1. Procedure summary

This procedure describes the primary maintenance required for the Innovox TOC analyzer.

Related Procedures

Total Organic Carbon (TOC) Determination Using the Sievers Innovox TOC Laboratory Analyzer	LC-07-003-003

Procedure impacts and concerns

Safety	Safety glasses, gloves and a lab coat should be worn at all times while performing this procedure. Please read all applicable MSDS forms prior to performing this procedure. Dispose of all used reagents and parts according to approved disposal practices.
Quality	Optimal operation and quality of data is dependent on the quality and timing of maintenance given to the Innovox TOC analyzer. Ensure that the maintenance schedule is followed in order to avoid delays in sample analysis and data reporting.
Delivery	Maintenance should be conducted on a daily, weekly, monthly, tri-monthly and yearly basis. After maintenance has been conducted the maintenance log should be filled out in order to keep track of all maintenance performed. See below for specifics requirements.
Environmental	Local policies and procedures for disposal of used parts and spent reagents should be followed as determined by the site leadership
Cost	Long term cost impacts may be incurred if equipment is not properly maintained.
Compliance	Compliance with OSHA's Hazardous Waste Operations and Response, and Hazardous Communication Standard in addition to the Sapphire Energy, Inc. Chemical Hygiene Plan is required (see 29 CFR 1910.120 and 1200).

Responsibilities and owners

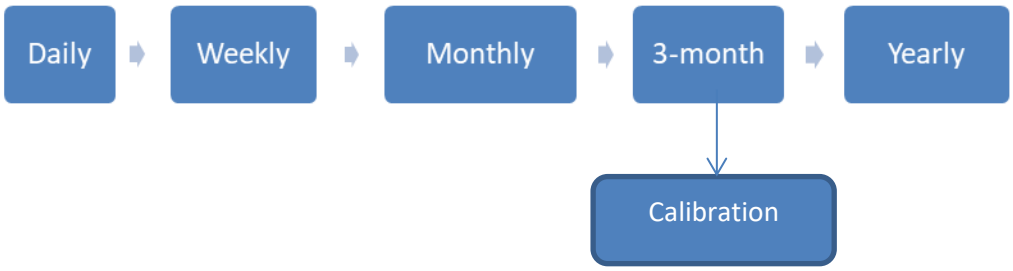
Document Owner	Manage content and distribution	Ronald Treminio
Process Owner	Responsible for content and process validation	Tonia Lane
Site Manager	Responsible for implementation and conformance	Nicole Heaps Becky Ryan (LCTS)

2. Process

2.1 Process description

The Innovox TOC analyzer requires a daily, weekly, monthly, tri-monthly and yearly schedule of maintenance in order to run and analyze samples properly.

2.2 Process diagram: Work Instruction



2.3 Equipment and Supplies

- InnovOx TOC Calibration standard 100mg/L (GE analytical # CSTD68310-01)
- InnovOx TOC Calibration standard 1000mg/L (GE analytica #CSTD68410-01)
- InnovOx TOC calibration standard 5000mg/L (GEanalytical #CSTD68450-01)
- InnovOx Oxiderzer reagent pack (GE analytical #CAPK68050-01)
- InnovOx Pinch tubing Kit (GE analytical # CAPK68100-010
- InnovOx Reactor seal kit (GE analytical # CAPK68110-01)
- Disc Filter (GEanalytical # CHTF34000)
- InnovOx NDIR Filter (GE analytical #CHFL68100-01)
- InnovOx Air filter cartridge (GE analytical #68120-01)
- Sodium Carbonate (Na_2CO_4, Sigma Aldrich # S7795-500G)

2.4 Process steps

<u>Daily Maintenance</u>

1. Open the right side of the analyzer unit by turning the thumb screws in the rear of the machine (Figure 1).
2. Check the water level of the gas-liquid separator, if low, fill to marked level by removing the center black hose from the large round filter and using a squirt bottle with nanopure water (See figure 2).
3. Check the cleanliness of the sparger and frit (frit is small white plug at the bottom of the sparger). Remove sparger from unit, disassemble and rinse with nanopure water if sparger is dirty (See figure 2).
4. Before running samples top off reagent water, 6M phosphoric acid and 30% persulfate (oxidizer) solution. Check to see that all tube ends extend into their respective solutions.
5. Empty waste into hazardous liquid, acid only waste container. Replace waste bottle and ensure that waste lines are properly inserted into the bottle.
6. Flush all lines via touch screen on analyzer unit (**Menu > Maintenance tab > Flush Reagents > Flush All**).
7. Once flush has completed, check air bubbles in lines. Flush independent lines if air bubbles remain.
8. Check sample line, if dirty, replace with clean sample line.

