STANDARD OPERATING PROCEDURE
Water TOC Analysis Using the Elementar TOC Vario Cube

KEY WORDS

Total Organic Carbon, TOC, water, calibration curve, NPOC, acidification, maintenance

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1.0 INTRODUCTION

1.1 Purpose

The Elementar TOC Vario Cube (herein referred to as TOC Vario; Figure 1) may be used to measure TOC in water samples. TOC is a nonspecific water quality indicator which is measured for all current CDPR surface water monitoring studies. Prior to operation of the TOC Vario, complete the online Elementar training and review the TOC Vario manual. Carefully read all safety information in the TOC Vario manual and in the appendix of this Standard Operating Procedure (SOP).

1.2 Scope

This SOP includes instructions for how to analyze TOC in water using the TOC Vario, as well as how to perform routine maintenance on the analyzer. These maintenance procedures are to be done at set intervals, which are specified in the SOP.

1.3 Definitions

DPR – California Department of Pesticide Regulation

TOC – Total Organic Carbon

NPOC – Non-Purgeable Organic Carbon

DOC – Dissolved Organic Carbon

HCl – hydrochloric acid

DI water – deionized water

PPE – personal protective equipment

2.0 MATERIALS

2.1 Materials Used to Analyze TOC in Surface Water Samples

2.1.1 TOC Vario Cube

2.1.2 10 ml test tubes

2.1.3 10% HCl

2.1.4 DI water

2.1.5 TOC standards

2.1.6 Foil (cut into 1-inch squares)

2.1.7 Stirring rods (for turbid or saline samples)

2.2 Materials Used to Prepare 10% HCl for TOC Analysis

2.2.1 Gloves

2.2.2 Lab coat

2.2.3 Eye protection

2.2.4 Bottle of stock HCl

2.2.5 500 ml beaker

2.2.6 100 ml graduated cylinder

2.2.7 DI water

2.3 Materials Used for Maintenance of the TOC Vario

2.3.1 Calibration solutions (for TOC Vario calibration)

2.3.2 Gloves (for handling all TOC Vario parts and consumables)

2.3.3 Heatproof gloves (for performing maintenance in or near the combustion tube)

2.3.4 Face shield (for performing maintenance in or near the combustion tube)

2.3.5 Heatproof surface (for setting the ash finger and protective tube during maintenance of the combustion tube)

2.3.6 Tongs (for replacement of ash finger)

2.3.7 Quartz wool (for filling the combustion tube)

2.3.8 Quartz chips (for filling the combustion tube)

2.3.9 Platinum catalyst (for filling the combustion tube)

2.3.10 Magnesium perchlorate (for filling the drying tube)

2.3.11 Brass wool (for filling the absorption tube)

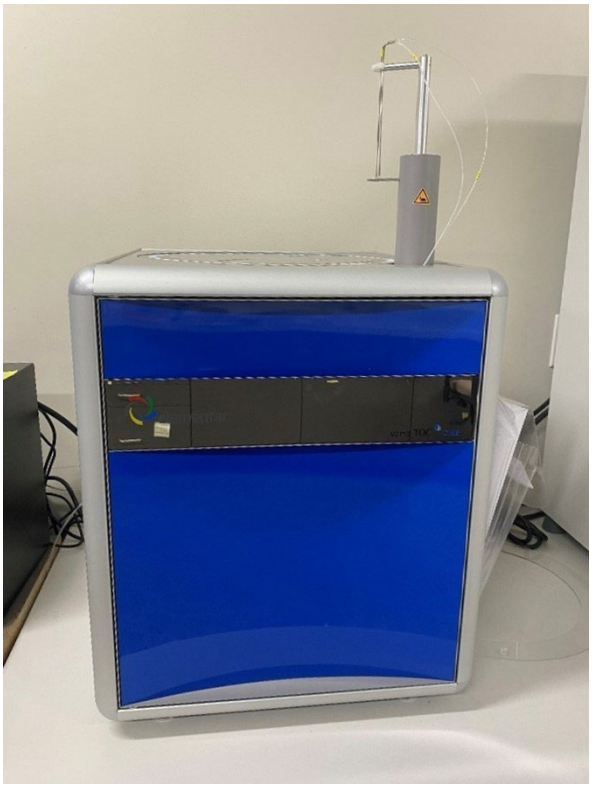


Figure 1: The TOC Vario Cube.

3.0 PROCEDURES

3.1 Procedures for Turning the TOC Vario On and Opening the "VarioTOC" Software

3.1.1 Turn on the TOC Vario using the green button on the right side of the analyzer. If the button is lit, and if you can hear noises from inside of the TOC Vario, then the analyzer is already on (Figure 2).

3.1.2 Log onto the TOC machine PC. Open the program, "VarioTOC," on the desktop. Enter the password provided on the piece of paper in front of the PC.

3.1.3 Verify that the TOC Vario is in liquid mode, which is specified in the title bar. See Figure 3 below for the VarioTOC window layout. If the analyzer is in solids mode, then see Section 3.8, below, for procedures on switching the analyzer to liquids mode.

3.1.4 If the TOC Vario is in sleep mode, then click on the alarm clock button in the toolbar to “wake up” the analyzer.

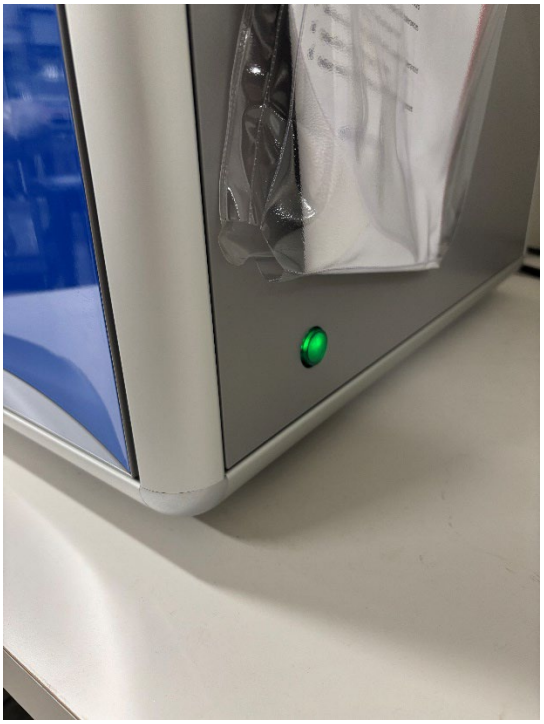


Figure 2: The green button to the front right is lit, indicating that the machine is powered on.

