
- 5.7. Press and **F4** to select the **Maintenance** screen.
- 5.8. Press **F1** to select the **Diagnostics** screen.
- 5.9. Use the volume button to highlight **4-20 mA outputs** and press the *-* button.

- 5.10. Depending on which output you wired in 5.3, press **F1** to test Analog output 1, press **F2** to test Analog output 2, or press **F3** to test Analog output 3.
- 5.11. Press **F1** to initiate an output.
- 5.12. Toggle the value between 4 or 20 mA by pressing the **F1** button.
- 5.13. The Sensor will send current to the analog output and the multimeter will reflect the reading.
- 5.14. Acceptance criterion: the difference between the multimeter reading and the value selected in Step 5.12 should be $\pm 3\%$.
- 5.15. Press **F4** three times and the 🖾 button multiple times until you return to your chosen main screen.
- 5.16. Consult the "Basic Operation and Menu Options" chapter of the Sensor's *Operation and Maintenance Manual* to configure your menu options.

4-20 mA Output Operation Verification Worksheet

Company Name	Date
Analyst Name	Firmware Version
Sensor Serial Number	
Analog output used (1, 2, or 3)	
Current selected (4 or 20 mA)	
Current displayed by multimeter	
% Different between expected and measured current	
Acceptance criterion: % Difference $\pm 3\%$	□ Fail
	- .
Performed By:	Date:
Reviewed By:	Date:
Verified By:	Date:

OPERATIONAL QUALIFICATION PROTOCOLS

- Flow Rate Calibration Protocol and Worksheet
- Conductivity Calibration/Verification Protocol and Worksheet
- TOC Calibration/Verification Protocol and Worksheet
- System Suitability (Response Efficiency) Protocol and Worksheet

Sample Flow Rate Calibration Protocol

- **1. Purpose:** To calibrate and then verify the sample flow rate in a CheckPoint Portable/On-Line TOC Sensor.
- 2. **Scope:** This protocol applies to CheckPoint Portable/On-Line TOC Sensors. This protocol is an optional part of the Sensor's operation verification. The flow rate calibration should be performed whenever the pump head is replaced. This procedure assumes CheckPoint is installed on a pressurized water system.

3. Materials:

3.1. Flow Rate Calibration Worksheet

- 3.2. Flow rate calibration kit components from the Sensor's accessories kit (tubing and 10-mL graduated cylinder)
- 4. **Definitions:** None

5. Procedure:

- 5.1. If the Sensor is taking measurements, press the **b**utton and **F1**.
- 5.2. Attach a USB flash memory drive to the Sensor's USB port and then press the source button to save the current system settings, in the event they need to be reloaded or referred to in the future.
- 5.3. Shut off the sample supply line. Disconnect the tubing from the Sensor's **Waste** port. If your Sensor has a **Bypass** port, connect a plug to the **Bypass** port. (Remove any other lengths of tubing and fittings from the waste line.)
- 5.4. Connect the short length of waste tubing from the accessories kit to the **Waste** port.
- 5.5. Place the other end of the tubing in the 10 mL graduated cylinder from the accessories kit.
- 5.6. Press the 🖶 button.
- 5.7. Press **F1** to select the **Mode** screen.
- 5.8. Press **F1** to select **Calibration**.
- 5.9. Press **F3** to select **Flow Rate**.
- 5.10. Confirm that the Sensor is connected to a water source and press **F1** to prepare the Sensor for flow rate calibration. The Sensor fills the pump tubing with water for approximately 20 seconds. Empty any water that has collected in the graduated cylinder before proceeding.

If the waste line has not filled in the allotted time, press F4 to cancel. Repeat from Step 5.9 until the fluid path has been filled.

- 5.11. Press **F1** to start the flow rate calibration process. The pump will operate at twice normal speed for 5 minutes.
- 5.12. After 5 minutes, measure the volume of water in the graduated cylinder to the nearest 0.1 mL. There should be close to 5 mL. Enter this volume as prompted by the Sensor and also record the volume on Line 2 of the Worksheet. Press **F1** to temporarily apply new settings.

- 5.13. To verify the new flow rate calibration, empty the graduated cylinder and replace the flow rate calibration drain tubing in the cylinder. Press **F1** to continue. The sensor again pumps water for 5 minutes.
- 5.14. After 5 minutes, measure the volume of water in the graduated cylinder to the nearest 0.1 mL. Record the volume on Line 3 of the Worksheet.
- 5.15. Apply the calibration if the following acceptance criterion is satisfied:
- Acceptance criterion: The volume collected during the second 5-minute test is between 4.8 and 5.2 mL.
- If this condition is satisfied, press **F1** to apply the new flow rate calibration and continue.
- If this condition is not satisfied, you may need to perform the sample flow rate calibration procedure again. However, first check that the sample tubing has been installed correctly. Also, look for any leaks inside the Sensor and correct them. Consult the chapter called "Troubleshooting" in the Sensor's *Operation and Maintenance Manual* to determine if there is another problem with the Sensor.
- 5.16. After flow rate calibration is complete, disconnect the tubing from the **Waste** port and reconnect the standard waste tubing and bypass lines.