9. After daily maintenance has been conducted, the maintenance log should be updated to reflect what maintenance was conducted.

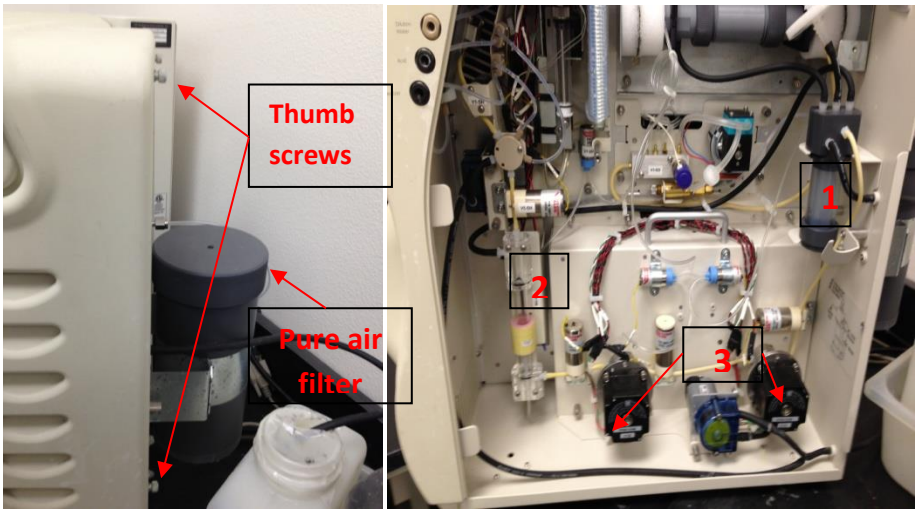


Figure 1 and 2. Figure 1 (left picture) shows a) the back panel thumb screws used to access the inside of the TOC analyzer and b) the pure air filter. Figure 2 (right picture) shows the inside of the TOC analyzer. Red 1 indicates location of the gas-liquid separator. Red 2 indicates the location of the sparger and frits. Red 3 indicates the high pressure valves.

Weekly maintenance

1. Run a 100 ppm Carbon standard (GE analytical # 68310-01)as a sample and review results. If the standard falls outside of 90-110 ppm range (±10% SD) run the standard again. If sample falls outside of 10% standard deviation again, recalibrate machine (see calibration steps below for calibration steps).
2. Clean sample line. Using a 1 mL syringe with a small gauge needle flush line with methanol. Then flush 3 times with nanopure water.
3. Replace water in water reservoir with nanopure water.
4. Open machine and remove gas-liquid separator dump water and disassemble. Clean the inside of the separator with nanopure water and kim-wipes (See figure 2). Re-assemble separator and place into the analyzer unit. Add nanopure water to refill up to marked level.
5. After weekly maintenance has been conducted the maintenance log should be updated to reflect the maintenance that was conducted.

Monthly maintenance

1. Clean sparger-tube and replace frit (See figure 2 and page 154 of operations manual).
2. Replace oxidizer (30 % persulfate solution) if older than 30 days.
3. After monthly maintenance has been conducted the maintenance log should be updated to reflect the maintenance that was conducted.

3 month maintenance

1. Replace air filter cartridge (See figure 1 and page 158 of operations manual).
2. Replace high pressure valve tips (See figure 2 and page 162-163 of operations manual).
3. After the three month maintenance has been conducted the maintenance log should be updated

to reflect the maintenance that was conducted.