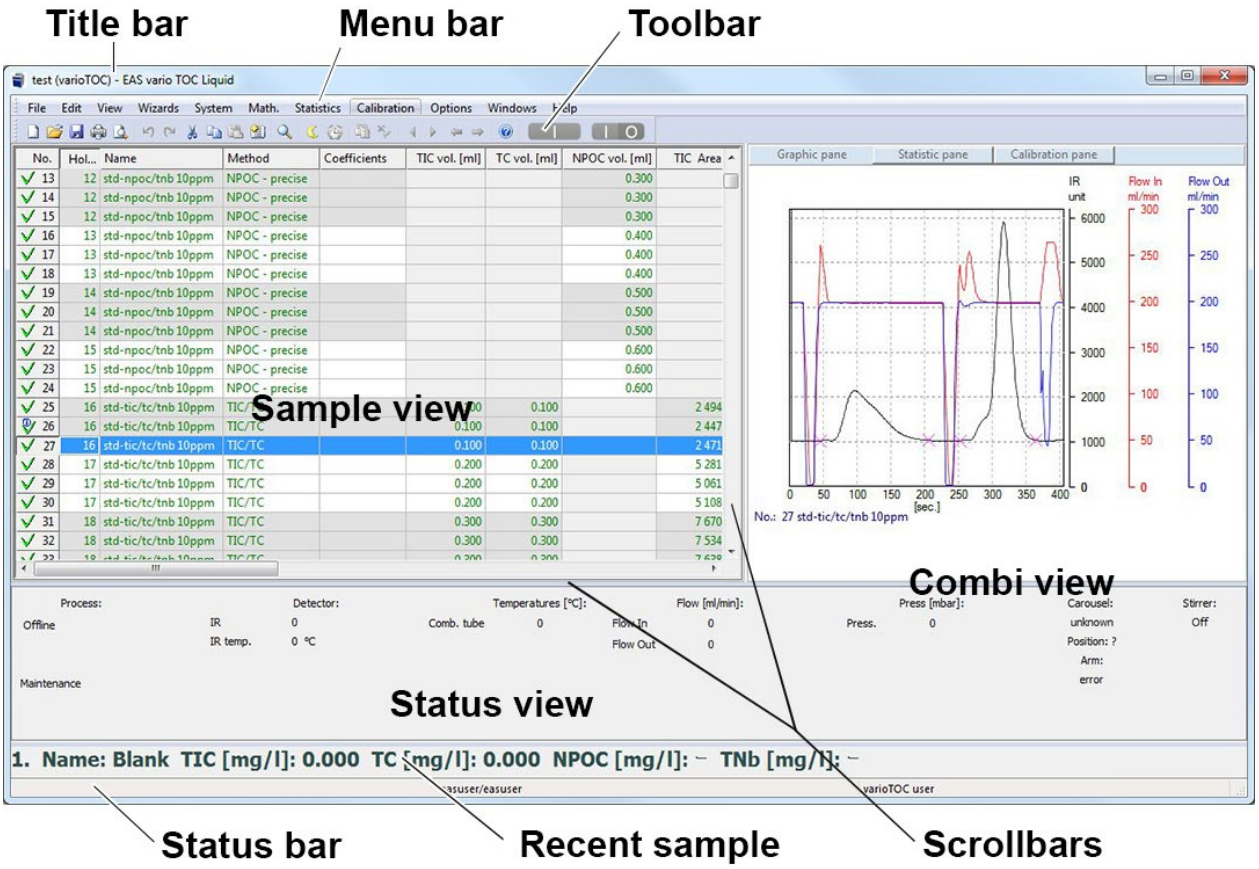


Figure 3: The window layout for the VarioTOC software. Image by: Elementar Analysensysteme GmbH.

3.2 Procedures for Measuring NPOC (TOC) in Conventional Samples

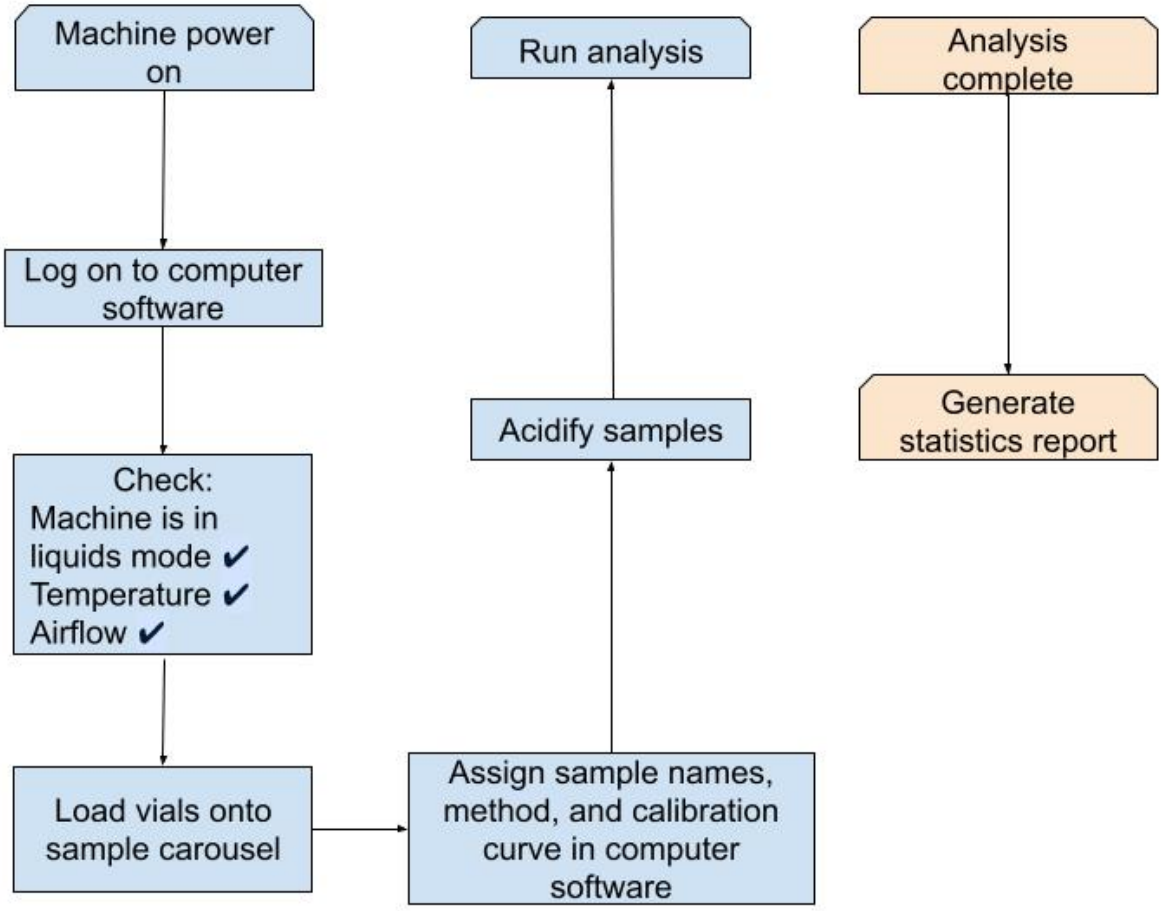
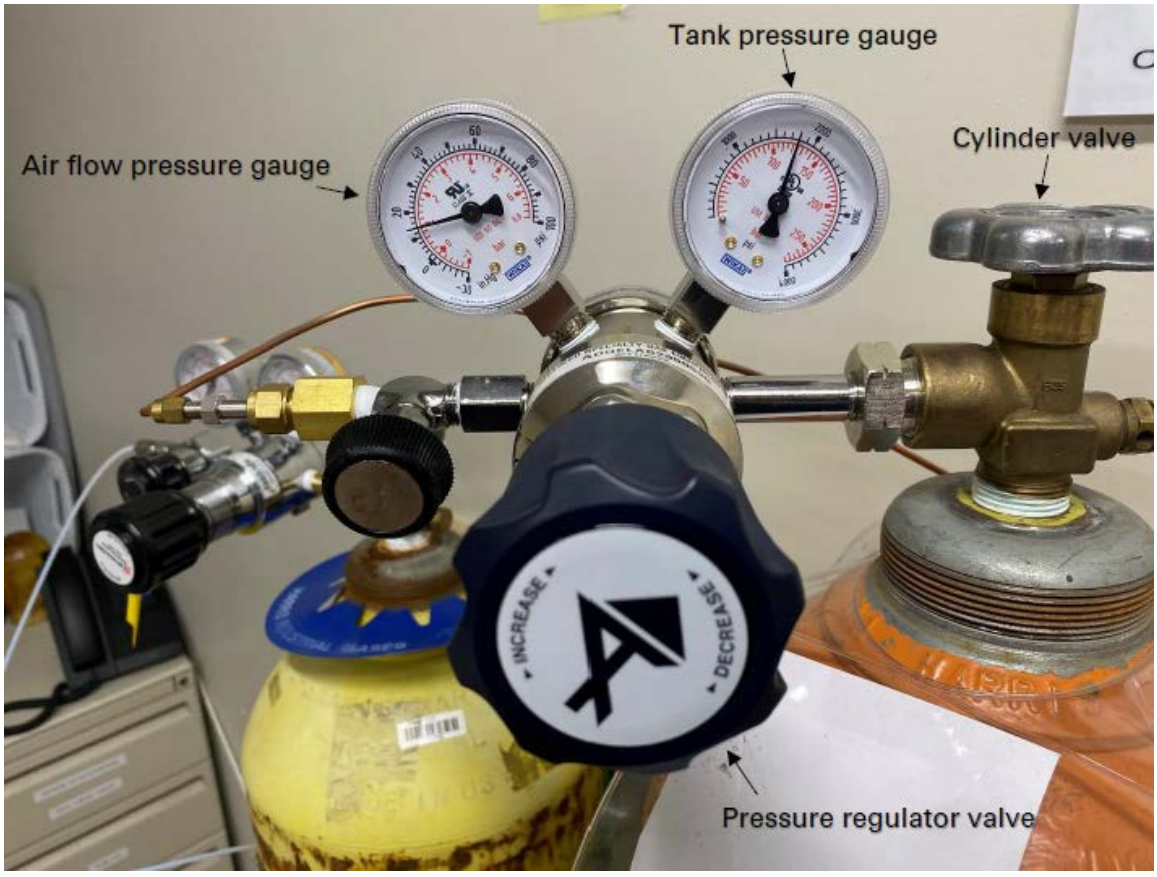


