Introduction
The Shimadzu TOC-V_CP/CPN (TOC) instrument measures the total amount of carbon (TC), inorganic carbon (IC), the total amount of organic carbon (TOC) and the total amount of non-purgeable organic carbon (NPOC). The TOC works on the principle of oxidative combustion infrared (IR) analysis. The carbon samples are combusted to form CO$_2$(g) and analyzed in a non-destructive infrared analyzer (NDIR). The TOC can measure total nitrogen (TN) in water samples using a chemiluminescence analysis of N$_2$(g). The limit of quantification (LOQ) for TOC for the TRACES Centre system is set from 200 ppm down to around 500 ppb. The limit of quantification (LOQ) for TN for the TRACES Centre system is set from 50 ppm down to around 1 ppm.

Purpose
The purpose of this document is to introduce the user to the routine operation of the Shimadzu TOC-TN. This document also outlines the special safety concerns introduced when using the instrument.

Scope
This procedure applies to established procedures of analysis using the standard configuration (NPOC/TN) for the TOC. Individuals must complete a hands-on one-on-one training session with a TRACES staff member. Other fields are measure are covered in the hands-on training if requested.

Referenced Documents
- Shimadzu TOC-VCPH/CPN & TOC- Control V Software (p/n 638-94241A)
- Shimadzu TOC Application Handbook (p/n G3430-90015)
- TOV Virtual Advisor: https://tocvva.ssi.shimadzu.com/home
- Shimadzu Model TOC-5000 Instruction Manual
- USP 643: Total Organic Carbon Determination

Responsibilities
1. TRACES Users
   1.1. All Users must obtain training with TRACES Staff prior to system operation. It is the responsibility of the User to ensure they have a good understanding of the instrument and all operation protocols.
   1.2. If additional training sessions are needed it is the responsibility of the User to schedule these with TRACES Staff.
   1.3. User must ensure that the compressed air cylinder required for impending analysis is available in TRACES*.
   1.4. Instrument time must be booked by the User via the online booking system prior to system operation.
2. **TRACES Staff**
   2.1. It is the responsibility of TRACES Staff to provide introductory and, if required, subsequent training to all users.

**Equipment**

- Shimadzu TOC-V
- Shimadzu TN
- Shimadzu ASI-V

**Safety**

**IMPORTANT:**
- The TOC uses very high temperatures.
- The compressed air cylinder uses high pressure gas.
  - Take the appropriate precautions
- Seek assistance from TRACES Staff if you have any concerns

**Initial Setup**

1. Ensure the compressed air contains MORE than 500 psi in the cylinder. If you expect a long run time contact the TRACES Lab Manager
   a. Turn the regulators to ‘OPEN’
   b. The compressed air delivery pressure should read between 45-55 psi
2. Fill the Cooler Drain Container (Fig.1) with deionized water by removing the rubber cap
   a. Using deionized water fill the container till it is visibly draining into the waste container
   b. Recap the drain
3. Fill the Humidifier Container (Fig.2) with deionized water by removing the rubber stopper
   a. Fill above the ‘Hi’ level
   b. Re-stopper the container
Standard Operating Procedure

4. Ensure that RINSE (to the left of the autosampler) and DILUTION Bottles (between autosampler and TOC) are topped up to the 2L mark with deionized water.
5. Check the ACID BOTTLE located between ASI-V and TOC-V. Ensure the acid tubing is near the bottom of the acid bottle and that the bottle is topped with 2M HCl.

TOC Initial Setup Section

1. Press the Power button located on the bottom right-hand corner of the TOC unit
2. Log on to computer using the username and password provided
3. Select the ‘TOC Control V’ icon on the desktop
   a. Provide a username
   b. From the opened window click the ‘Sample Table Editor’ and press OK
   c. Label the sample table accordingly and press “Save” and then OK,
      i. Select a filename with a date representation (i.e., Apr5_2014_Lake Water13) or the use of the generated filename
      ii. Save the Sample Table in the appropriate directory
      iii. A new blank table will now be displayed
4. Connect to the instrument via the instrument menu & clicking on the toolbar icon
   a. Select ‘Instrument Settings on PC’
   b. Wait to ensure that connection to the instrument has been established
      i. No error message is displayed
5. The TOC Carrier gas flow rate should read 150mL/min
   a. Carrier gas should not leak (bubble) from the top of the drain tubing
   b. Instrument will begin to run the startup procedure
6. The TOC Carrier Gas Pressure Regulator should read 200KPa

