

CN Analyzer Operating Instructions for Liquid Analysis
Version 2.2
1/13/14

Please note: Total carbon (TC) and total organic carbon (NPOC) analyses can be performed in the same sequence, but separate calibration and sample vials are needed for each analysis.

- 1) Press the “on” button on the front of the main instrument. Open the valve for the air tank. If TN analysis is desired, open the valve for the oxygen tank and flip on the switch on the right side of the TN unit.
- 2) Check that the solids/liquids toggle switch is set properly. This switch is located just under the top lid, in the back of the instrument. The line in front is for liquids and the one in back is for solids (lines should be labeled but the labels sometimes fall off).
- 3) Open the front of the instrument and check that the humidifier water level is between “high” and “low” and the water level in the coolant drain container is about 1 cm below the drain arm. Refill these with de-ionized water if necessary.
- 4) Check and re-fill (if necessary) the e-pure water reservoir for the autosampler and the e-pure water, HCl, and IC reagent reservoirs for the CN analyzer. The HCl bottle contains 2 M HCl, while the IC bottle contains diluted phosphoric acid (prepared by diluting 50 mL of Reagent grade 85 % phosphoric acid to a final volume of 250 mL with e-pure water).
- 5) Prepare calibration standards. For ASTM D5176-08, these should be made from pyridine.
- 6) Start the TOC-Control V software and open the Sample Table Editor. Enter user name if desired or just click *OK*. Choose *File*→*New*→*Sample Table*.
- 7) On the “Select H/W Settings” pop-up window select “liquid”.
- 8) Click *File*→*Save As* and enter the desired sample table name.
- 9) Select the *Connect* button. This procedure establishes communication between the software and the instrument.
- 10) From the sample table, click *File*→*New*→*Calibration Curve*. (Calibration curve wizard page 1) Select the desired system. Click *Next*.
- 11) (Calibration curve wizard page 2) Choose the desired calibration curve. This is usually *Edit Calibration Points Manually* coupled with *Dilution from Standard Solution*. Click *Next*.
- 12) (Calibration curve wizard page 3) Fill in the requested information, including the filename for the calibration curve. Generally the *Zero Shift* box is left unchecked. Check the *Multiple Injections* checkbox. Click *Next*.
- 13) (Calibration curve wizard page 4) Fill in the requested information. Click *Next*.
- 14) (Calibration curve wizard page 5) Click on a row and select *Add*. For the blank, enter a standard concentration of 0 mg/L and a dilution factor of 1. Hit *OK*. Add additional rows for the standards to be analyzed, keeping the dilution factor of 1.

- Alternatively, calibration points can be created with fewer standards or a single standard by entering the actual standard concentration, using a higher dilution factor (e.g. 10-15), and then using the same standard in subsequent rows with varying lower dilution factors. Injection Volume is 50 ul. Click *Next*.
- 15) (Calibration curve wizard page 6) Keep the default values on this page. Click *Finish*.
 - 16) Repeat steps 10-14 if another calibration curve is needed for additional analyses.
 - 17) Highlight the first line of the sample table. Select *Insert*→*Calibration Curve*. Specify the name of the desired calibration curve and click *Open*. Click *View*→*Vial Settings* or select the “View Vial Settings” icon located in the upper right of the screen. Type the position of each vial into the appropriate row, or alternatively place the cursor in the appropriate row and double-click the desired vial position on the autosampler diagram. Click *OK*. Place the vial(s) into the appropriate position(s) in the vial rack.
 - 18) Highlight the second row of the sample table and enter the second calibration curve using the same method as in step 17 above. Separate vials of standard are recommended if using two calibration curves to ensure sufficient quantities for analysis.
 - 19) Save the sample table.
 - 20) From the sample table, select *File*→*New*→*Method*. The method specifies the parameters to run unknown samples.
 - 21) (Method wizard page 1) Select “liquid” as the system to be used for the analysis. Click *Next*.
 - 22) (Method wizard page 2) If analyzing both N and C in the samples, select either “TC/TN” or “NPOC/TN” from the drop-down menu, otherwise choose “TC”, “NPOC”, or “TN”. Enter the rest of the requested information. The method file name must end in “.met”. Click *Next*.
 - 23) (Method wizard page 3) Enter the requested calibration curve into the method by browsing with the “...” button. Click *Next*.
 - 24) (Method wizard page 4) Change values if desired. Check the “Multiple Injections” and “Auto correction of inj vol and dilution” boxes, then click *Next*.
 - 25) (Method wizard page 5) Use default settings, then click *Next*.
 - 26) If both C and N will be analyzed with this method, the wizard goes back to page 3 for the calibration curve that was not entered previously to be entered now. Enter it and click *Next*.
 - 27) (Method wizard pages 4 and 5) Set parameters according to steps 23 and 24 above. Click *Finish*.
 - 28) Insert the first unknown sample into the sample table by highlighting the next empty line of the sample table. Select *Insert*→*Sample*. Insert the proper method file. Click the check box next to *Skip remaining wizard pages and use measurement parameters from the source* if the sample is to be measured exactly according to the method. If changes are desired, click through the various screens, make the desired changes, then click *Finish*. Usually the default sample name and/or default sample ID will need to be changed.
 - 29) Click *View*→*Vial Settings* or select the “View Vial Settings” icon located in the upper right of the screen. Type the position of the vial into the appropriate row,