Sample Flow Rate Calibration Worksheet

Company Name		Date
Analyst Name	Firmware Version	
Sensor Serial Number		_
Sample pump head replace	d before sample flow rate	calibration (Y/N): (Line 1)
Volume collected for first 5	minute period: (Line	e 2)
Volume collected for second	d 5 minute period: ((Line 3)
Flow rate calibration accept	ance criterion:	
• The volume of	collected during the secon	nd 5-minute test is between 4.8 and 5.2 ml
Calibration Action:	□ Applied	□ Canceled
Performed By:		Date:
Reviewed By:		Date:
Validated By:		Date:

Conductivity Calibration and Verification Protocol

- **1. Purpose:** To perform a conductivity calibration for the CheckPoint Portable/On-Line TOC Sensor.
- 2. **Scope:** This procedure applies to all CheckPoint Portable/On-Line TOC Sensors. Note that a Vial Sampling Kit is required to perform the protocol.

Conductivity calibration should be performed after replacing the pump head if the Sensor is used for conductivity or resistivity measurements. The conductivity calibration is not necessary for low-level applications where conductivity or resistivity values are not being used.

Note: Use only Sievers standards purchased from Veolia.

The analyst performing this protocol should be familiar with the terminology and operation of the Sensor.

Note: CheckPoint reports temperature-compensated conductivity values while running this protocol, regardless of the Units of Measure configuration in the **Setup** menu.

3. Materials:

- 3.1. CheckPoint Portable/On-Line TOC Sensor
- 3.2. Vial Sampling Kit
- 3.3. Conductivity Calibration Worksheet (see page 25)
- 3.4. Conductivity calibration standards from Veolia, comprising:
- 1 vial of 25 µS/cm Conductivity Standard (as HCI)

All standards should be warmed to room temperature prior to use.

Shake standards vials for 10-30 seconds prior to use.

4. Definitions:

- 4.1. DI deionized
- 4.2. TOC total organic carbon

5. Procedure:

- 5.1. If the Sensor is taking measurements, press the \blacksquare button.
- 5.2. Make sure the Vial Sampling Kit is connected, per the instructions in the Sensor's *Operation and Maintenance Manual*.
- 5.3. Attach a USB flash memory drive to the Sensor's USB port and then press the button to save the current system settings, in the event they need to be re-loaded or referenced in the future.
- 5.4. Press the 🖶 button.
- 5.5. Press **F1** to select the **Mode** screen.
- 5.6. Press **F1** to select **Calibration**.

- 5.7. Press F2 to select Conductivity.
- 5.8. Press F1 to select Conductivity Calibration.
- 5.9. Insert the 25 μ S/cm HCl standard into the vial port.
- 5.10. Press **F1** to start conductivity calibration. Sample is flushed through the Sensor and measurements on the HCl solution begin.

When measurements are complete, the Sensor displays the mean measured and adjusted temperature-corrected conductivity values for both cells and C1 expected conductivity. Press **F1** to accept the calibration or **F4** to cancel.

If a printer has been installed, the CheckPoint will print the results of each measurement upon completion.

There are no formal pass/fail criteria for the conductivity calibration, but Veolia recommends that the calibration be applied if the average values are between 24-26 μ S/cm and the RSD is 0.5% or less.

- 5.11. If you also want to perform a calibration verification, continue with the following steps.
- 5.12. Press **F1** to select Calibration. (If starting from the **Main** menu.)
- 5.13. Press **F2** to select Conductivity. (If starting from the **Cal** menu.)
- 5.14. Press F1 to select Conductivity Calibration. (If starting from the Cond menu.)
- 5.15. Make sure the 25 μ S/cm HCl standard is in the vial port. Press **F1**.
- 5.16. When the measurements are complete, record the values on the worksheet. Do not apply the calibration.