Figure 4: Water TOC analysis flowchart.

3.2.1 See flowchart summarizing the procedure above (Figure 4). Prior to sample analysis, supply the ultrazero air by turning the cylinder valve on the oxygen tank counterclockwise (Figure 5). Check the pressure in the status view of the of the VarioTOC software. The pressure of air being supplied to the TOC Vario should be approximately 1000 mbar (14.5 psi). Use the pressure regulator valve on the top of the tank to control the pressure of air available to the TOC Vario. Adjust such that the pressure in the status view is as close to 1000 mbar as possible. If the gauge on the right, which measures the amount of air left in the tank, reaches < 1000 psi, then the tank should be replaced.



3.2.2 Check that the temperature of the furnace is equal to 850 °C.

3.2.2.1 Set the furnace temperature: In the VarioTOC software, from the menu bar, click “Options” > “Settings” > “Parameters”.

3.2.3 Load vials containing blanks, standards, and samples into the carousel on top of the analyzer.

3.2.3.1 If needed, reposition the carousel (“System” > “Carousel Position.”). The injection needle may be blocking access to hole no. 1 in the initial loading. Entering “30,” for example, will change the carousel position to allow for access to hole no. 1.

3.2.3.2 Load holes no. 1-5 with “blanks” or test tubes containing DI water, for quality assurance. When run, these allow for system stabilization.

3.2.3.3 Load hole no. 6 with ultrazero water, serving as the 0-ppm standard. Load hole no. 7 with the 10-ppm standard. Load these standards again after every 20 samples for quality assurance.

3.2.3.4 Load vials containing samples in the subsequent holes. Load the DOC (filtered) samples first, followed by the TOC samples.

3.2.3.5 Ensure vials are covered using one-inch squares of foil to preserve the TOC contained in the samples.

3.2.4 In the VarioTOC software, enter the corresponding sample names in the “name” column. See Figure 6 below as an example. Note: the Elementar TOC Vario Cube is set to analyze samples in three replicates. When the sample name is entered, the replicates are automatically populated.

3.2.4.1 When entering the names of the standards, **do not** select the names from the dropdown menu. These standard names, displayed in green text, are for calibration only. Green text for the blanks is correct. (use dropdown for blanks, write in “0” and “10” for standards).

3.2.5 Within the method column, select the method, “NPOC – precise – 0.5” from the drop-down menu on line 1 of the spreadsheet. Press “enter” to automatically populate the method to the rest of the samples.

3.2.5.1 Note: “0.5” refers to the injection volume, 0.5 ml, which is optimal for analyzing surface water samples using this analyzer.

3.2.5.2 For method selection of turbid or saline samples, see "3.3 Procedures for Measuring NPOC in Saline or Turbid Samples." **Selecting the incorrect method for saline or turbid samples may result in clogging the injection needle, which will result in inaccurate measurements for subsequent samples and will require replacement of the injection needle.**

3.2.6 Select the calibration curve in the "Coefficients" column (if needed). The TOC Vario will automatically use the default coefficient if this field is left blank. For our purposes, the default coefficient should be the most updated calibration curve and should be used unless instructed otherwise.