ALLOW 60 MINUTES OF WARM-UP TIME

TOC Calibration Table Setup

1. The TOC software is able to monitor various calibration curves (MAXIMUM of THREE) to apply to your sample. It would be advantages at this point to setup several high concentration and low concentration calibration curves to obtain a more accurate results for your unknowns
2. Open a New ‘Sample Table’ from the File menu
3. Click on the Calibration Curve icon in the Sample Table window and press ‘OK’
4. Calibration Curve Wizard will commence
   a. Select TOC-Vcsh, ASI, TNM-1 or TOC, TN (->next)
   b. Select Manual Calibration
   c. Select Dilution from Std Solution- for external dilution (->next)
   d. Select NPOC, TC, IC, TN. Below is default for NPOC analysis:
      i. Analysis: NPOC
      ii. Default Sample Name:
      iii. Default Sample ID:
      iv. Calculation Method: Linear Regression
v. Check box for Zero Shift: Leave blank
vi. Check box for Multiple Injections: select
vii. Calibration File Name: MUST BE COMPLETED (->next)
e. Select appropriate Calibration Parameters. Below is default for NPOC analysis:
   i. Units mg/L
   ii. Number of injections: Min=2 Max=3
   iii. Number of washes 2
   iv. SD Max 0.1000
   v. CV Max 2.0%
   vi. 1:30min for sparing time & 0.5% Acid Addition (->next)

f. Calibration Points List must start with highest calibrant. Follow step below:
   i. Add calibrants starting with highest concentration
   ii. Enter Standard Solution concentrations
   iii. Select ‘Auto Dilution Factor’
   iv. Number of injections: Min=2 Max=3
   v. SD Max 0.1000
   vi. CV Max 2.0%
   vii. Click ‘OK’

g. Peak Time Parameters – change as appropriate
h. Do NOT enable ‘History Log’
i. Click ‘Finish’

5. Inserting Calibration Standard
   a. Highlight/cursor position on first line of the Sample Table
   b. Open ‘Insert Menu’ and select the ‘Calibration File’ (from above)
   c. Sparging/Acid Addition window is available
      i. Set vial positions (Vial 0 uses TOC Water as Blank)
      ii. OK
   d. From the ‘Instrument’ Menu select ‘Start’
   e. Standby Window will appear
      i. Ensure that the vial numbers and concentrations match
      ii. Selection of Post-Analysis Calculations
      iii. Select Standby
      iv. OK

6. Calibration analysis will commence at this point
7. Once the calibration has been ‘COMPLETED’
   a. Select the calibration curve icon
   b. Minimum correlation coefficient for this instrument is 0.995 for TOC and 0.99 for TN

Note: The greater the accuracy of the standard concentration the greater the accuracy of your measurement. I advise all users to calculate concentrations using weight (analytical balance (4 decimal places)).
TOC Method Setup

1. Open a New Sample Table from the File menu
2. Click on the Method icon in the Sample Table window and press ‘OK’
3. Method Wizard will commence
   a. Select NPOC, IC, NPOC/TN. Below is default for NPOC analysis:
      i. Analysis: NPOC
      ii. Default Sample Name& ID:
      iii. Default Values: Determinations
      iv. Default Values: Dilution
      v. Method Name: (->next)
   b. Select appropriate calibration curve (max. of three)
   c. ) Select appropriate Sample Injection Parameters. Below is default for NPOC analysis:
      i. Units: mg/L
      ii. Number of injections: Min=2 Max=3
      iii. Number of washes: 2
      iv. Autodilutions: 1
      v. SD Max: 0.1000
      vi. CV Max: 2.0%
      vii. 5:00 min for sparing time & 0.5% Acid Addition (->next)
      viii. Shimadzu recommends not enabling any of the remaining parameters (-> next)

TOC Sample Analysis Setup

1. Select AutoGenerate from Insert Menu
2. Select the appropriate Method (->next)
3. Enter the number of samples to be run, vial locations and ID the samples (->next)
4. Select the appropriate calibration curve (several can be chosen) (->next)
   a. In most cases the previously run calibration method is chosen
5. Controls can be designated in the Calibration Curve Check section (->next)
6. Shimadzu recommends not changing or enabling any of the remaining parameters unless there is a mitigating reason for it (-> next)
7. Measurement Start (review all locations and values).
   a. At this point you can select to;
      i. Keep running (if you wish to run a further sample set)
      ii. Shutdown (after sample analysis is completed)
TOC Data Evaluation

1. TOC data will appear on ‘Sample Table’ once the analysis is completed
2. If data manipulation is required, it is STRONGLY urged to save the existing data with a different filename to avoid the loss of the original data set
3. All ‘Print’ functions will provide a PDF copy of the desired results
   a. No physical printers are available
   b. Data can be transferred via email or ftp servers
4. Print the ‘Table’
   a. Printing of the sample name and concentration values of the sample
5. Print the ‘Sample Report’
   a. Printing of the sample data, area, mV response of each injection analysis for all the samples and calibration data located in the ‘Sample Table’
6. ‘Ascii Export’
   a. ‘Export Sample Table and Each Injection’ using common CSV data output
   b. ‘Export by Analysis Type’ segregates each analysis (TOC, NPOC, TN..) separately using Common CSV data output

*The TRACES Manager will provide further details during hands-on training.*