Calculate the Percent Difference via the following equation:

% Diff = <u>Measured Concentration - Expected Standard Concentration</u> x 100% Expected Standard Concentration

Acceptance criteria are as follows:

- RSD is 0.5% or less
- % Diff ±2%

Conductivity Calibration Worksheet

Company Name	Date
Analyst Name	Firmware Version
Sensor Serial Number	Standards Expiration Date
Standards Set Lot No. (optional)	

Value	Cell 1	Cell 2
Exp		
Meas		
RSD		
Adj		

Recommended acceptance criteria:

- Average values between 24-26 µS/cm
- RSD values of Cell 1 and Cell 2 are 0.5% or less

Calibration Result	s: 🛛 Passed	Failed

Calibration Action:	Accepted	Canceled
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Performed By:	Date:
Reviewed By:	Date:
Verified By:	Date:

Conductivity Verification Worksheet

Company Name	Date
Analyst Name	Firmware Version
Sensor Serial Number	Standards Expiration Date
Standards Set Lot No. (optional)	

Value	Cell 1	C	Cell 2	
Exp		(line 1)_		(line 2)
Meas		(line 3)_		(line 4)
RSD				
Adj				

Recommended acceptance criteria:

Calculate the Percent Difference via the following equation:

% Diff = <u>Measured Concentration - Expected Standard Concentration</u> x 100% Expected Standard Concentration



- RSD values of Cell 1 and Cell 2 are 0.5% or less
- % Diff values of Cell 1 and Cell 2 are ±2%

Verification Results:	□ Passed	□ Failed		
Performed By:			Date:	
Reviewed By:			Date:	
Verified By:			Date:	

TOC Calibration and Verification Protocol

- 1. **Purpose:** To perform a TOC calibration for the CheckPoint Portable/On-Line TOC Sensor.
- 2. **Scope:** This procedure applies to all CheckPoint Portable/On-Line TOC Sensors. Note that a Vial Sampling Kit is required to perform the protocol.

TOC Calibration should be performed after you have replaced the pump head. Remove the TOC calibration standards from the refrigerator and allow them to warm to room temperature (~ 1 hour) before starting calibration.

Note: Use only Sievers standards purchased from Veolia.

The analyst performing this protocol should be familiar with the terminology and operation of the Sensor.

3. Materials:

- 3.1. CheckPoint Portable/On-Line TOC Sensor
- 3.2. Vial Sampling Kit
- 3.3. **TOC Calibration Worksheet** (see page 30)
- 3.4. TOC calibration standards from Veolia, comprising:
- 2 vials of TOC Calibration Blank
- 1 vial of 500 ppb TOC (as sucrose)

Note: 1ppm = 1 mg C/L, 1 ppb = 1 μ g C/L

All standards should be warmed to room temperature prior to use.

Shake standards vials for 10-30 seconds prior to use.

4. Definitions:

4.1. TOC — total organic carbon

5. Procedure:

- 5.1. If the Sensor is taking measurements, press the **I** button.
- 5.2. Make sure the Vial Sampling Kit is connected, per the instructions in the Sensor's *Operation and Maintenance Manual.*
- 5.3. Press the 🖶 button.
- 5.4. Press **F1** to select the **Mode** screen.
- 5.5. Press **F1** to select **Calibration**.
- 5.6. Press **F1** to select **TOC**.
- 5.7. Press **F1** to select **TOC Calibration**.
- 5.8. Insert the reagent water blank into the vial port.
- 5.9. Press **F1** to start TOC Calibration. Reagent water is flushed through the Sensor and measurements on the blank begin.

- 5.10. When prompted, remove the reagent water blank and insert the 500 ppb Sucrose calibration standard into the vial port.
- 5.11. Press **F1**.
- 5.12. The standard is flushed through the Sensor and measurements on the standard begin.
- 5.13. When measurements are complete, the Sensor displays calibration results, including the mean TOC values for the blank and 500 ppb sucrose standard (measured), the expected value (500 ppb plus blank), and RSD of the 500 ppb sucrose standard replicates. Press **F1** to accept the calibration or **F4** to cancel.

Use the V button to scroll through the complete calibration results.

If a printer has been installed, the CheckPoint will print the results of each measurement.

There are no specific pass/fail criteria for the TOC calibration, but Veolia recommends that the calibration be applied if the TOC of the blank is \leq 60 ppb, the measured TOC of the 500 ppb standard is between 300-750 ppb, and the RSD of the replicate measurements of the 500 ppb standard is \leq 3%.

If a high reagent water blank is observed, this may be a result of a contaminated vial sampling kit. In this case, the kit should be flushed with low TOC water (such as the reagent water blank) using the grab mode until low TOC values are observed; then the calibration should be repeated.

The TOC calibration is somewhat dependent on ambient temperature, and it is recommended that the calibration be performed at the same ambient temperature at which the Sensor is normally operated.

Standard deviation and relative standard deviation are calculated as follows:

Standard Deviation = $\sqrt{\frac{n \sum x^2 - (\sum x)^2}{n(n-1)}}$ $\sum = \text{Sum of}$ x = Each Resultn = Number of Measurements in a set(# of repetitions - # of rejections)

Relative Standard Deviation (RSD) = $\frac{\text{Standard Deviation}}{\text{Measured TOC Concentration}} \times 100$

- 5.14. If you also want to perform a calibration verification, continue with the following steps.
- 5.15. Press F4 until the Mode screen displays.
- 5.16. Press **F2** to select **Grab Sample**.
- 5.17. Insert the unused reagent water blank into the vial port.
- 5.18. Press **F1** to begin analysis.
- 5.19. After the vial has been analyzed, the results screen is displayed. Record the values of the average and the RSD as Blank.