No.	Hole ...	Name	Method	Coefficients	NPOC [mg/l]
1	1	Blank	NPOC - precise - 0.5		0.000
2	1	Blank	NPOC - precise - 0.5		0.000
3	1	Blank	NPOC - precise - 0.5		0.000
4	2	Blank	NPOC - precise - 0.5		0.000
5	2	Blank	NPOC - precise - 0.5		0.000
6	2	Blank	NPOC - precise - 0.5		0.000
7	3	Blank	NPOC - precise - 0.5		0.000
8	3	Blank	NPOC - precise - 0.5		0.000
9	3	Blank	NPOC - precise - 0.5		0.000
10	4	Blank	NPOC - precise - 0.5		0.000
11	4	Blank	NPOC - precise - 0.5		0.000
12	4	Blank	NPOC - precise - 0.5		0.000
13	5	Blank	NPOC - precise - 0.5		0.000
14	5	Blank	NPOC - precise - 0.5		0.000
15	5	Blank	NPOC - precise - 0.5		0.000
16	6	0	NPOC - precise - 0.5		0.000
17	6	0	NPOC - precise - 0.5		0.000
18	6	0	NPOC - precise - 0.5		0.000
19	7	10	NPOC - precise - 0.5		0.000
20	7	10	NPOC - precise - 0.5		0.000
21	7	10	NPOC - precise - 0.5		0.000
22	8	3105516	NPOC - precise - 0.5		0.000
23	8	3105516	NPOC - precise - 0.5		0.000
24	8	3105516	NPOC - precise - 0.5		0.000
25	9	3105526	NPOC - precise - 0.5		0.000
26	9	3105526	NPOC - precise - 0.5		0.000
27	9	3105526	NPOC - precise - 0.5		0.000
28	10	3105536	NPOC - precise - 0.5		0.000
29	10	3105536	NPOC - precise - 0.5		0.000
30	10	3105536	NPOC - precise - 0.5		0.000
31	11	3105546	NPOC - precise - 0.5		0.000
32	11	3105546	NPOC - precise - 0.5		0.000
33	11	3105546	NPOC - precise - 0.5		0.000
34	12	3105556	NPOC - precise - 0.5		0.000
35	12	3105556	NPOC - precise - 0.5		0.000
STOP 36	12	3105556	NPOC - precise - 0.5		0.000

Figure 6: An example of an analysis schedule for surface water samples. First, the analyzer will run blanks, followed by analyzing the standards and the surface water samples. The appropriate methods are selected for the conventional and turbid samples. The coefficients column is left blank, so the default calibration curve will be used for this set of analyses.

3.2.7 Acidify samples.

3.2.7.1 Wear the following PPE when handling acids: gloves, lab coat, closed toe shoes, safety glasses.

3.2.7.2 Pour the 10% HCl solution into the test tube marked, "HCl".

3.2.7.2.1 See "3.4 Procedures for Preparing 10% HCl for TOC Analysis" for instructions on preparing the acid solution.

3.2.7.3 Place the test tube containing the 10% HCl solution into hole no. 79.

3.2.7.4 Use the VarioTOC software to perform automatic sample acidification.

3.2.7.4.1 From the menu bar, click "System" > "Acidify Samples." See Figure 7 below.

3.2.7.4.2 In the "acid container" box, enter "79."

3.2.7.4.3 In the "Samples range" box, specify the samples to acidify. E.g., "7-20,22-40." Do not include spaces. Do not acidify blanks or standards.

3.2.7.4.4 Click "OK" to initiate automatic acidification.

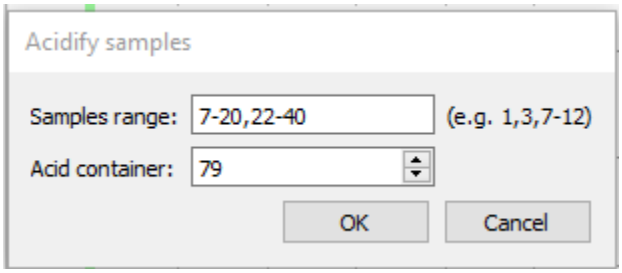


Figure 7: The sample acidification window, with correct numerical formatting for surface water sample acidification.

3.2.8 Once automatic acidification has been completed, click on the large green button in the toolbar to begin TOC analysis.

3.2.9 When TOC analysis has been completed, see Section "3.5 Procedures for Generating a Statistical Report" and Section "3.7 Engaging Standby Mode/Turning the TOC Vario Off".

3.3 Procedures for Measuring NPOC in Saline or Turbid Samples

3.3.1 Follow the instructions for conventional samples above to load the samples onto the carousel and entering the corresponding sample names into the VarioTOC computer software.

3.3.1.1 It is best practice to run at least one blank after each saline or turbid sample to ensure that leftover particles in the TOC Vario system don't affect subsequent results.

3.3.2 For any turbid or saline samples, place a small, white, magnetic stirring rod into the test tube. This prevents the injection needle from clogging (Figure 8).

3.3.3 Engage the automatic stirring of samples.

3.3.3.1 From the menu bar, click "System" > "Stirrer" > "On". The stirrer status is displayed on the bottom right of the status view.

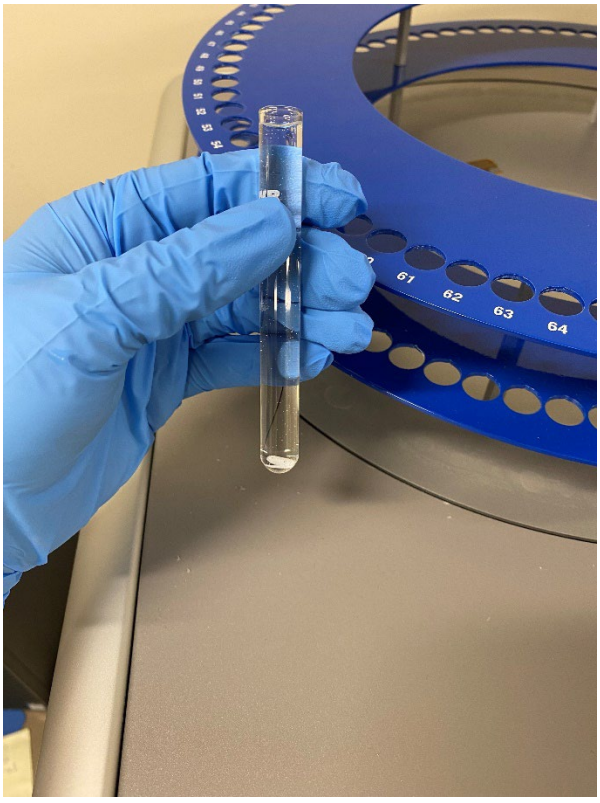


Figure 8: A test tube containing a surface water sample with a stir rod at the bottom.

3.3.4 Select the method, "NPOC – particle – 0.2" for the turbid and saline samples.

3.3.4.1 **Warning: Selecting the incorrect method for saline or turbid samples may result in clogging the injection needle, which will result in inaccurate measurements for subsequent samples and will require replacement of the injection needle.**

3.3.5 Follow the instructions for conventional samples above to select coefficients, acidify samples, and run the TOC Vario to begin analysis.

3.3.6 After the analysis of saltwater samples has been completed, flush salts from the combustion tube. Set the furnace temperature to 0 °C and ensure that the TOC Vario furnace has cooled < 55 °C before proceeding. Follow the procedures in Section 3.10 to remove the combustion tube. Flush the tube with enough DI water to fill the top of the tube. Allow water to drain and reinstall the combustion tube.