Calibration TOC/TC protocol

1. Click **Protocol> Calibration> Open** in the sample input screen.
2. In the calibration window that opens up, select Default TC/TOC calibration and click **Open.**
3. Then click **Protocol> Calibration> Setup.**
4. In the Calibration set up Window input the following parameters:
   a. Calibration Mode: TC/TOC
   b. Number of IC points: **4** (selected from drop down menu)
   c. Range (ppm): **up to 5000** (selected from drop down menu)
   d. Acid %:**1.00**
   e. Oxidizer %: **15.0**
   f. Blank Correction: **Of**f
   g. Auto Dilution**: Off**
   h. Calibration Type: **Point to Point** (selected from drown menu)
5. In the Point (ppm) column enter the following standard concentrations[1].
   a. Vial 1 : 0 ppm (nano pure water)
   b. Vial 2 : 50 ppm [made by mixing 0.221 grams of Sodium Carbonate (Na_2CO_4, oven dried overnight) in 500ml of nanopure water].
   c. Vial 3: 100 ppm[mix 0.221 grams Sodium carbonate in 250ml nanopure water]
   d. Vial 4 : 500 ppm [mix 0.884 grams Sodium carbonate in 200 ml nanopure water]
   e. Vial 5 : 0 (used as a blank)
   f. Vial 6 : 0 ppm (nanopure water)
   g. Vial 7 : 100 ppm Standard (TOC calibration standard)
   h. Vial 8 : 1000 ppm Standard (TOC calibration standard)
   i. Vial 9 : 5000 ppm Standard (TOC calibration standard)
6. In repeat column, select **OFF**
7. In "Number of Repeats" column, enter **8**
8. In "Number of rejects" column enter **3**
9. In Flush column, select **OFF**
10. Place standards in appropriate spaces in sample tray (i.e. vial 1 should be placed in space 1 on the sample tray).
11. Click **Save As**
12. In the "Save as" window give the calibration protocol a name that contains the date that calibration is being conducted. Click **Save.**
13. Then click **RUN.**
14. After the run is complete, a window containing the results will appear on screen, scroll to the bottom of the window and click **APPLY** to set the calibration.

[1]Vials 1-4 are inorganic carbon standards Vial 5 is a blank water vial. Vials 6-9 are total organic carbon standards.

Calibration NPOC protocol

1. Click **Protocol> Calibration> Open** in the sample input screen.
2. In the calibration window that opens up, select **Default NPOC** calibration and click **Open.**
3. Then click **Protocol> Calibration> Setup.**
4. In the Calibration set up Window input the following parameters:
   a. Calibration Mode: **NPOC**
   b. Number of Points: 4 (selected from drop down menu)
   c. Range (ppm): **up to 5000** (selected from drop down menu)
   d. Acid %:**2.00**
   e. Oxidizer %: **15.0**

f. Blank Correction: **Off**
g. Auto Dilution**: Off**
h. Calibration Type: **Point to Point** (selected from drown menu)

5. In the Point (ppm) column, enter corresponding standards acquired from GE analytical:
   a. Vial 1 : 0 ppm (nano pure water)
   b. Vial 2 : 100 ppm TOC standard
   c. Vial 3 : 1000 ppm TOC standard
   d. Vial 4 : 5000 ppm TOC standard
6. In the Repeat column select **OFF**
7. In the Number of Repeats column, enter 8
8. In the Number of Rejects column, enter 3
9. In Flush column select OFF
10. Place standards in appropriate spaces in sample tray (i.e. vial 1 should be placed in space 1 on the sample tray).
11. Click **Save As**
12. In the "Save as" window give the calibration protocol a name that contains the date that calibration is being conducted. Click **Save.**
13. Then click **RUN.**
14. After the run is complete, a window containing the results will appear on screen, scroll to the bottom of the window and click **APPLY** to set the calibration.

Annual maintenance

1. Replace pinch valve tubing (see pages 155 -157 of operations manual).
2. Replace air pump diaphragm (see pages 163-165 of operations manual).
3. Replace disk filter (see pages 160-161 of operations manual).
4. Replace NDIR filter (see pages 159 -160 of operations manual).
5. Replace reactor peristaltic pump head and motor and reagent persistaltic pump motor (see page 146 of operations manual).
6. After annual maintenance has been conducted the maintenance log should be updated to reflect the maintenance that was conducted.

3. Required documents

3.1 Input documents

Sievers InnovoxOx Laboratory TOC Analyzer Operation and Maintenance Manual (DLM 68088-07) - (N:\Analytical\Instrumentation\GE-TOC)

3.2 Output documents

Maintenance log

4. Document control

Revision history

R0 – Initial Release – Ronald Treminio	9/9/2013

Document approval

Nicole Heaps 9/11/2013
Becky Ryan (LCTS)

Document reviewers

Tonia Lane	9/11/2013
Nicole Heaps	9/11/2013
Eric Ruvalcaba	9/12/2013

5. Risk analysis

6. Appendix

6.1. Maintenance Log

GE-TOC Maintenance Log

Date	Initials	Maintenance Performed	Notes