- 5.20. Press **F4** to continue.
- 5.21. Press **F2** to select Grab Sample.
- 5.22. Insert the 500 ppb sucrose Calibration standard into the vial port.
- 5.23. Press F1 to begin analysis.
- 5.24. After the vial has been analyzed, the results screen is displayed. Record the values of the average and the RSD as Measured.
- 5.25. Calculate the Expected value as 500 ppb + the average value of the reagent water blank and record the value.
- 5.26. Calculate the Percent Difference via the following equation:

% Diff = <u>Measured Concentration - Expected Standard Concentration</u> x 100% Expected Standard Concentration

Acceptance criteria are as follows:

- RSD of the replicate measurements of the 500 ppb standard is $\leq 3\%$
- % Diff ±10%

TOC Calibration Worksheet

Company Name	
Analyst Name	
Sensor Serial Number	
Standards Set Lot No. (optional)	

Date
Firmware Version
Standards Expiration Date

	Value
Blank	
Expected	
Measured	
Measured RSD	
Adjusted	

Recommended acceptance criteria:

- TOC of the blank is \leq 60 ppb
- Measured TOC of the 500 ppb standard is between 300-750 ppb
- RSD of the replicate measurements of the 500 ppb standard is $\leq 3\%$

Calibration Results:	□ Passed	Failed		
Calibration Action:	□ Accepted	□ Canceled		
Performed By:			Date:	
Reviewed By:			Date:	
Verified By:			Date:	

TOC Verification	Worksheet		
Company Name		Date	
Analyst Name		Firmware Version	
Sensor Serial Number		Standards Expiration Date	
Standards Set Lot No. (optio	nal)	_	
	Value	RSD%	
Blank	(Line 1)		
Expected	(Line 2)		
Measured	(Line 3)		
Calculate the Expected value record the value.	e as 500 ppb + the average	ge value of the reagent water blank and	
Expected = 500 ppb + (Line 1) = (Line 2)			
Calculate the Percent Different	nce via the following equ	ation:	
Measure	d Concentration - Expected	Standard Concentration	
% Diff = Expected Standard Concentration x 100%			
	(Line 3) –	(Line 2)	
% Diff =	() in a ()	x 100% =	
	(Line 2)	
Recommended Acce	ptance criteria are as follo	ows:	
RSD of the replicate mea	surements of the 500 ppl	o standard is \leq 3%	
• % Diff ±10%			
Verification Results: □ Pas	sed 🛛 Failed		
Performed By:		Date:	
Reviewed By:		Date:	
Verified By:		Date:	

System Suitability (Response Efficiency) Verification Protocol

- **1. Purpose:** To perform a system suitability verification test on the CheckPoint Portable/On-Line TOC Sensor.
- **2. Scope:** This procedure applies to all CheckPoint Portable/On-Line TOC Sensors. A Vial Sampling Kit is required to perform this protocol.

Note: Use only Sievers standards purchased from Veolia.

The analyst performing this protocol should be familiar with the terminology and operation of the Sensor.

3. Materials:

- 3.1. CheckPoint Portable/On-Line TOC Sensor
- 3.2. Vial Sampling Kit
- 3.3. System Suitability Worksheet (see page 34)
- 3.4. System suitability standards set from Veolia, comprising:
- 1 vial of Reagent Water— Rw
- 1 vial of 500 ppb TOC (as USP Sucrose) Rs
- 1 vial of 500 ppb TOC (as USP 1,4-Benzoquinone) Rss

Note: 1ppm = 1 mg C/L, 1 ppb = 1 μ g C/L

All standards should be warmed to room temperature prior to use.

Shake standards vials for 10-30 seconds prior to use.

4. Definitions:

- 4.1. DI deionized
- 4.2. TOC total organic carbon

5. Procedure:

- 5.1. Make sure the Vial Sampling Kit is connected, per the instructions in the Sensor's *Operation and Maintenance Manual.*
- 5.2. If the Sensor is taking measurements, press the **b**utton.
- 5.3. Press the 🖶 button.
- 5.4. Press **F1** to select the **Mode** screen.
- 5.5. Press **F2** to select Grab Sample.
- 5.6. Put the Rw vial in the vial port.
- 5.7. Press **F1** to start analysis.