3.3.6.1 Set the furnace temperature: In the VarioTOC software, from the menu bar, click "Options" > "Settings" > "Parameters".

Figure 9: Flushing of the combustion tube after saltwater TOC analysis. Turn off furnace and ensure the combustion tube has cooled prior to flushing.

3.4 Procedures for Preparing 10% HCl for TOC Analysis

3.4.1 When preparing acid solutions, wear the following PPE: gloves, lab coat, closed toe shoes, and eye protection. Work in the fume hood of the testing lab with the ventilation turned on.

3.4.2 Pour 9 ml of DI water into a beaker.

3.4.3 Pour a small amount of the HCl into the beaker marked, "acid prep".

3.4.4 Using a pipette, transfer 1 ml of HCl into the graduated cylinder containing the DI water, bringing the solution volume to 10 ml.

3.4.4.1 **Always add acid to water. Do not add water to acid.**

3.4.5 Swirl the graduated cylinder to homogenize the solution.

3.4.6 Carefully pour the solution into the test tube marked, "HCl". Do not fill tube all the way; leave at least 0.5 cm of space at the top so that acid does not pour on top of the analyzer during acidification.

3.4.7 Dispose of any leftover HCl into the HCl waste bottle located beneath the fume hood.

3.4.8 Place the test tube in hole no. 79 of the carousel. See step 3.2.7 for instructions for automatic acidification.

3.5 Procedures for Generating a Statistical Report

3.5.1 Generating a statistical report will export the analysis results into a PDF. The analysis must be completed before the report is generated. Note: the user who was logged on at the time of analysis must be logged on to generate the report.

3.5.2 To generate a statistical report, click "Statistics" > "By multiple determination" in the menu bar.

3.5.2.1 Click "yes" in the dialog box that clears static memory

3.5.2.2 A table will populate in the statistics pane, which provides the mean from the results measured from the three injections.

3.5.3 Save the statistical report as a PDF.

3.5.3.1 From the menu bar, click "File" > "Print".

3.5.3.2 In the "configure report" dialogue box, select "statistic" from the dropdown menu on the lower left side, and click "OK" (Figure 10).

3.5.3.3 In the "print" dialogue box, select "Microsoft Print to PDF" in the "Name" dropdown menu.

3.5.3.4 Save PDF (press OK).

3.5.4 Save the results as a file in the VarioTOC software.

3.5.4.1 From the menu bar, click "File" > "Save."

3.5.4.2 Name the file according to the analysis type and date performed. Click "save" and the file may be viewed in the software later. Note: saved files may only be viewed from the user who was logged on at the time of file save.

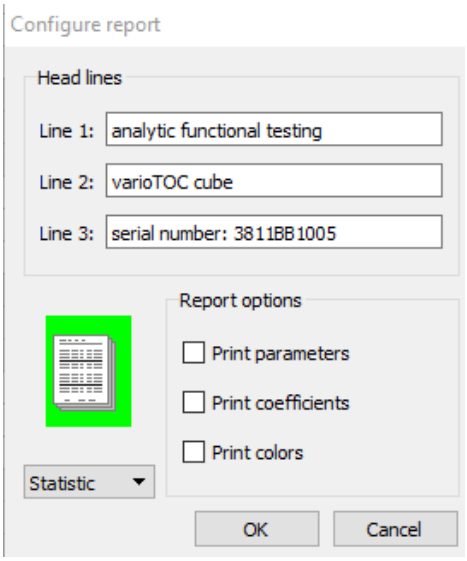


Figure 10: The report configuration window. "Statistic" is selected from the dropdown menu to the bottom left.

3.6 Procedures for Calibrating the TOC Vario Cube

3.6.1 Open the VarioTOC computer software.

3.6.2 On the carousel located on top of the Vario Cube, load holes no. 1-5 with "blanks," or test tubes containing DI water, for quality assurance.

3.6.3 Load TOC standard calibration solutions into the carousel.

3.6.3.1 Hole no. 6: ultrazero water (0 ppm standard).

3.6.3.2 Hole no. 7: 10 ppm standard.

3.6.3.3 Hole no. 8: 25 ppm standard.

3.6.3.4 Hole no. 9: 50 ppm standard.

3.6.3.5 Hole no. 10: 100 ppm standard.

3.6.4 Enter the names of the blanks and calibration standards in the "name" column of the VarioTOC window.

3.6.4.1 Select the corresponding standard names from the **dropdown** menu. The concentrations of these standards have previously been entered into the software. The text for the blanks and standards should be green.

3.6.5 Select the method, "NPOC – precise – 0.5".

3.6.5.1 Note: coefficients generated from this calibration will still apply to other NPOC methods, such as "NPOC – particle".

3.6.6 Press the green button to start auto-analysis.

3.6.7 When the analysis is completed, complete calibration.

3.6.7.1 From the menu bar, click "Calibration" > "Calibrate." The calibration pane will open on the right side of the window.

3.6.7.2 Select "one calibration range" and leave the polynomial order set to 1.

3.6.7.3 Use the small blue arrows to view individual results. If necessary, delete any extraneous points using the red "X" to achieve the highest r^2 value.

3.6.7.4 Confirm the final parameters in the calibration coefficients and click "OK" to finalize calibration.

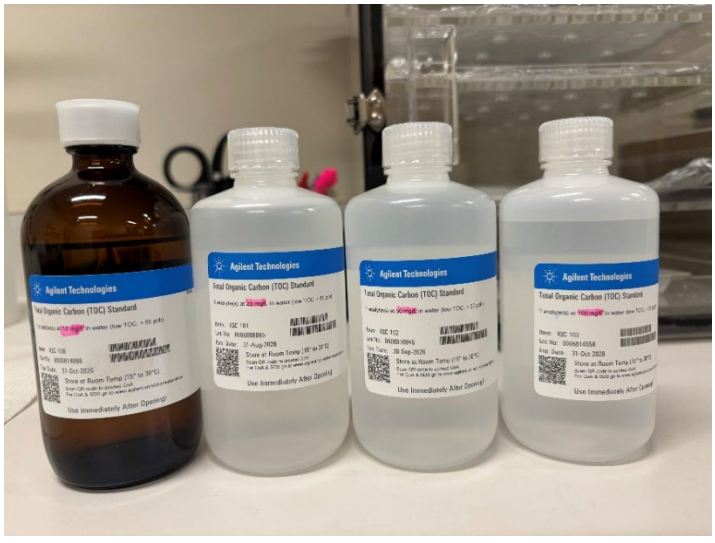


Figure 11: The solutions used for the calibration of the TOC machine. From left to right: 10 ppm, 25 ppm, 50 ppm and 100 ppm.

