CN Analyzer Operating Instructions for Solid Analysis
Version 1.0
1/16/08

1) Open the valves for the oxygen and air tanks. Press the "on" button on the front of the main instrument (the TN unit should come on as well). Turn on the SSM unit by flipping the switch, located on the right side of the unit near the back.
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 removable panel on the right side of the SSM. Check the water level in the vessel. The level should be just below the drain outlet. Top up the vessel with e-pure water, if necessary. If performing IC analyses, check the volume of the acid dispenser and top up (if necessary) with phosphoric acid that has been diluted with two parts water.
4) Start the TOC-Control V software and open the Sample Table Editor. Hit "Enter" when prompted for username. Choose *File→New→Sample Run*.
5) Click the "System" tab and select the desired system ("SSM"or "TC/TN solids") from the drop-down list.
6) Click *File→Save As* and enter the desired sample table name.
7) Select *Instrument→Connect* (or click the lightning bolt toolbar button). In the *Parameter Configuration* dialog box, click *Use Settings on PC*. This procedure establishes communication between the software and the instrument.
8) Select *Options→Default Measurement Parameters*. Select the units to be used for sample measurement. Set the number of injections to 1.
9) Select *Options→Display Settings→Table Settings*. Click *Select All*. Re-save the file.
10) From the sample table, click *File→New→Calibration Curve→OK*. (Calibration curve wizard page 1) Select the desired system ("SSM" or "TC/TN solids"), then click *Next*.
11) (Calibration curve wizard page 2) Choose the desired calibration curve. This is usually *Edit Calibration Points Manually*. Click *Next*.
12) (Calibration curve wizard page 3) Select analysis type, either SSM-TC for total carbon, SSM-IC for inorganic carbon, or TN for total nitrogen. Set the rest of the requested information, including the filename for the calibration curve. Select *Zero Shift* (if desired) to force the calibration curve through the origin. 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*. If running a blank, enter a standard value of 0. Hit *OK*. Add additional rows for the standards to be analyzed and fill in the requested information. Click *Next*.
15) (Calibration curve wizard page 6) Keep the default values on this page. Click *Next*.
16) (Calibration curve wizard page 7) Keep the default values on this page. Click *Finish*.
17) Place the cursor on the first line of the sample table. Select *Insert→Calibration Curve*. Specify the name of the calibration curve and click *Open*.
18) If the samples will be analyzed using the same parameters as the calibration curve, skip to step 26.

19) From the sample table, select *File→New→Method→OK*. The method specifies the parameters to run unknown samples.
20) (Method wizard page 1) Select the system to be used for the analysis ("SSM" or "TC/TN solids"). Click *Next*.
21) (Method wizard page 2) Enter the requested information. The method file name must end in ".met". Click *Next*.
22) (Method wizard page 3) Enter a value in the Expected Concentration Range box if the expected sample concentration is known (if applicable). Enter the requested calibration curve into the method by browsing with the "…" button. Click *Next*.
23) (Method wizard page 4) (if applicable) Enter a value in the Expected Concentration Range box if the expected sample concentration is known. Enter other requested information. Click *Next.*
24) (Method wizard page 5) Change values if desired, then click *Next*.
25) (Method wizard page 7) Click *Finish*.
26) Place the cursor on the next empty line of the sample table.
    a) To set the parameters to measure a single sample, select *Insert→Sample*. Set the source of the measurement parameters (either "method" or "calibration curve") and browse for the desired 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 using parameters from the source file (i.e., either the method file or calibration curve file). If changes are desired, click through the various screens, make the desired changes, then click *Finish*.
    b) To measure multiple samples with the same parameters, select *Insert→Auto Generate*. (Page 1) Set the source of the measurement parameters (either "method" or "calibration curve") and browse for the desired file. (Page 2) Enter the number of samples and other requested information. (Page 3) Enter desired calibration curves (if any) and click appropriate check boxes. (Page 4) Enter desired controls (if any) and click appropriate check boxes, then click *Finish.*
27) The SSM needs approximately 45 minutes to heat up and stabilize the furnace temperatures (900C for TC and 200C for IC) before running analyses. This is a good time to weigh the samples and/or standards into sample boats if it has not been previously done (unless measuring liquid samples). Use gloves when handling sample boats to minimize contamination.
28) Place the cursor in the first row of the sample table (the calibration curve file) and click the *Start* icon or select *Instrument→Start*. The "TOC measurement dialog box" is displayed. Click *Start*.
29) The "Enter Sample Amount" dialog box is displayed. Enter the sample amount.
30) Open the appropriate sample boat cover ("TC" or "IC") and place the proper sample boat on the sample boat transfer plate with long forceps. Securely close the sample boat cover.
    a) For carbon analysis, if the carbon content of the sample is 3 mg or less, wait 2 minutes after closing the sample boat cover before pressing *Start* to avoid inclusion of an atmospheric CO_2 peak. Otherwise, hit *Start* immediately.
    b) For nitrogen analysis, push the sample into measuring position, wait 1 minute, then hit *Start*. Skip to step 32.

31) The message "Push the sample boat to the measurement position" is displayed on the screen.
    a) For TC analysis, gently push the sample boat transfer knob all the way to the back (the "Measuring" position).
    b) For IC analysis, pump the IC acid injector to inject phosphoric acid into the sample boat, and push the sample boat transfer knob all the way to the back (the "Measuring" position).
32) 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.
33) When sample measurement is completed,
    a) the message "Pull the sample boat back to the COOLING position" is displayed for TC and TN analysis. Pull the knob to the COOLING position.
    b) the message "Pull the sample boat back to the PREPARATION position" is displayed for IC analysis. Pull the knob to the CHANGE position and skip to step 35.
34) For TC and TN analysis, the sample boat transfer rod is cooled while in the COOLING position. After 30 seconds have elapsed, the message "Pull the sample boat back to the PREPARATION position" is displayed. Pull the knob to the CHANGE position.
35) The "TOC Measurement" dialog box is displayed. Click *Repeat* to perform additional measurements with the same sample, or click *Next* to proceed to the next sample. Click *Stop* to finish measurement.
    a) To reject the measurement data, highlight the data to be excluded and click *Exclude*.
    b) Occasionally, IC acid accumulates on the IC sample boat holder. Inspect and clean the sample boat holder, if necessary, during sample changes.
36) For sample analysis, repeat steps 29-35 as necessary until all samples have been analyzed.
37) 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 the graph tab to view the data in graphical form. Select the data tab to view calibration statistics such as standard deviation and coefficient of variation. 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.
38) 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.
39) 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*.

40) To export the data to Excel, first select *File→ASCII Export Options* to determine the information to be exported. Once this is done, select *File→ASCII Export* 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.
41) When all analyses are complete, select *Instrument→Standby*, then select "shutdown instrument" and *Standby*. This turns off power to the electric furnace and stops the carrier gas flow. Do not turn off the main power switch on the SSM unit until 30 minutes have elapsed to allow the cooling fan to run inside the instrument. The power to the TOC-V unit will turn off automatically after 30 minutes. VERY IMPORTANT: close the valves on the compressed gas cylinders when all analyses have been completed.
    a) Flush the phosphoric acid delivery line with water if the IC portion of the unit will not be used for an extended period of time.
    b) Use a cotton swab to remove acid that may have come in contact with components inside the sample port, including the sample boat holder and sample boat push rod. Rinse off any especially corrosive substances with water.