- 5.8. After the vial has been analyzed, the results screen is displayed. Record the values of the three repetitions and the average on the Worksheet. Press **F4**.
- 5.9. Remove the Rw vial from the vial port, and then insert the Rs vial in the vial port.
- 5.10. Press F2 to select Grab Sample. Press F1 to start analysis.
- 5.11. After the vial has been analyzed, the results screen is displayed. Record the values of the three repetitions and the average on the Worksheet. Press **F4**.
- 5.12. Remove the Rs vial from the vial port and insert the Rss vial in the vial port.
- 5.13. Press F2 to select Grab Sample. Press F1 to start analysis.
- 5.14. After the vial has been analyzed, the results screen is displayed. Record the values of the three repetitions on the Worksheet.
- 5.15. Remove the Rss vial from the vial port.
- 5.16. Acceptance criteria for USP System Suitability is response efficiency between 85% and 115%.

Response efficiency is calculated as follows:

Response Efficiency (RE) =
$$\frac{(Rss - Rw)}{(Rs - Rw)} \times 100$$

Response Limit is calculated as follows:

Response Limit = Rs - Rw

5.17. Complete the **System Suitability Worksheet**.

- 5.17.1. Calculate the Response Efficiency and record the value on the Worksheet.
- 5.17.2. Calculate the Response Limit and record the value on the Worksheet.

5.17.3. Check the Pass or Fail checkbox, based on the results.

5.18. Return the Sensor to On-Line mode and resume normal operation.

System Company Nar	Suitability	Workshee	t	Date
Analyst Name				Firmware Version
Sensor Serial	Number			Standards Expiration Date
Standards Set	t Lot No. (optional)_			
	RW	Rs (sucrose)	Rss	(benzoquinone)
Rep 1				
Rep 2				
Rep 3				
Average TOC	of Reagent Water (Rw)		(Line 1)
Average TOC	of 500 ppb Sucrose	e Standard (Rs)	_	(Line 2)
Average TOC	of 500 ppb Benzoa	uinone Standard ((Rss)	(Line 3)
Response Effi	ciency		()	(Line 4)
Response Limit		(Ling 5)		
	int.			
	Res	ponse Efficiency (R	E) =	<u>Rss – Rw)</u> (Rs – Rw) x 100
	Res	ponse Limit = Rs	- Rw	
Acceptance ci	riteria for USP Syste	em Suitability is re □ Pass	spons □ I	e efficiency between 85% and 115% Fail
Performed By	:			Date:
Reviewed By:				 Date:
Verified By:				Date:

PERFORMANCE QUALIFICATION PROTOCOLS

- (Optional) Linearity Verification Protocol and Worksheet
- Limit of Detection and Limit of Quantitation Extrapolation Verification Protocol and Worksheet

or

• Limit of Detection and Limit of Quantitation Repetitive Measurement Verification Protocol and Worksheet

Linearity Verification Protocol

- **1. Purpose:** To demonstrate the linearity of a CheckPoint Portable/On-Line TOC Sensor.
- **2. Scope:** This procedure applies to all CheckPoint Sensors. It is assumed that the Sensor is calibrated.

Note: Use only Sievers standards purchased from Veolia.

The test compound used in the protocol is sucrose at TOC concentrations of 250, 375, and 500 ppb; linearity may be demonstrated using other compounds with minor changes to this protocol. The analyst performing this protocol should be familiar with the terminology and operation of the Sensor. This protocol should require approximately one hour to perform.

3. Materials:

- 3.1. CheckPoint Portable/On-Line TOC Sensor
- 3.2. Linearity Worksheet (see page 38)
- 3.3. Linearity standards from Veolia, comprising:
 - 1 vial of High Level Linearity Standard (500 ppb sucrose)
 - 1 vial of Mid Level Linearity Standard (375 ppb sucrose)
 - 1 vial of Low Level Linearity Standard (250 ppb sucrose)
 - 1 vial of Linearity Blank

NOTE: 1ppm = 1 mg C/L, 1 ppb = 1 μ g C/L

All standards should be warmed to room temperature prior to use.

4. Definitions:

4.1. TOC — total organic carbon

5. Procedure:

- 5.1. Make sure the Vial Sampling Kit is connected, per the instructions in the Sensor's *Operation and Maintenance Manual.*
- 5.2. If the Sensor is taking measurements, press the **I** button.
- 5.3. Press the 🖶 button.
- 5.4. Press **F1** to select the **Mode** screen.
- 5.5. Press **F2** to select **Grab Sample**.
- 5.6. Put the CheckPoint Linearity Blank vial in the vial port.
- 5.7. Press **F1** to start analysis.
- 5.8. After the vial has been analyzed, the results screen is displayed. Record the values of the three repetitions and the average on the Worksheet.
- 5.9. Remove the Linearity Blank vial from the vial port. Press **F4**. Insert the Low Level Linearity Standard vial in the vial port.