3.7 Procedures for Engaging Standby Mode/Turning the TOC Vario Off

3.7.1 Note: the TOC Vario should be powered off if no samples are to be measured for longer than one week.

3.7.2 To engage standby mode:

3.7.2.1 Close the VarioTOC software.

3.7.2.2 Turn the cylinder valve above the ultrazero air tank clockwise to cut off the supply of air to the analyzer.

3.7.2.3 After a period of inactivity in the VarioTOC software, the analyzer will automatically enter standby mode. If the TOC Vario will not be used again within a week, then power it off.

3.7.3 To power off the TOC Vario:

3.7.3.1 Set the furnace temperature to 0 °C in the VarioTOC software. From the menu bar, click “Options” > “Settings” > “Parameters”.

3.7.3.2 Wait until the TOC Vario has cooled < 55 °C before proceeding. This will take a few hours. Warning: the TOC Vario cannot properly ventilate when powered off. Powering the analyzer off before it has cooled may result in overheating and damage.

3.7.3.3 Shut off gas supply and close the VarioTOC software.

3.7.3.4 Press the green button on the right side of the analyzer to power off.

3.8 Procedures for Switching the Analyzer from Solids Mode to Liquids Mode

3.8.1 Set the TOC Vario to part replacement mode before proceeding. Click “Options” > “Maintenance” > “Replace Parts…”

3.8.2 Set the furnace temperature to 0 °C in the VarioTOC software. From the menu bar, click “Options” > “Settings” > “Parameters.” Wait until the TOC Vario has cooled < 55 °C before proceeding.

3.8.3 Unscrew the knobs at the top of the carousel. Remove the carousel and cover plate (Figure 12).

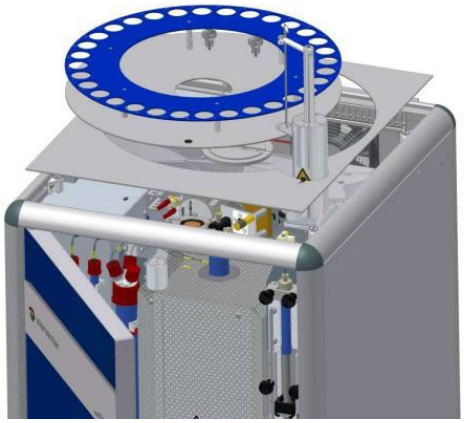


Figure 12. The removal of the cover plate and the carousel. Image by: Elementar Analysensysteme GmbH.

3.8.4 Remove the solid mode ball valve from the top of the combustion tube.

3.8.4.1 Loosen the locking screws and set them aside.

3.8.4.2 Unclip the tubing which connects the flow of gas to the combustion tube. The tubing is marked, number 10.

3.8.4.3 Remove the solids mode ball valve from the top of the combustion tube using careful back-and-forth motions (Figure 12).

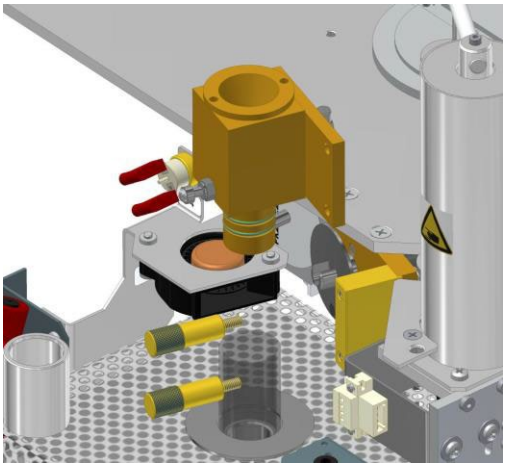


Figure 13: The removal of the ball valve from the top of the combustion tube. Image by: Elementar Analysensysteme GmbH.

3.8.5 Remove the solids mode combustion tube from the clamp at the base of the tube. Replace with liquid mode combustion tube. For filling the liquid mode combustion tube, see section 3.9.

3.8.6 Place multiway valve on top of the combustion tube.

3.8.6.1 Remove multiway valve from dock (see Figure 14). Place on top of the combustion tube. Use some force to plug the bottom of the valve back into the tube. Make sure that the valve is level, locked into its position, and the back of the valve is aligned with the tappet of the motor.

3.8.6.2 Reconnect the tubing which brings gas to the combustion tube, marked number 10.

3.8.6.3 Screw the locking screws to lock the valve into position.

3.8.7 Place the cover plate and carousel back onto the top of the analyzer.

3.8.8 Set the TOC Vario to liquids mode.

3.8.8.1 From the menu bar in the VarioTOC software, click “System” > “Mode”.

3.8.8.2 Check the box for TC/TIC/NPOC liquid analysis. Close the window.

3.9 Procedures for Replacing the Ash Finger

3.9.1 Follow the below procedures after the analyses of 800 samples. Please note that no part replacements should take place during the analysis. Set the TOC Vario to part replacement mode before proceeding.

3.9.2 Remove the used ash finger.

3.9.2.1 Remove the cover plate and carousel from the analyzer (Figure 12).

3.9.2.1.1 From the menu bar, click “System” > “Arm up”.

3.9.2.1.2 Loosen the thumbscrews on top of the analyzer. Set aside. Remove the carousel.

3.9.2.1.3 Remove the cover plate.

3.9.2.2 Remove the multiway valve from the top of the combustion tube (Figure 14).

3.9.2.2.1 Caution: the combustion tube is approximately 850 °C. Wear a face shield and the green heatproof protective gloves provided by Elementar before proceeding.

3.9.2.2.2 Note: It is recommended by Elementar to perform ash finger replacement with the furnace powered on, as regularly changing the temperature can lead to increased wear of internal parts of the machine. However, it is also an option to work with the furnace turned off. Personal safety is always the highest priority.

3.9.2.2.3 Loosen the brass locking screws from the multiway valve and set them aside.

3.9.2.2.4 Unclip the tubing which connects the flow of gas to the combustion tube. The tubing is marked, number 10.

3.9.2.2.5 Loosen the plug of the multiway valve with careful back-and-forth motions from the combustion tube.

3.9.2.2.6 Pull the multiway valve out from the top of the combustion tube. Rest valve on the docking station on the near left side of the plate covering the combustion tube.

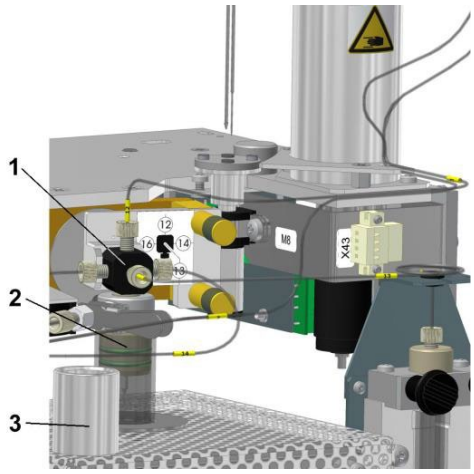


Figure 14: The analyzer components above the combustion tube. 1. Multiway valve. 2. Combustion tube. 3. Dock for multiway valve. Image by: Elementar Analysensysteme GmbH.

3.9.2.3 Remove the protective tube and ash finger from the combustion tube (Figure 15).

3.9.2.3.1 Caution: the combustion tube is approximately 850 °C. Wear the heatproof green protective gloves provided by Elementar, as well as a face shield, before proceeding. Place protective tube and used ash finger on a heatproof surface and do not touch until they have completely cooled.

3.9.2.3.2 Insert the tongs into the top of the combustion tube. Pull out the protective tube, which rests on top of the ash finger, by fitting the tongs into the slits on the sides of the tube. Place the protective tube on a heatproof surface.

3.9.2.3.3 Once the protective tube has been removed, insert the tongs into the top of the combustion tube to remove the ash finger. Pull out ash finger by fitting the tongs into the slits on the sides of the tube. Place the ash finger on a heatproof surface. Dispose of used ash finger once it has completely cooled.

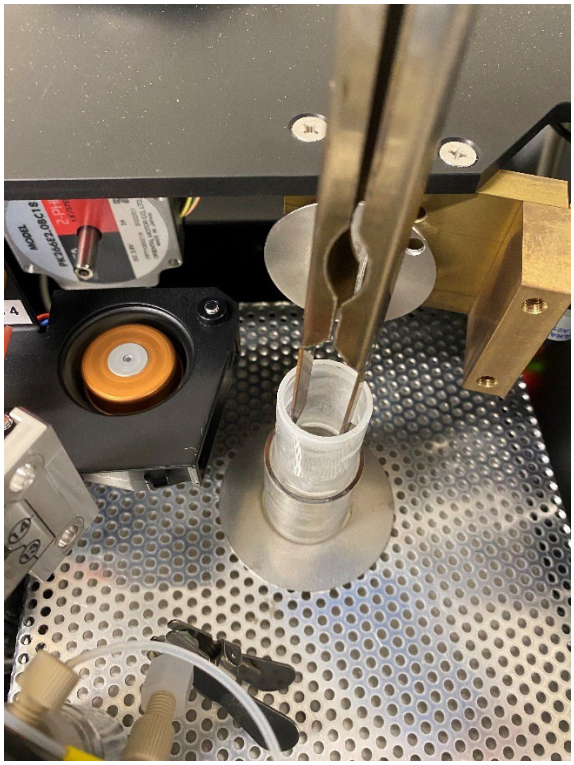


Figure 15: The use of tongs to remove the ash finger from the top of the combustion tube.

3.9.3 Continue to wear PPE for this step. Place new ash finger into the combustion tube by picking it up with the tongs, using the slits on the sides as guides, and inserting the ash finger at the bottom of the combustion tube.

3.9.4 Continue to wear PPE for this step. Place the protective tube into the combustion tube by picking it up with the tongs, using the slits on the sides. Then insert the protective tube into the combustion tube, placing it on top of the ash finger.

3.9.5 Reassemble multiway valve.

3.9.5.1 Remove multiway valve from dock. Place on top of the combustion tube. Use some force to plug the bottom of the valve back into the tube. Make sure that the valve is level, locked into its position, and the back of the valve allows for aligned screw holes.

3.9.5.2 Reconnect the tubing, marked 10.

3.9.5.3 Screw the locking screws to lock the valve into position.

3.9.6 Place the cover plate and carousel back onto the top of the analyzer.

3.10 Procedures for Removing and Filling the Combustion Tube

3.10.1 The filling of the combustion tube should be replaced after every 1000 samples. The whole combustion tube should be replaced after every 3000 samples.

3.10.2 Turn off the furnace of the analyzer. Only proceed with the following steps once the combustion tube is **cool**, and when the TOC Vario has been set to **part replacement mode**.

3.10.2.1 From the menu bar in the TOC Vario software, click “Settings” > “Parameters.”

3.10.2.2 Enter “0” for furnace temperature.

3.10.3 Follow steps above from “3.9. Procedures for Replacing the Ash Finger” to remove the carousel, cover plate, multiway valve, protective tube, and ash finger from the TOC Vario.

3.10.4 Unclamp the combustion tube from the glass tubing at the bottom. Lift the combustion tube from the furnace area by hand.

3.10.5 Dispose of the used quartz wool, quartz chips, and platinum catalyst within the combustion tube (Figure 16).

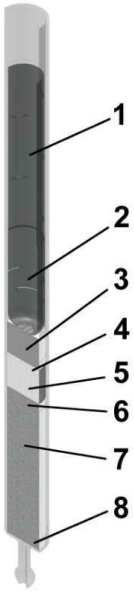


Figure 16: Components within the combustion tube: 1) Protective tube. 2) Ash finger. 3) Quartz chips, coarse (15 mm). 4) Quartz wool (5 mm). 5) Platinum catalyst (25 mm). 6) Quartz wool (5 mm). 7) Quartz chips, coarse (85 mm). 8) Quartz wool (5 mm). Image by: Elementar Analysensysteme GmbH.

3.10.6 Lay the combustion tube flat on a level surface. Place a ruler parallel to the tube, starting at the base (Figure 17). Using a Sharpie®, draw lines at the following marks: 0.5 cm, 9 cm, 9.5 cm, 11 cm, 11.5 cm, 13 cm.

Figure 17: The proper placement of the ruler when measuring the placement of fill lines to be drawn.

3.10.7 Place the combustion tube in the holder provided by Elementar. Fill the combustion tube such that it matches Figure 16 above and Figure 18.

3.10.7.1 Place quartz wool at the bottom of the combustion tube. Use the compression tool to compress the quartz wool, such that it reaches the 0.5 cm mark.

3.10.7.2 Pour the coarse quartz chips into the tube until it is level with the 9 cm mark.

3.10.7.3 Place quartz wool on top of the quartz chips. Compress the quartz wool such that it reaches the 9.5 cm mark.

3.10.7.4 Place the platinum catalyst on top of the quartz wool until it reaches the 11 cm mark.

3.10.7.5 Place quartz wool on top of the platinum catalyst. Compress such that the wool reaches the 11.5 cm mark.

3.10.7.6 Pour the coarse quartz chips on top of the quartz wool until it is level with the 13 cm mark.

3.10.7.7 Place the ash finger on top of the quartz chips.

3.10.7.8 Place the protective tube on top of the ash finger.

3.10.8 Place the filled combustion tube back into the furnace area. Clamp to the glass tubing at the base of the tube.

3.10.9 Follow steps above from “3.9. Procedures for Replacing the Ash Finger” to refit the multiway valve, cover plate, and carousel.

3.10.10 Set furnace to 850 °C before resuming water analysis.

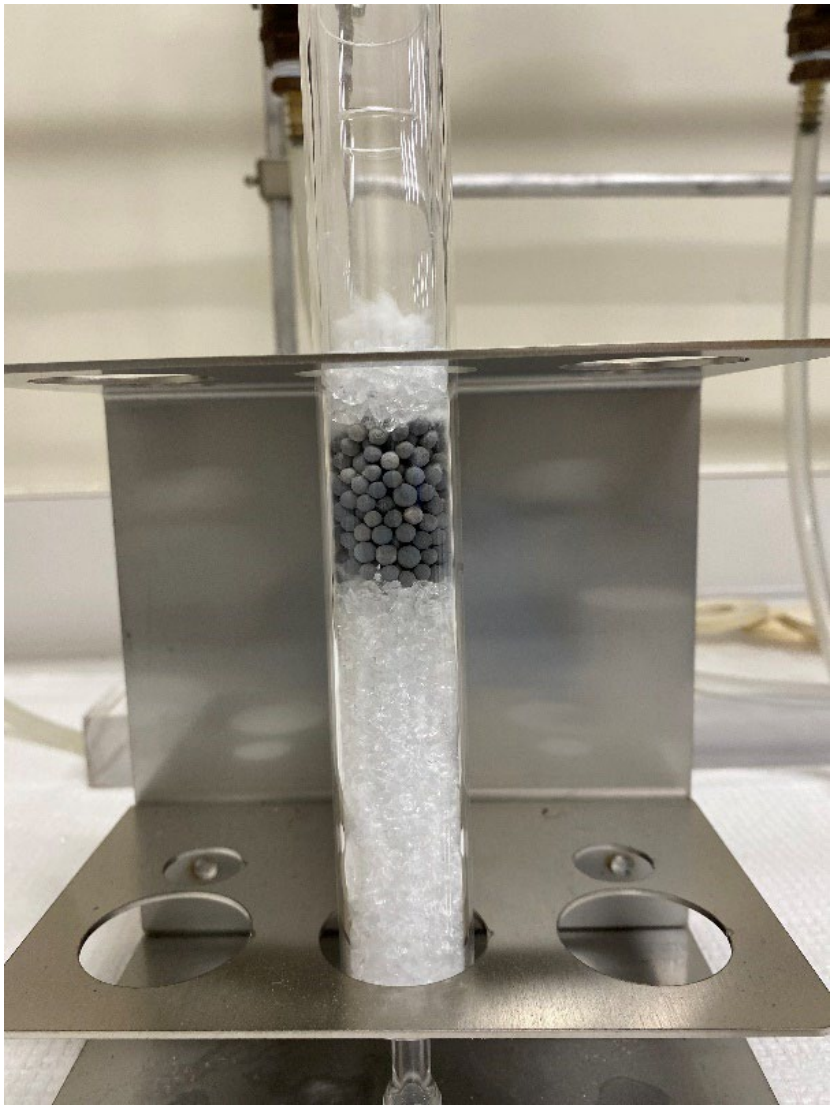


Figure 18: The filling of the combustion tube has been completed, and it is ready to be placed into the TOC Vario for use.

3.11 Filling the Drying Tube

3.11.1 Caution: the drying agent, magnesium perchlorate, is a caustic chemical. Be sure to wear gloves when handling. Set the TOC Vario to parts replacement mode before proceeding.

3.11.2 Follow the below steps of replacing the drying agent after the analysis of every 4000 samples.

3.11.3 Open the front of the TOC Vario. Unscrew the red sealing caps on both side of the drying tube. Remove the drying tube from its clamps. The filter pad should hold contents in place but be careful not to spill them (Figure 19).

3.11.4 Use a pair of forceps to remove the filter pad from the top of the drying tube. Pour the used drying agent into the solid waste bottle stored under the fume hood.

3.11.5 Pour the fresh drying agent into the drying tube. Fill to the top, such that there are no gaps. Place the filter pad back at the top of the tube.

3.11.6 Snap the drying tube back into the clamps. Tightly seal the tube with the red sealing caps.

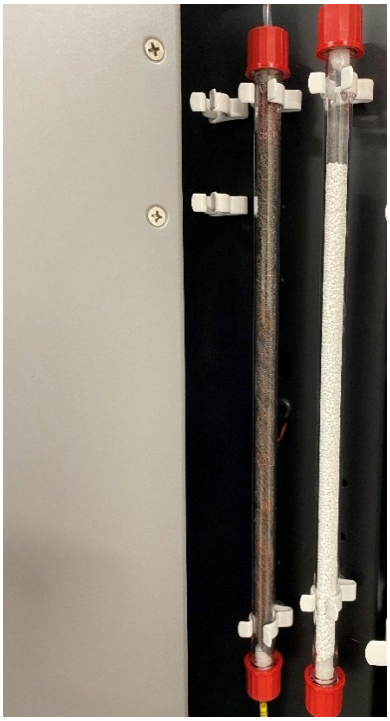


Figure 19: The absorption tube (left) and the drying tube (right). The brass wool in the absorption tube has dullened to a significant degree, indicating that replacement is required.

3.12 Filling the Absorption Tube

3.12.1 The absorption tube contains brass wool, which protects the IR sensor by removing halogens. Refill the absorption tube after the analysis of every 4000 samples, or when the brass wool has dullened to a significant degree. Set the TOC Vario to part replacement mode before proceeding.

3.12.2 Open the front of the TOC Vario. Unscrew the red sealing caps on both sides of the absorption tube. Remove the tube from its clamps (Figure 19).

3.12.3 Use a pair of forceps to remove the filter pads from the top of the absorption tube. Remove and dispose of the used brass wool.

3.12.4 Pack new brass wool tightly into the absorption tube, without leaving any gaps (Figure 20). Plug both sides of the tube with the filter pad. Reinstall the absorption tube.

Figure 20: Packing fresh brass wool into the absorption tube.

4.0 REFERENCES

Operating instructions: TOC Vario cube and TOC Vario select. Version 27.08.2018 (2018). Elementar Analysensysteme GmbH.

5.0 APPENDIX

For any procedures not outlined in this SOP, please refer to the Elementar TOC manual. Contact service-us@elementar.com for any questions related to maintenance, or to schedule a technician to service the TOC Vario, on-site.

Safety Information:

- Wear gloves for personal protection and to prevent sample contamination. Wear closed toe shoes when conducting TOC analysis. Wear safety glasses with side shields or goggles for eye protection, especially when using HCl.
- When preparing acid solutions, work in the fume hood with the ventilation turned on.
- Wear a face shield, as well as the provided heatproof gloves, when performing maintenance in or above the combustion tube while the furnace is on.
- Use a heatproof surface to place hot glassware during parts replacement (i.e., ash finger and protective tube).
- Do not dispose of hot glassware until it has cooled completely.
- Ensure the air tanks are secured to the wall.
- Do not remove the cover plate or open the front of the TOC Vario during calibration or sample analysis.
- Keep hands away from moving components (e.g., syringe injector).
- Dispose of any wastes in the designated waste containers.
- Ensure that the drying agent, magnesium perchlorate, does not contact skin. Wear gloves when handling the drying agent.