- or alternatively place the cursor in the appropriate row and double-click the desired vial position on the autosampler diagram. Click *OK*. Place the vial into the appropriate position in the vial rack.
- 30) If using the same method for all unknown samples, enter the rest of the unknown samples according to steps 28-29 above. If multiple methods need to be created for unknown samples, repeat steps 20-27 to create new methods, then steps 28-29 for new unknown samples. Double-check sample positions in the vial rack when finished. Replace the autosampler cover. Save the sample table.
 - 31) Make sure the green “Ready” button is displayed in the upper right corner before starting analyses. If the red “Not ready” button is displayed, check the status of the instrument by clicking the *Monitor* button. Check both the TOC and TN tabs if running both analyses. Close the background monitor window when finished because it cannot be open during analysis.
 - 32) Place the cursor in the first row of the sample table and click the *Start* button or select *Instrument*→*Start Measurement*. The *Measurement Start* window appears. Choose “shut down instrument” and click *Start*, unless the instrument will be run again the same day, in which “keep running” followed by *Start* should be selected. Make sure to select “shut down instrument” in the *Measurement Start* window when starting the last sequence of the day.
 - 33) If desired, the measurements can be monitored during analysis by viewing the sample window. To open the sample window, click the icon or select *View*→*Sample Window*. The current injection is displayed with asterisks in the injection table. The drop-down list above the graph is used to set the graph display. Blue peaks are carbon and red peaks are nitrogen.
 - 34) To check the calibration curve results, place the cursor in the row of the sample table that contains analysis information for the calibration. Click the icon or select *View*→*Calibration Curve*. Select “cal curve” from the drop-down menu to view the data in graphical form. Highlight a row and hit *Exclude* to exclude any points. An “E” will be displayed in the row of the excluded point. A prompt will be displayed to recalculate the data; click *Yes*. To include a previously excluded point, highlight the row of the excluded point and hit *Exclude*. The data will need to be recalculated again.
 - 35) To check peak profiles place the cursor in the desired row of the sample table and click the sample icon or select *View*→*Sample Window*. Use the drop-down list at the top of the graph to select the peak profiles to view.
 - 36) The sample results are displayed in the sample table. They can also be viewed individually by placing the cursor in the desired row of the sample table and selecting *View*→*Sample Window*.
 - 37) To export the data to Excel, first select *File*→*ASCII Export Setting* to determine the information to be exported. Once this is done, select *File*→*ASCII Export*, select “Normal” or “Detail”, and save the data as a text file. Open the text file in Excel using the “delimited” option in the “Text Import Wizard” window, and save it in Excel format.
 - 38) When all analyses are complete, the instrument should shut off automatically after about 30 minutes if “shut down instrument” was properly selected in the *Measurement Start* window. If for some reason this wasn’t done, select the

Shutdown button, then select “shut down instrument” and *OK*. **VERY IMPORTANT:** close the valves on the compressed gas cylinders when all analyses have been completed.