- 5.10. Press F2 to start Grab Sample. Press F1 to start analysis.
- 5.11. After the vial has been analyzed, the results screen is displayed. Record the values of the three repetitions and the average on the Worksheet.
- 5.12. Remove the Low Level Linearity Standard vial from the vial port. Press **F4**. Insert the Mid Level Linearity Standard vial in the vial port.
- 5.13. Press F2 to start Grab Sample. Press F1 to start analysis.
- 5.14. After the vial has been analyzed, the results screen is displayed. Record the values of the three repetitions and the average on the Worksheet.
- 5.15. Remove the Mid Level Linearity Standard vial from the vial port. Press **F4**. Insert the High Level Linearity Standard vial in the vial port.
- 5.16. Press F2. Press F1 to start analysis.
- 5.17. After the vial has been analyzed, the results screen is displayed. Record the values of the three repetitions and the average on the Worksheet.
- 5.18. Remove the High Level Linearity Standard vial from the vial port.
- 5.19. Complete the Linearity Worksheet.
 - 5.19.1. Calculate the Blank Corrected value for each standard by subtracting the average Blank value from the respective average Standard value.
 - 5.19.2. Using a common spreadsheet program, determine the linearity correlation coefficient R2 of the three blank-corrected TOC values by plotting measured versus expected.

$$R^2 \ge 0.96$$

where

$$R^{2} = \left(\frac{n\left(\sum xy\right) - \left(\sum x\right)\left(\sum y\right)}{\sqrt{\left[n\sum (x^{2}) - (\sum x)^{2}\right]\left[n\sum (y^{2}) - (\sum y)^{2}\right]}}\right)^{2}$$

X = Certified values of TOC standards

Y = Measured values of TOC standards (blank-corrected)

Linearity Worksheet

Company Name	Date
Analyst Name	Firmware Version
Sensor Serial Number	Standards Expiration Date
Standards Set Lot No. (optional)	

	CheckPoint Linearity Blank	CheckPoint Linearity Low Level Standard	CheckPoint Linearity Mid Level Standard	CheckPoint Linearity High Level Standard
Rep 1				
Rep 2				
Rep 3				
Average				
Copy Average	e Blank Value Here			
Subtract Blanl	k Value From Avera	ge		
Blank Correct	ed Value			

$$R^{2} = \left(\frac{n\left(\sum xy\right) - \left(\sum x\right)\left(\sum y\right)}{\sqrt{\left[n\sum \left(x^{2}\right) - \left(\sum x\right)^{2}\right]\left[n\sum \left(y^{2}\right) - \left(\sum y\right)^{2}\right]}}\right)^{2}$$

X = Certified values of TOC standards

Y = Measured values of TOC standards (blank-corrected)

	Correlation Coefficient $(R^2) \ge 0.96$	
	🗆 Pass 🛛 Fail	
Performed By:		Date:
Reviewed By:		Date:
Validated By:		Date:

Limit of Detection and Limit of Quantitation Verification Protocol Using Extrapolation

- **1. Purpose:** To demonstrate how to determine the Limit of Detection (LOD) and limit of quantitation (LOQ) for a CheckPoint Portable/On-Line TOC Sensor.
- 2. Scope: This protocol outlines a procedure determining the LOD and the LOQ for CheckPoint Portable/On-Line TOC Sensors. Veolia determined the instrument LOD of 0.21 ppb; this procedure is not intended to verify this value. The LOD was evaluated using high-quality water under controlled conditions. For additional information on this LOD, contact Veolia. This procedure applies to CheckPoint Portable/On-Line TOC Sensors. It is assumed that the instrument is calibrated. The analyst performing this protocol should be familiar with the terminology and operation of the Sensor.

LOD is used to evaluate when a signal is a result of instrument noise or analyte response. The end user may validate these values; however, an extremely high-quality water system is required.

The limit of quantitation is the value established by the user to provide guidance on meaningful versus non-meaningful data. This incorporates all conditions at the end-user site. While the CheckPoint Portable/On-Line TOC Sensor may be able to measure carbon concentration at very low levels, it is important to document the level above which the TOC result is meaningful.

Data obtained above the LOD indicates the presence of the analyte carbon, but if below the LOQ may be part of the variability of the sampling method, contamination, and so forth. Values in this range are considered suspect. Data above the user determined LOQ are considered valid.

Veolia has performed this evaluation based upon two methodologies. **The protocol presented here uses Method One for LOD/LOQ determination.**

Method One involves measuring a set of CheckPoint Linearity Standards and projecting the standard deviation of those measurements through the y- intercept to allow an estimation of the standard deviation at zero analyte concentration.

Method Two involves taking repeated measurements of ultrapure water and calculating the standard deviation of the measurements. The LOD is estimated as three times the standard deviation of this result.

3. Materials:

- 3.1. CheckPoint Portable/On-Line TOC Sensor
- 3.2. LOD/LOQ Using Extrapolation Worksheet (see page 42)
- 3.3. A completed CheckPoint Linearity Worksheet
- 3.4. Computer program or calculation equipped with the ability to perform statistical analysis.

4. Definitions:

- 4.1. DI deionized
- 4.2. LOD Limit of Detection, a value based upon evaluation of the maximum sensitivity of an analytical instrument. This value provides guidance as to what is noise and what is a real signal. This value is typically established at three times the standard deviation of the instrument noise level.
- 4.3. LOQ Limit of Quantitation, the point at which the measurements become quantitatively meaningful (defined as ten times the standard deviation).
- 4.4. TOC total organic carbon

5. Procedure:

- 5.1. Acquire linearity data.
 - 5.1.1. If the Linearity protocol **has been run** prior to beginning this protocol, obtain a copy of the linearity data.
 - 5.1.2. If the Linearity protocol **has not been run** prior to beginning this protocol, perform the **Linearity Verification Protocol** prior to calculating the LOD and LOQ. Once the **Linearity Verification Protocol** has been performed and the associated worksheet has been completed, obtain a copy of the data.
- 5.2. Using the data recorded on the **TOC Linearity Verification Worksheet**, complete the following information on the **LOD/LOQ Extrapolation Method Worksheet**.
 - 5.2.1. Record the measured TOC value of the Low Level Linearity Standard (250 ppb TOC as sucrose) for each Rep #.
 - 5.2.2. Record the measured TOC value of the Mid Level Linearity Standard (375 ppb TOC as sucrose) for each Rep #.
 - 5.2.3. Record the measured TOC value of the High Level Linearity Standard (500 ppb TOC as sucrose) for each Rep #.
 - 5.2.4. Record the average measured TOC values (not blank corrected) for each Standard.
 - 5.2.5. Calculate and record the Standard Deviation of the measured TOC values for each Standard.
- 5.3. Calculate Limit of Detection and Limit of Quantitation through extrapolation of Linearity data.
 - 5.3.1. Plot the average TOC concentration (not blank corrected) on the x axis versus the standard deviation on the y axis.
 - 5.3.2. Using a standard spreadsheet program, determine the y-intercept of the least squares linear regression line through the three points. The y-intercept represents the standard deviation at zero analyte concentration (y intercept = $|s_0|$). Record the y-intercept on line 1.
 - 5.3.3. Calculate the Limit of Detection on line 2 ($LOD = |y-intercept| \times 3$).
 - 5.3.4. Calculate the Limit of Detection on line 3 ($LOD = |y-intercept| \times 10$).

5.4. References.

- 5.4.1. Taylor, John K., *Quality Assurance of Chemical Measurements*, Lewis Publishers imprint of CRC Press (1987).
- 5.4.2. American Chemical Society Committee Report, "Guidelines for Data Acquisition and Data Quality Evaluation in Environmental Chemistry," *Anal. Chem.* 52:2242 (1980).
- 5.4.3. ASTM D5997 "On-line Monitoring of Total Carbon, Inorganic Carbon in Water by Ultraviolet, Persulfate Oxidation and Membrane Conductivity Detection."

LOD/LOQ Using Extrapolation Worksheet

Com	pany	Name
-		

Analyst Name _____

Date

Firmware Version_____

Sensor Serial Number_____

Populate the following table with data (Rep 1, Rep 2, Rep 3, and Average) from the Linearity Worksheet.

	CheckPoint Linearity Low Level Standard	CheckPoint Linearity Mid Level Standard	Chec Linea High Stand	kPoint arity Level dard	
Rep 1					
Rep 2					
Rep 3					
Average					
Standard Deviation					
y-intercept of th	ne regression line (standard deviation	on at zei	ro, s₀):	(line 1)
Limit of Detecti	ion:	ercept	x 3 =	LOD	(line 2)
Limit of Quantit	tation: y-inter	x	x 10 =	LOQ	(line 3)
Performed By:				Date:	
Reviewed By:				Date:	
Validated By:				Date:	

Limit of Detection and Limit of Quantitation Verification Protocol Using Repetitive On-line Measurements

- 1. **Purpose:** To demonstrate how to determine the Limit of Detection (LOD) and limit of quantitation (LOQ) for a CheckPoint Portable/On-Line TOC Sensor.
- 2. Scope: This protocol outlines a procedure determining the LOD and the LOQ for CheckPoint Portable/On-Line TOC Sensors. Veolia determined the instrument LOD of 0.21 ppb; this procedure is not intended to verify this value. The LOD was evaluated using high-quality water under controlled conditions. For additional information on this LOD, contact Veolia. This procedure applies to CheckPoint Portable/On-Line TOC Sensors. It is assumed that the instrument is calibrated. The analyst performing this protocol should be familiar with the terminology and operation of the Sensor.

LOD is used to evaluate when a signal is a result of instrument noise or analyte response. The end user may validate these values; however, an extremely high-quality water system is required.

The limit of quantitation is the value established by the user to provide guidance on meaningful versus non-meaningful data. This incorporates all conditions at the end-user site. While the CheckPoint Portable/On-Line TOC Sensor may be able to measure carbon concentration at very low levels, it is important to document the level above which the TOC result is meaningful.

Data obtained above the LOD indicates the presence of the analyte carbon, but if below the LOQ may be part of the variability of the sampling method, contamination, and so forth. Values in this range are considered suspect. Data above the user determined LOQ are considered valid.

Veolia has performed this evaluation based upon two methodologies. **The protocol presented here uses Method Two for LOD/LOQ determination.**

Method One involves measuring a set of CheckPoint Linearity Standards and projecting the standard deviation of those measurements through the y- intercept to allow an estimation of the standard deviation at zero analyte concentration.

Method Two involves taking repeated measurements of ultrapure water and calculating the standard deviation of the measurements. The LOD is estimated as three times the standard deviation of this result.

3. Materials:

- 3.1. CheckPoint Portable/On-Line TOC Sensor
- 3.2. LOD/LOQ Repetitive Method Worksheet (see page 46)
- 3.3. Computer program or calculator equipped with the ability to perform statistical analysis

4. Definitions:

- 4.1. DI deionized
- 4.2. LOD Limit of Detection, a value based upon evaluation of the maximum sensitivity of an analytical instrument. This value provides guidance as to what is noise and what is a real signal. This value is typically established at three times the standard deviation of the instrument noise level.
- 4.3. LOQ Limit of Quantitation, the point at which the measurements become quantitatively meaningful (defined as ten times the standard deviation).
- 4.4. TOC total organic carbon

5. Procedure:

- 5.1. If the Sensor is taking measurements, press the **b**utton.
- 5.2. (Optional) If **Password** protection is enabled, log in to the Sensor with the User ID and Password.
- 5.3. Install the Sensor on-line by following the instructions in the "Installation" chapter of the Sensor's *Operation and Maintenance Manual.*

Make sure that you are using the same output rate for the TOC measurement as you plan to use in normal operation.

- 5.4. Press the button to start analysis.
- 5.5. Perform the analysis.
- 5.6. After the Sensor has taken 30 measurements, press the **I** button.
- 5.7. Press the \bowtie button.

The Sensor displays time, TOC result, and conductivity for the last four TOC measurements collected.

- 5.8. Record the last nine TOC measurements on the **LOD/LOQ Repetitive Method Worksheet**.
- 5.9. Perform calculations and record values on the LOD/LOQ Repetitive Method Worksheet.
 - 5.9.1. Calculate the average TOC (line 1) and the standard deviation (line 2) of the last nine TOC values.
 - 5.9.2. Calculate the Limit of Detection on line 3 ($LOD = Standard Deviation \times 3$).
 - 5.9.3. Calculate the Limit of Quantitation on line 4 (*LOQ* = *Standard Deviation x 10*).

6. References:

- 6.1. Taylor, John K., *Quality Assurance of Chemical Measurements*, Lewis Publishers imprint of CRC Press (1987).
- 6.2. American Chemical Society Committee Report, "Guidelines for Data Acquisition and Data Quality Evaluation in Environmental Chemistry," *Anal. Chem.* 52:2242 (1980).

6.3. ASTM D5997 "On-line Monitoring of Total Carbon, Inorganic Carbon in Water by Ultraviolet, Persulfate Oxidation and Membrane Conductivity Detection."

LOD/LOQ Repetitive Method Worksheet

Company Name_____

Date____

Analyst Name_____

Firmware Version_____

Analyzer Serial Number_____

Last nine TOC readings:

Reading Number	TOC Reading	Reading Number	TOC Reading	Reading Number	TOC Reading
1		4		7	
2		5		8	
3		6		9	

Average TOC of the 9 readings

Standard Deviation of the 9 readings:

Standard Deviation =
$$\sqrt{\frac{n \sum x^2 - (\sum x)^2}{n (n-1)}}$$

∑ = Sum of

x = Each Resultn = Number of Measurements in a set (# of repetitions - # of rejections)

(line 1)

_____ (line 2)

Limit of Detection:	x 3 =	(line 3)
	(Standard Deviation)	. ,

Limit of Quantitation: x 10 = _____ (line 4) (Standard Deviation)

Performed By:	Date:
Reviewed By:	Date:
Validated By:	Date: