Latulippe Research Group

Shimadzu TOC-L Analyzer Manual

1. Purpose

This manual is prepared to help the users of the Shimadzu TOC-L analyzer instrument (located in JHE 364). Its purpose is to measure the concentration of carbon in material samples. However, each user should be trained by one of the qualified individuals in the Latulippe Lab and this manual should not be used to replace the in-person training. This manual should used as a reference guide only.

2. Introduction & Background

The Shimadzu TOC-L Analyzer uses a combustion catalytic oxidation method which achieves total combustion of samples in an oxygen-rich environment. It has the capacity to effectively oxidize organic compounds, including insoluble and macromolecular organic compounds. It can take a variety of measurements including TC (Total Carbon), IC (Inorganic Carbon), TOC (Total Organic Carbon) and non-purgeable organic carbon (NPOC).

TC Measurement - The sample undergoes combustion by heating to 680 °C with a platinum catalyst and is fed by purified air. The sample decomposes and is converted to carbon dioxide, which is cooled and dehumidified. This carbon dioxide is then detected using a Non-Dispersive Infra-Red detector (NDIR). The NDIR operates by shining an infra-red beam through a sample cell containing CO_2 and measuring the amount of infra-red absorbed by the sample at the necessary wavelength. The NDIR has a detection limit of 4 μg/L. TC concentration is obtained by the comparison to a calibration curve.

IC Measurement – The oxidized sample from the TC process is subjected to sparging, where carbon dioxide is isolated at a low pH (less than 3) requiring a supply of acid. The carbon dioxide, which is from the inorganic carbon of the sample, is cooled and dehumidified then measured by the NDIR.

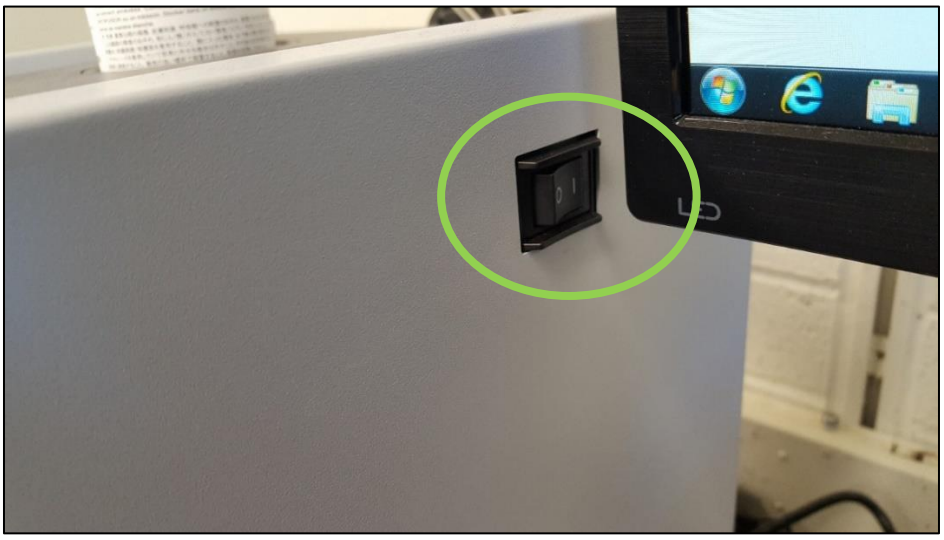
TOC Measurement – The TOC concentration is calculated by subtracting the IC concentration from the TC concentration, described above (TOC = TC - IC).

NPOC Measurement – Some samples contain a high inorganic carbon component and there may be significant error if the TOC method is used. Thus, the NPOC method is used, where the sample undergoes sparging (low pH, < 3) to remove the carbon dioxide from the inorganic carbon component first, and then the TOC is measured in the remaining sample and measured by the NDIR. This TOC could not be “purged” by the sparging stage and is thus called non-purgeable organic carbon (NPOC).

3. User Guide

3.1 Instrument Start-Up

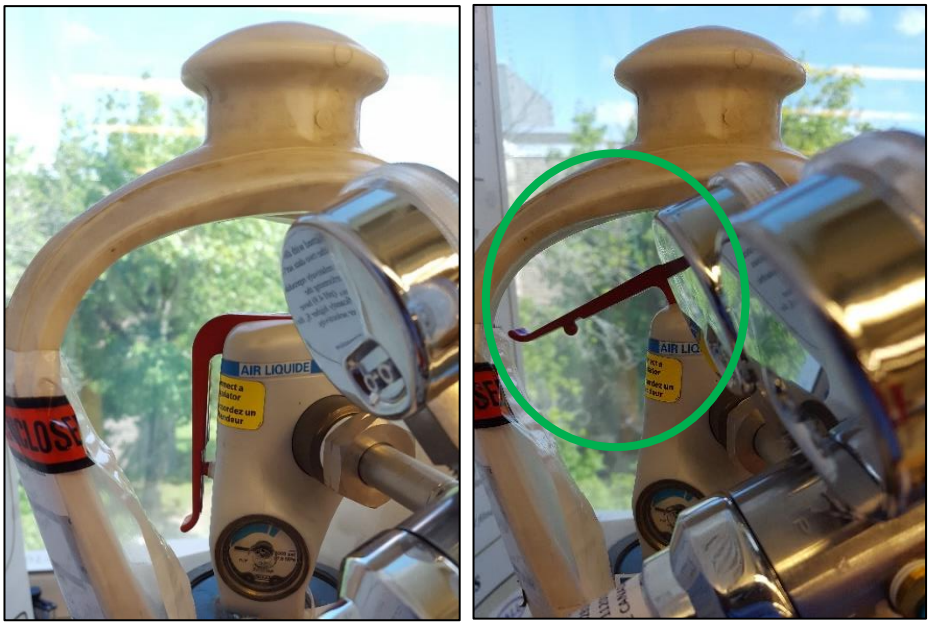
1. Firstly, ensure make sure that the main power switch is on the ‘on’ position. In most cases this switch is left on. This switch is located at the upper right corner of the right side of the instrument next to the computer screen.

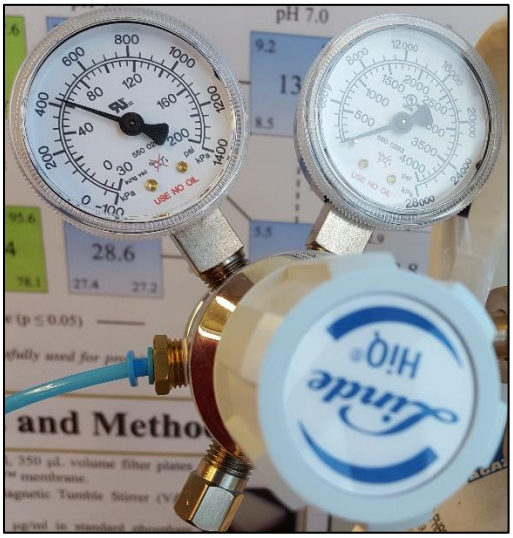


2. Next, press the power button to turn the instrument on. This button is located at the front panel of the instrument. The instrument needs around 15 minutes for its furnace to reach the appropriate temperature for running a sample. Plan experiments ahead of time to account for instrument start-up.



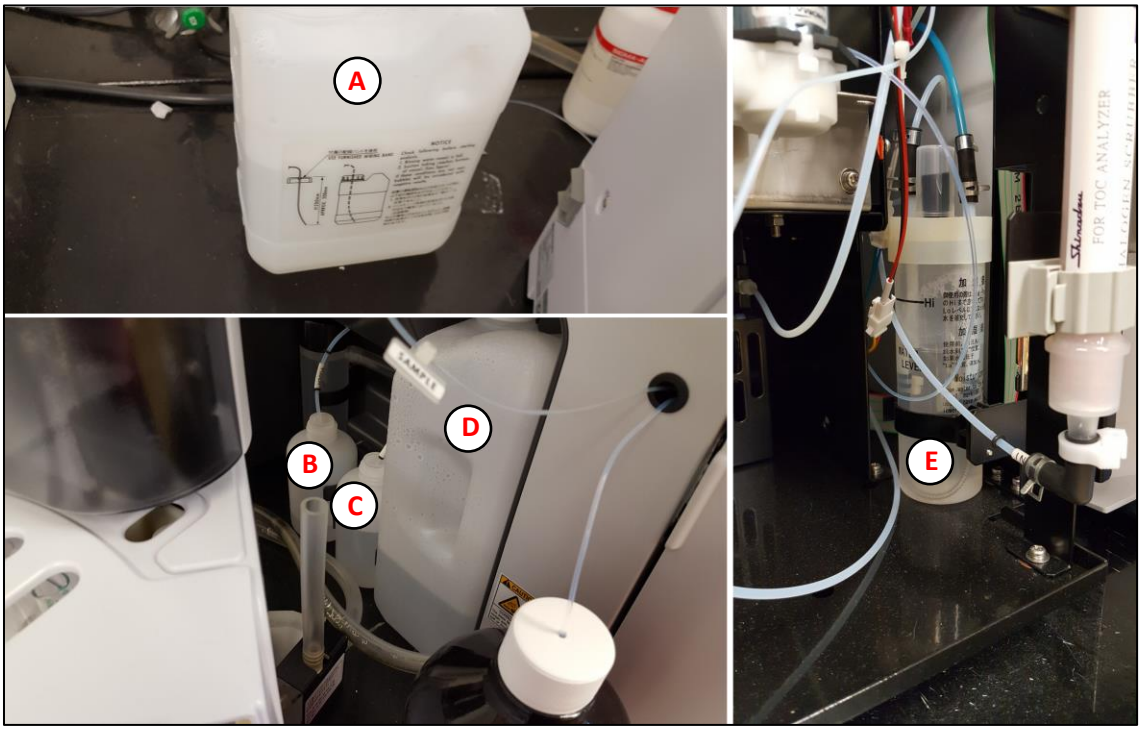
3. Open the main valve of the carrier gas (air). It is the best to open this valve after turning on the instrument and close it after turning off the instrument. Below, open (right-hand-side) and closed (left-hand-side) positions of the valve are shown. You can also read the pressure from the pressure gauge on the regulator which should be around 60 psi. Please note that all individuals should have "Gas Cylinder" training completed in MOSAIC to work with the cylinder.



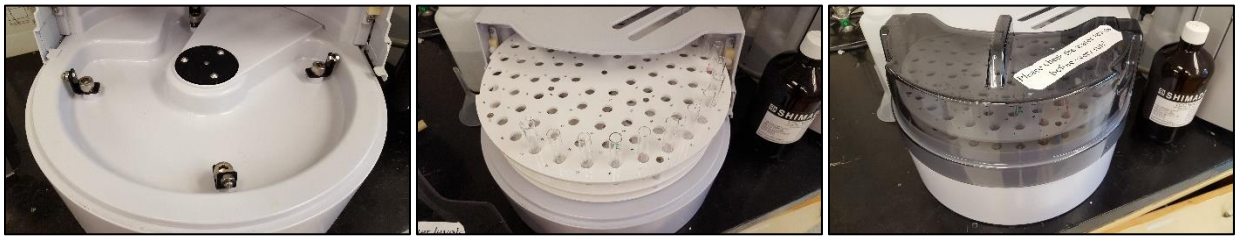


4. Before performing each run, make sure to check the liquid levels in the reagent containers that supply the TOC, listed below. In all cases, only Milli-Q water should be used to fill the containers. Tap water and DI water from the DI tap in the lab should be under no circumstances. If you are unfamiliar with using the instrument or with filling the container, seek aid from other senior students who work with the system regularly.

a) The water level used for the washing water (the container is behind the auto-sampler)
b) The level of H_3PO_4 and HCl solutions (the containers are under the TOC analyzer and accessible from the left-hand side). The acid is available on the lab bench in Room 364 and is supplied by Caledon Laboratory Chemicals. Acid solutions should be diluted to 25 % by volume using Milli-Q water.
c) The water level of the auto-dilution water (the container is placed under the instrument close to the acid containers)
d) The water level inside the dehumidifier (inside the instrument)
e) The water level of the container placed on the left side of the instrument, near the back.



5. To place your samples, either use 9 mL or 40 mL TOC vials with the corresponding vial tray. The picture below shows (from left to right) the auto-sampler without the vial tray, with the 9 mL vial tray and with the ASI cover engaged. **The ASI cover should be used always to run the TOC otherwise the instrument will stop running the sample immediately**.



3.2 Software Guide

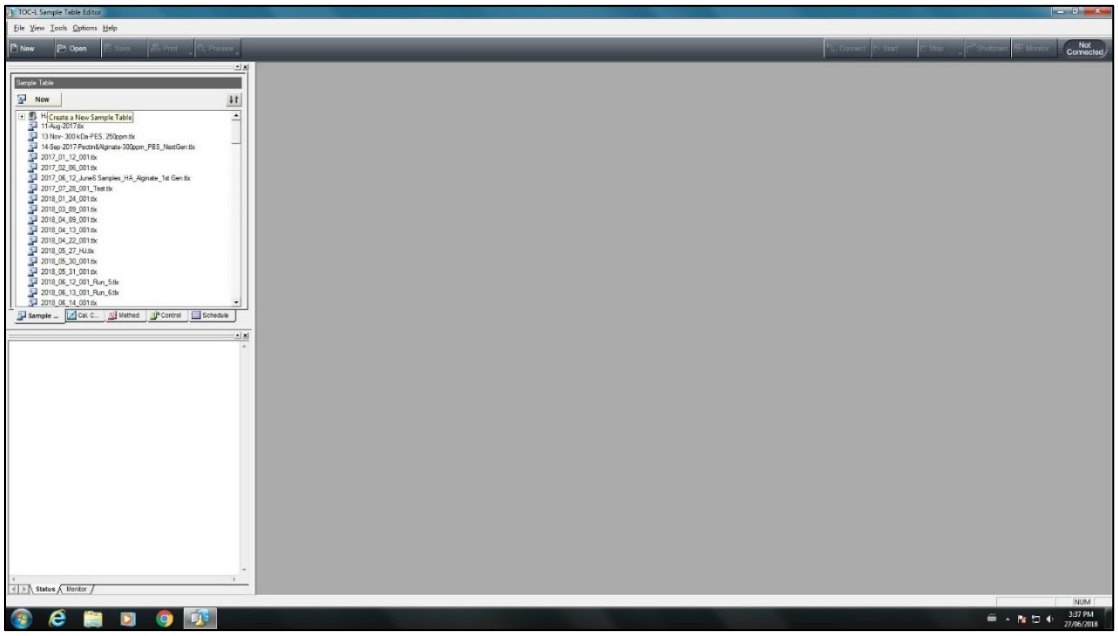
1. Open the "TOC-L Sample Table" software on the computer desktop.



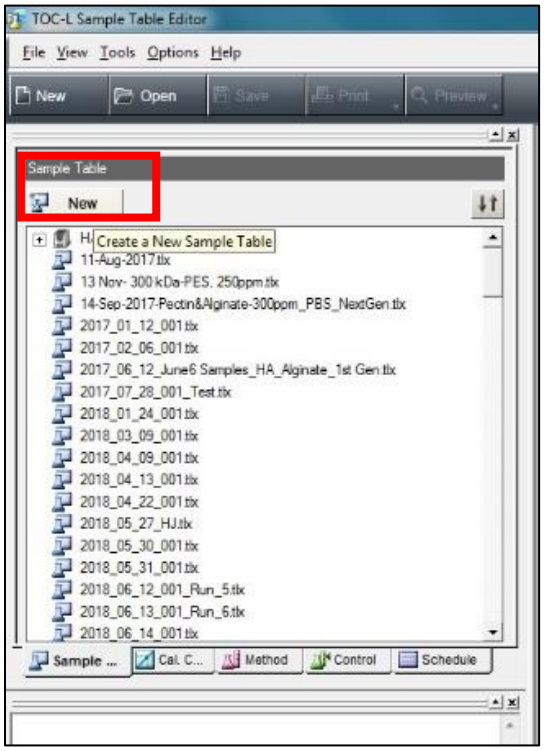
2. Enter your user name (MACID) and password as prompted below. If you do not have a username and a password, contact one of the administrators (University Technology Services).



3. After entering your username (MACID) and password, you will initially see the software interface shown below.



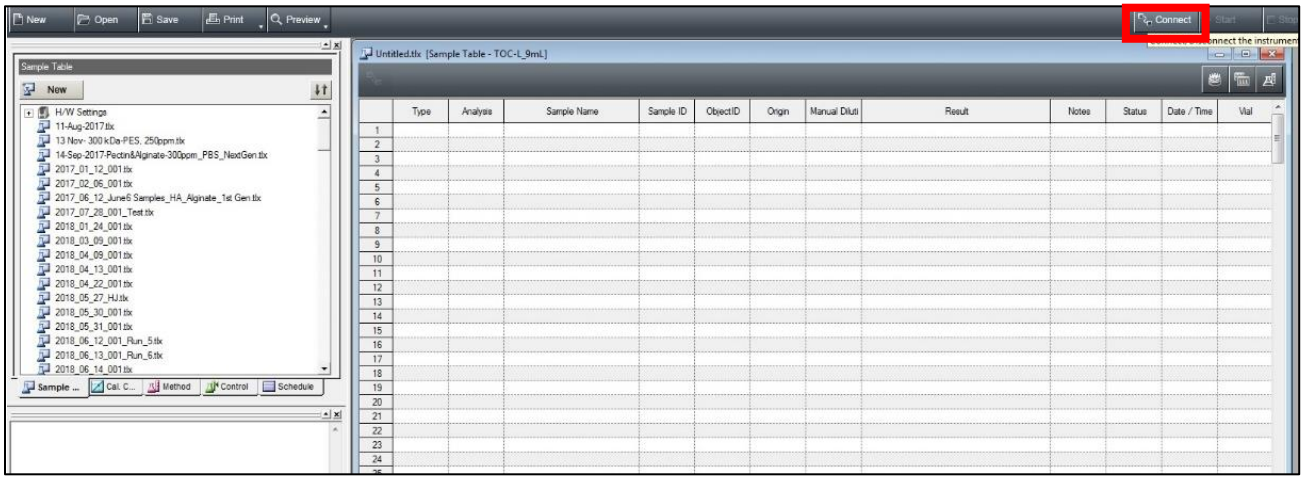
4. In the "Sample Table" tab click on "New" to create a new sample table.



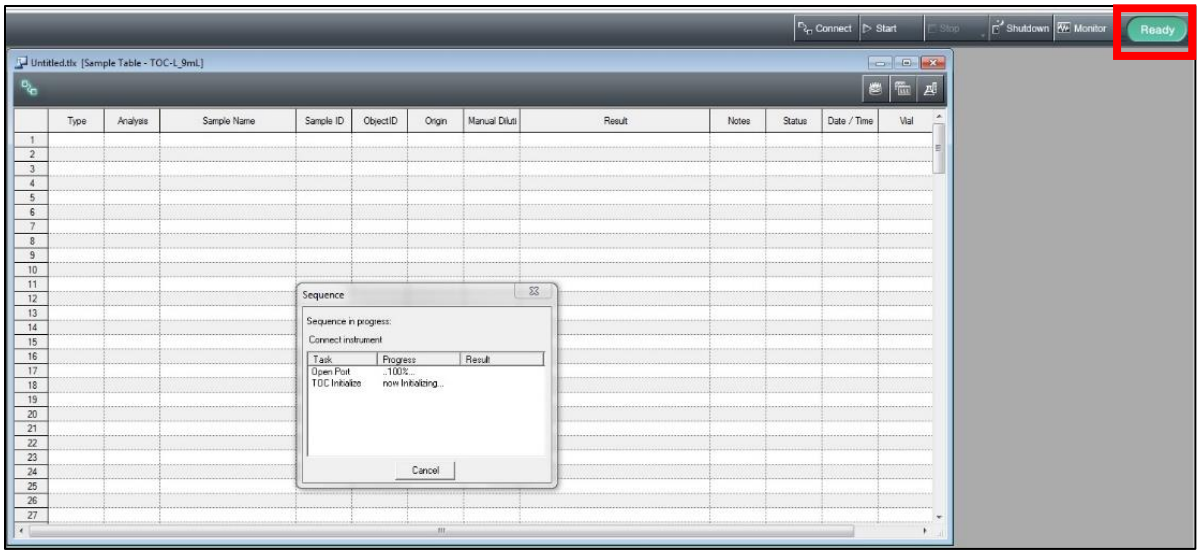
5. Select the appropriate vial size in the menu (9 mL or 40 mL) and proceed to the next step, by selecting "OK". Ensure that the correct tray is installed to match the vial size selection (refer to step 5 of Instrument Set-up, section 3.1).



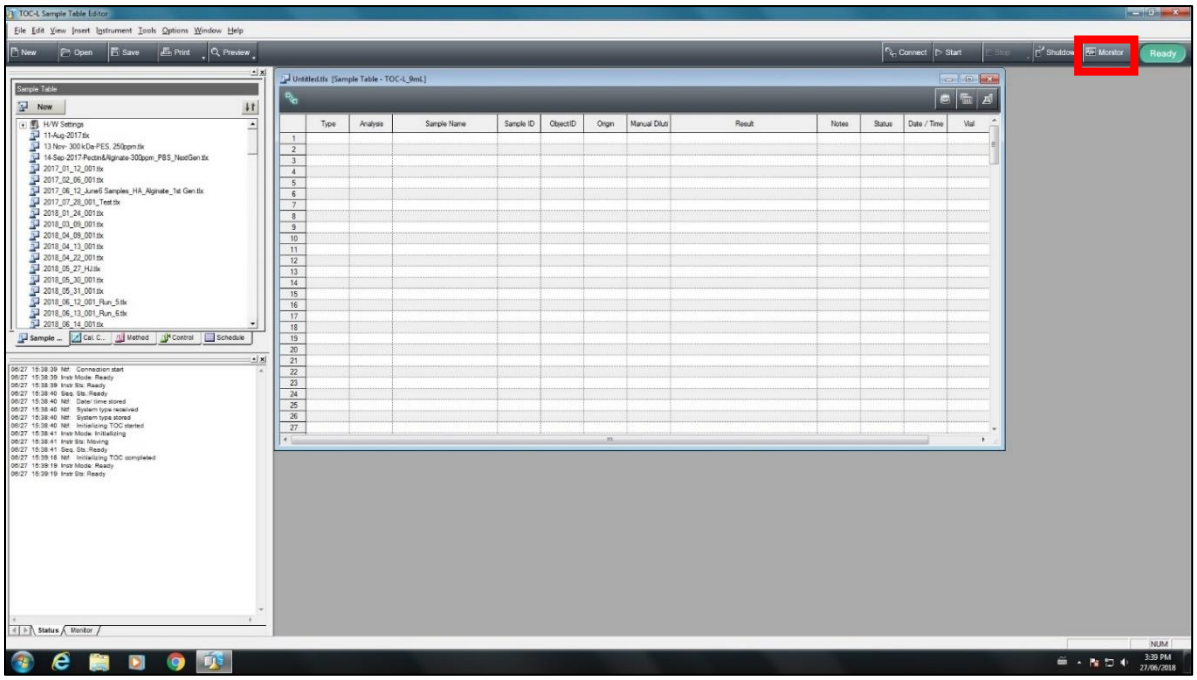
6. A new sample table is created, as seen in the picture below. Now, click on "Connect", in the upper right corner to connect the software to the instrument.



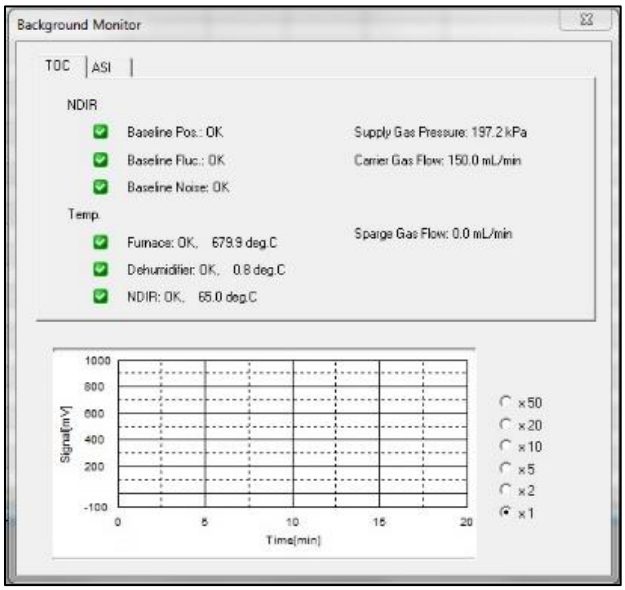
7. A green "Ready" icon can be seen on the top right corner once the furnace reaches the required temperature, the carrier gas is stable, and the signal has reached steady state. If not, an orange "Not Ready" icon will be displayed.



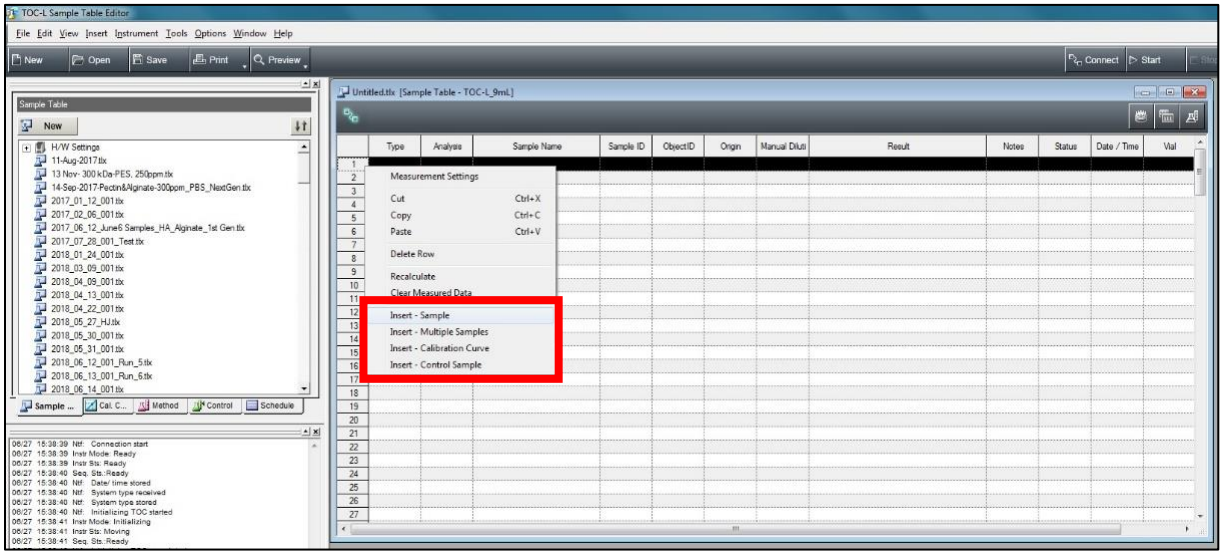
8. To monitor the status of the instrument (e.g. furnace temperature, carrier gas flow, etc.) wait for the initial window to disappear and click on "Monitor".



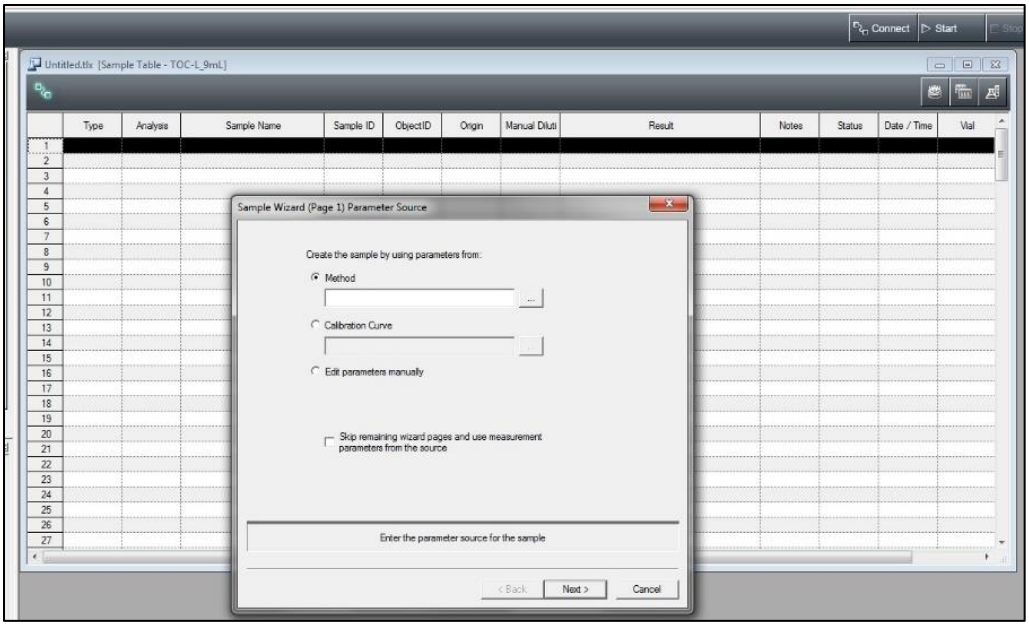
9. The monitor window should appear as below:



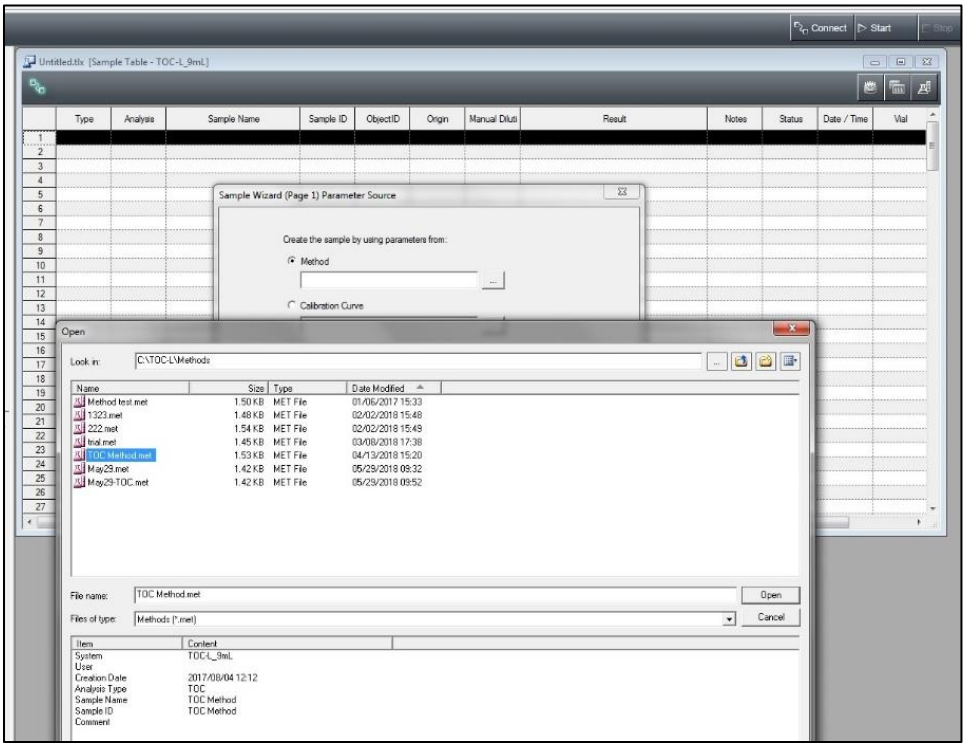
10. Now, right-click on the first row (or the first available row) to add a sample to be measured. Select "Insert – Sample" or "Insert – Multiple Samples" to add one or more samples, respectively.



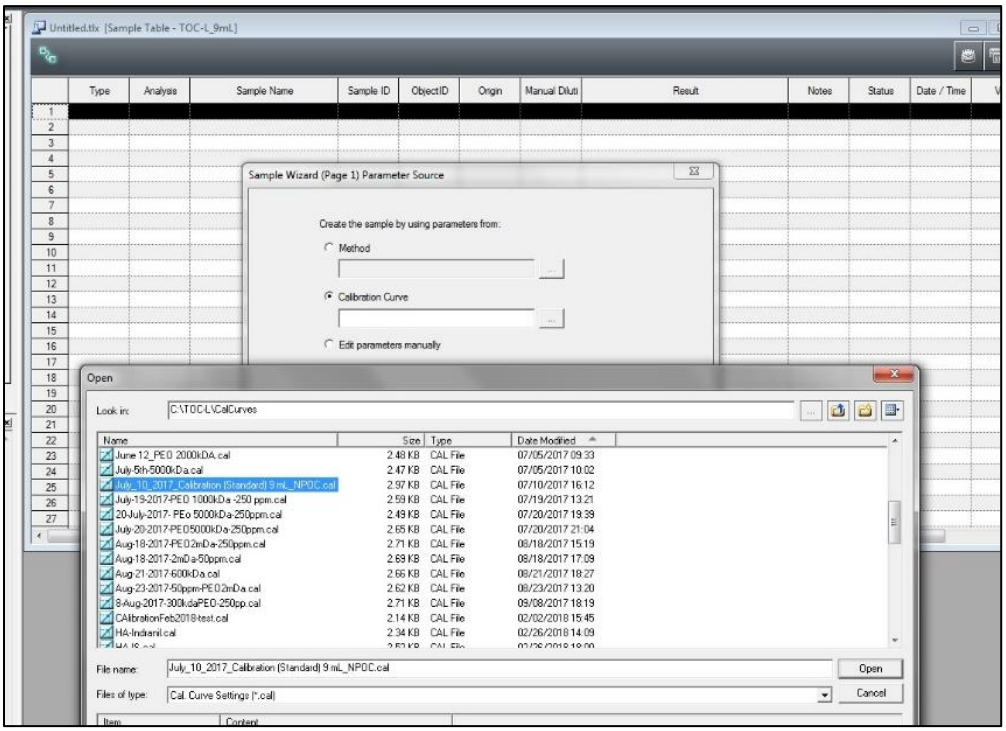
11. To define a sample, you need to either select a method (TOC Method for traditional TOC analysis) or a calibration curve (IC, TC or NPOC) for other analyses.



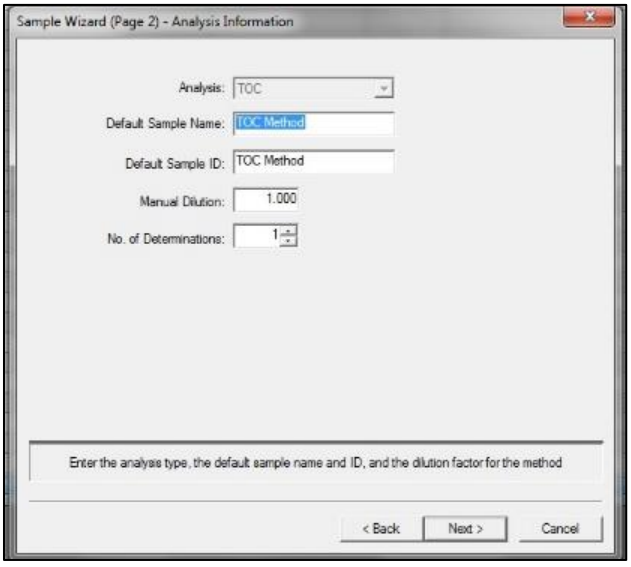
TOC Analysis – Select TOC Method to run TC and IC measurements on each sample and to calculate the TOC defined as TC-IC (see *Background*). This document was written primarily using TOC method as an example.



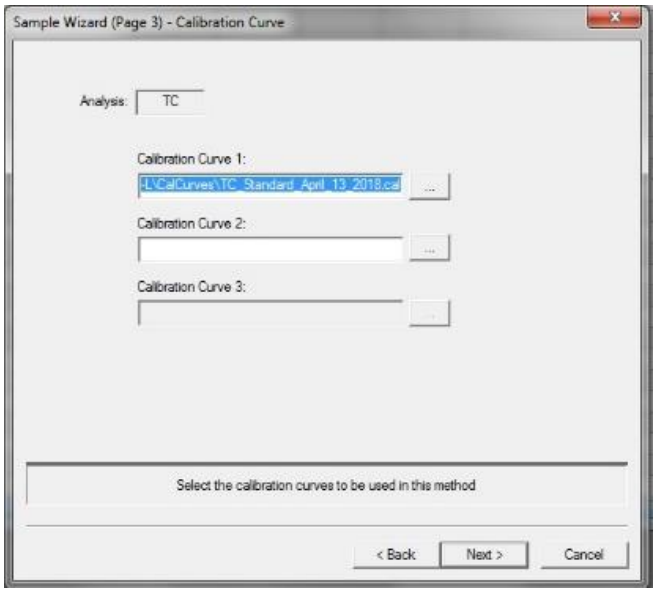
NPOC Analysis - Alternatively, NPOC standard calibration can be selected to determine the TOC using the NPOC method.



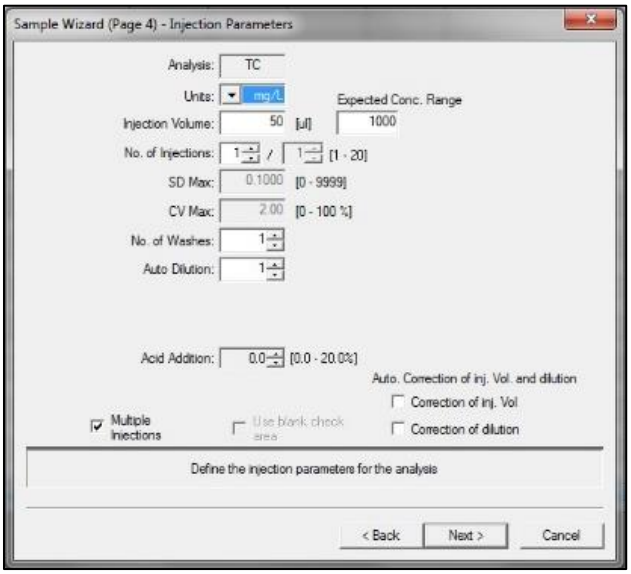
12. Next on Page 2, using the TOC method as an example, you can choose the sample name, the sample ID, the number of manual dilutions and the number of determinations.



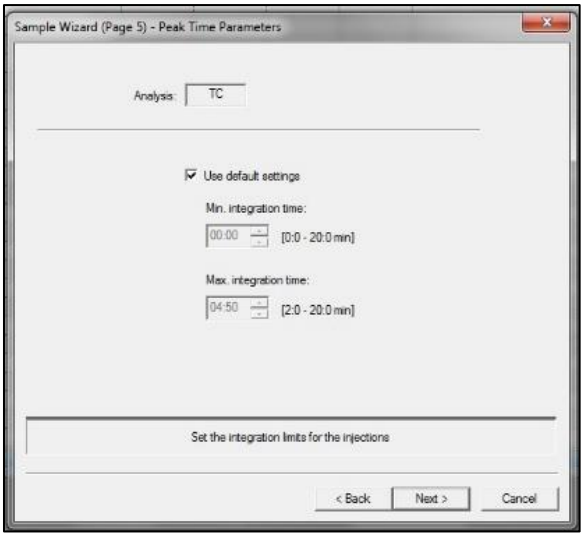
13. Next on Page 3, you can choose and/or edit the calibration curve used in the method for TC calculations.



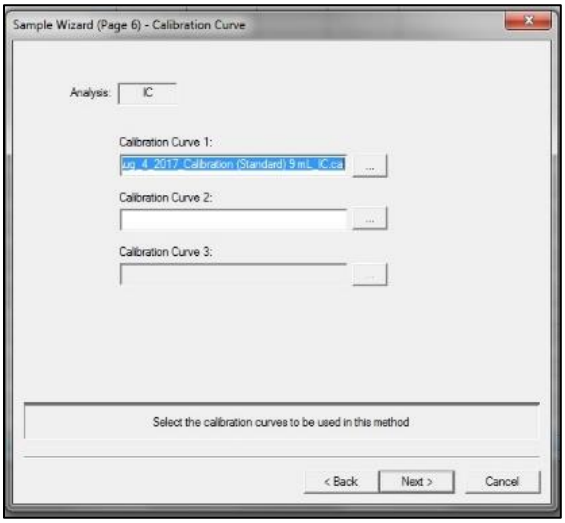
14. Next on Page 4, the units, injection volume of the sample, the number of injections, the number of washes and the auto dilution parameter can be defined. The default setting here is based on the settings used for the calibration curve. The default settings are typically acceptable but can be altered if necessary. Also, statistical parameters (standard deviation & coefficient of variation) can be defined to control the number of injections based on variations if the minimum number of injections is defined greater than 1.

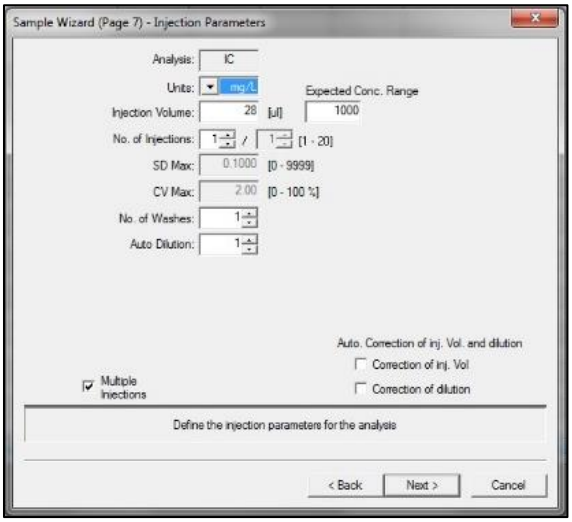


15. The integration time and max integration time can also be changed but the default settings are recommended for most applications.

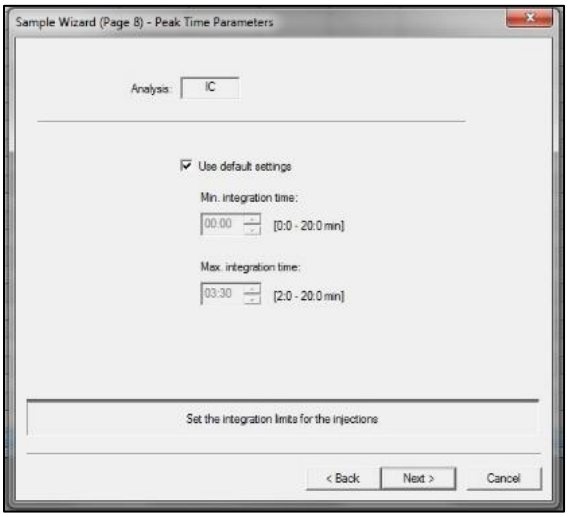


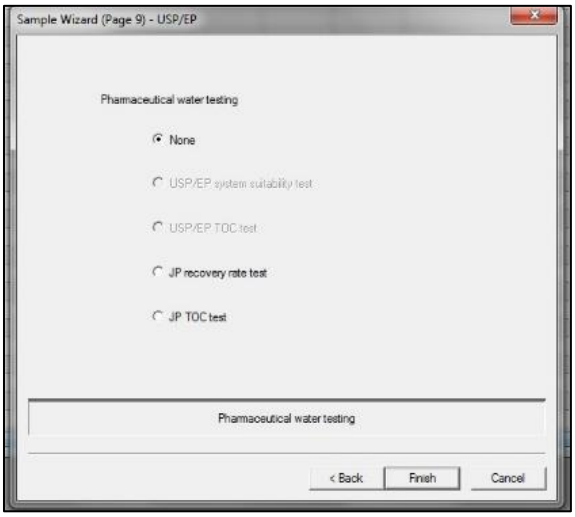
16. The same steps (*i.e.* checking/editing the proper calibration curve and injection settings) should be followed for the IC measurements defined in the TOC method. Note that the default settings are based on the settings used for the IC calibration curve and can be different from the TC calibration curve settings. This is not an error.



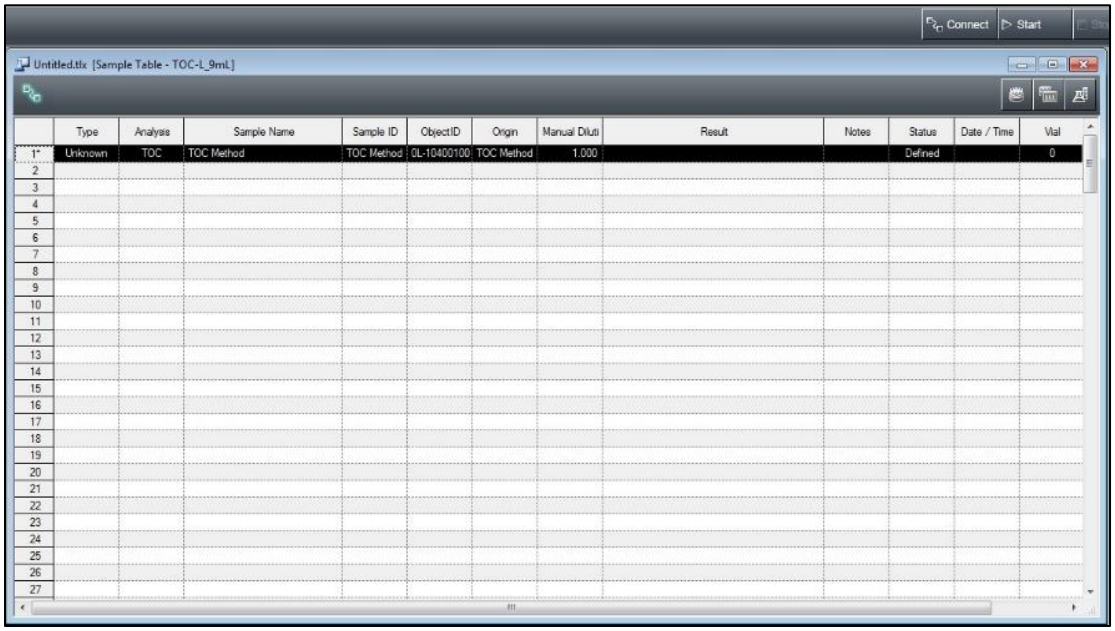


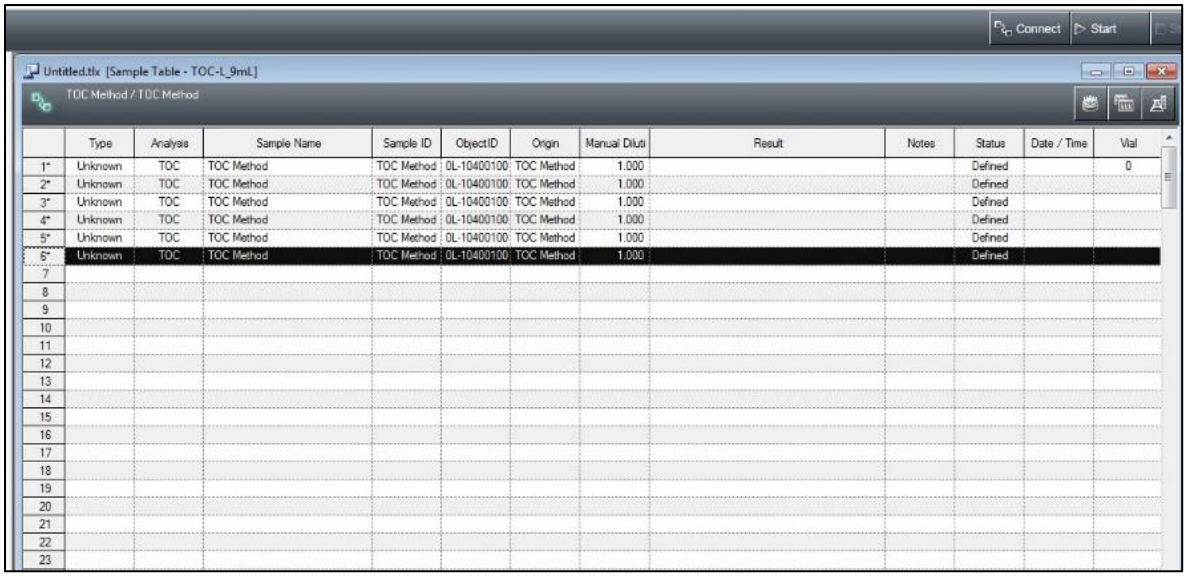
17. Click “Next” and select “Use default settings” and “None” in the following windows (Page 8 and 9).





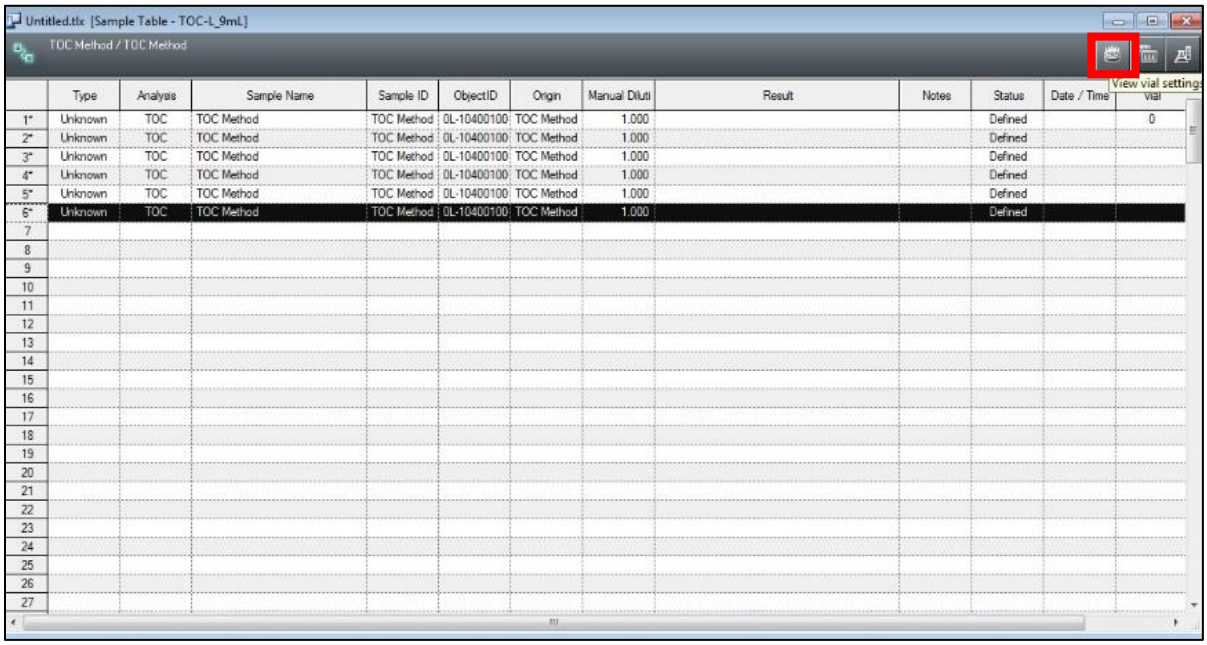
18. Now we have defined a new sample in our sample table. We can add additional samples by following the same steps or by copying and pasting our first sample. For example, six samples are defined below.





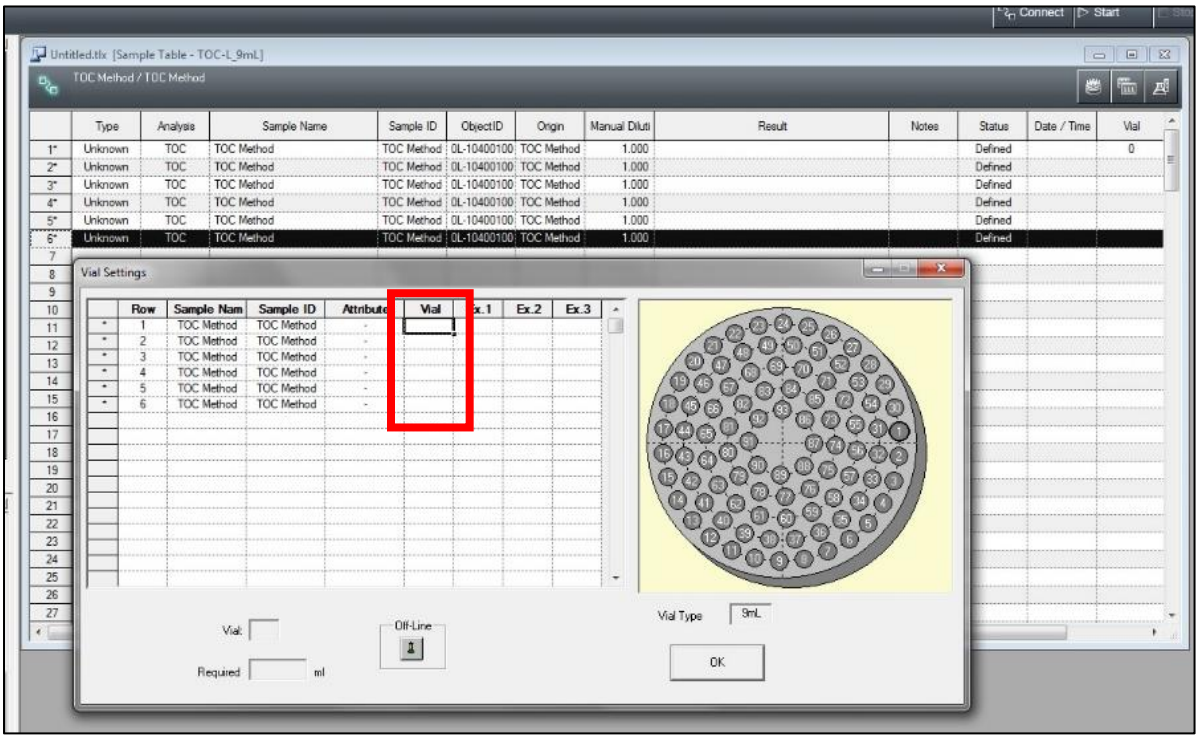
	Type	Analysis	Sample Name	Sample ID	ObjectID	Origin	Manual Dilut	Result	Notes	Status	Date / Time	Vial
1*	Unknown	TOC	TOC Method	TOC Method	0L-10400100	TOC Method	1.000			Defined		0
2*	Unknown	TOC	TOC Method	TOC Method	0L-10400100	TOC Method	1.000			Defined		
3*	Unknown	TOC	TOC Method	TOC Method	0L-10400100	TOC Method	1.000			Defined		
4*	Unknown	TOC	TOC Method	TOC Method	0L-10400100	TOC Method	1.000			Defined		
5*	Unknown	TOC	TOC Method	TOC Method	0L-10400100	TOC Method	1.000			Defined		
6*	Unknown	TOC	TOC Method	TOC Method	0L-10400100	TOC Method	1.000			Defined		
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21												
22												
23												

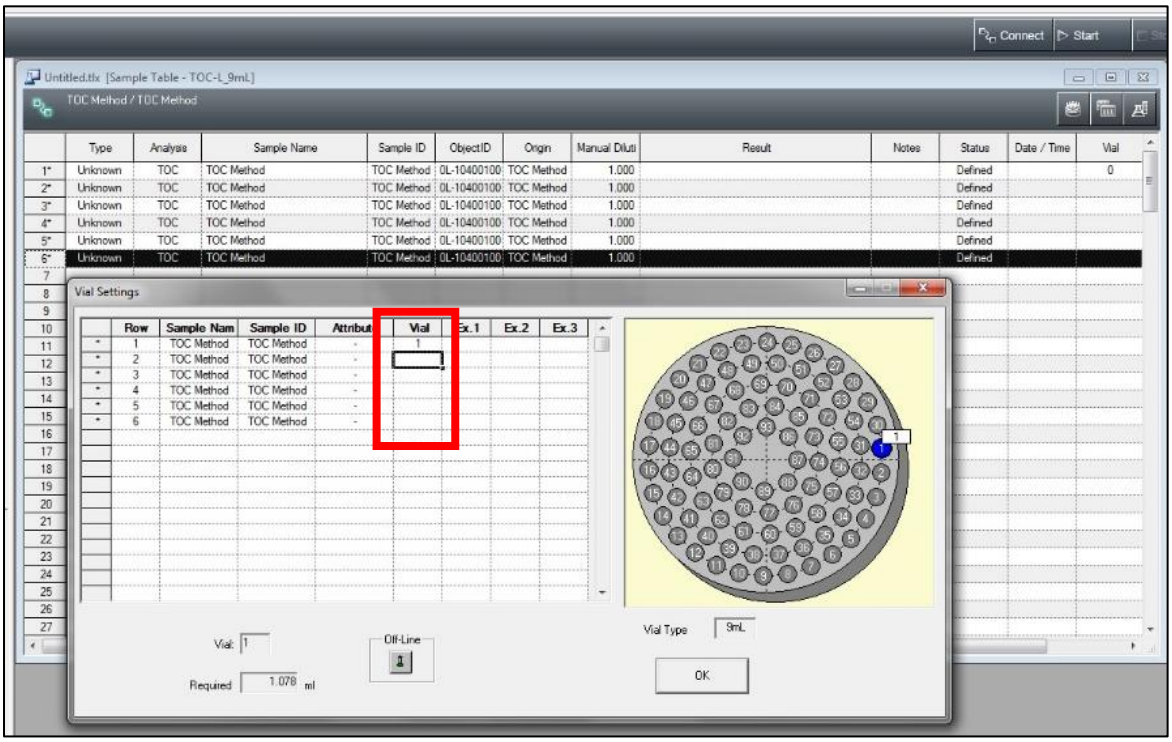
19. Now we need to choose the corresponding vial for each sample (*i.e.* the physical location on the sample tray) using the view vial settings icon (the icon resembles a birthday cake).



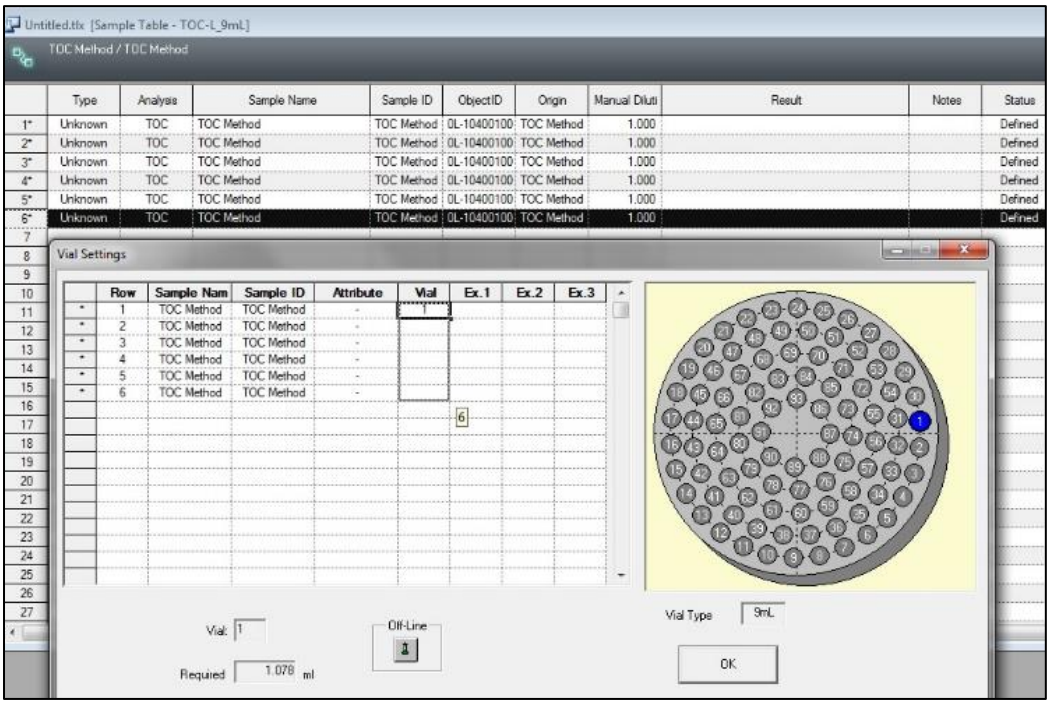
	Type	Analysis	Sample Name	Sample ID	ObjectID	Origin	Manual Dilut	Result	Notes	Status	Date / Time	Vial
1*	Unknown	TOC	TOC Method	TOC Method	0L-10400100	TOC Method	1.000			Defined		0
2*	Unknown	TOC	TOC Method	TOC Method	0L-10400100	TOC Method	1.000			Defined		
3*	Unknown	TOC	TOC Method	TOC Method	0L-10400100	TOC Method	1.000			Defined		
4*	Unknown	TOC	TOC Method	TOC Method	0L-10400100	TOC Method	1.000			Defined		
5*	Unknown	TOC	TOC Method	TOC Method	0L-10400100	TOC Method	1.000			Defined		
6*	Unknown	TOC	TOC Method	TOC Method	0L-10400100	TOC Method	1.000			Defined		
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21												
22												
23												
24												
25												
26												
27												

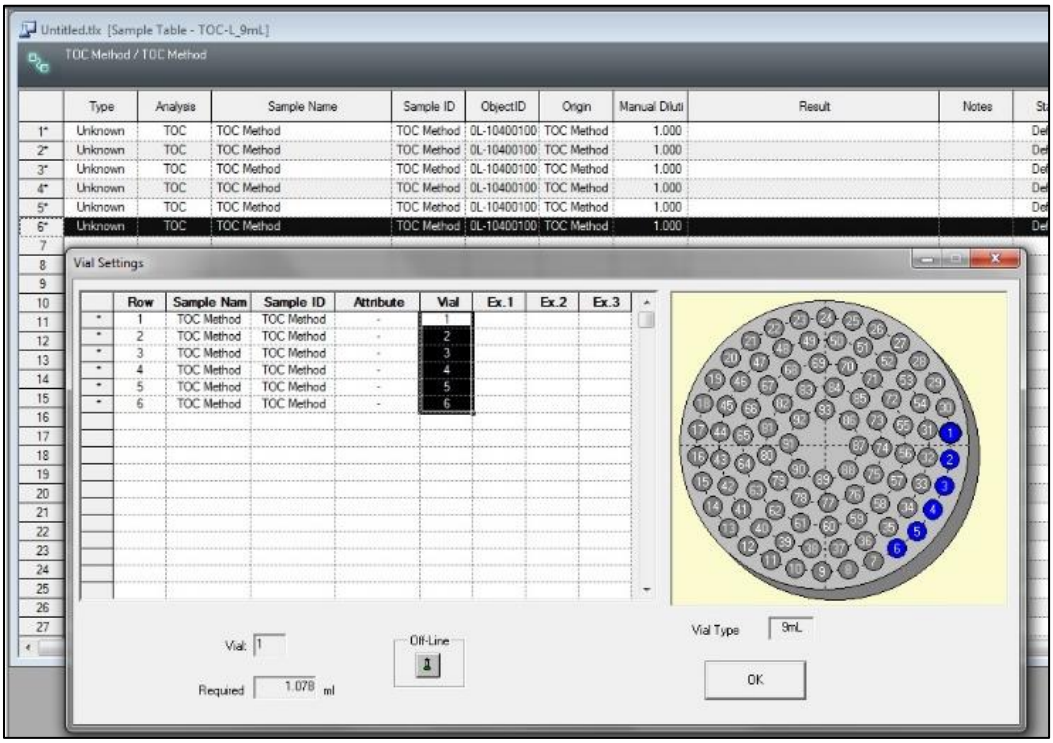
In the window below, we can choose the vial number corresponding to each sample from the tray diagram or simply by typing the vial number corresponding to the location on the sample tray.

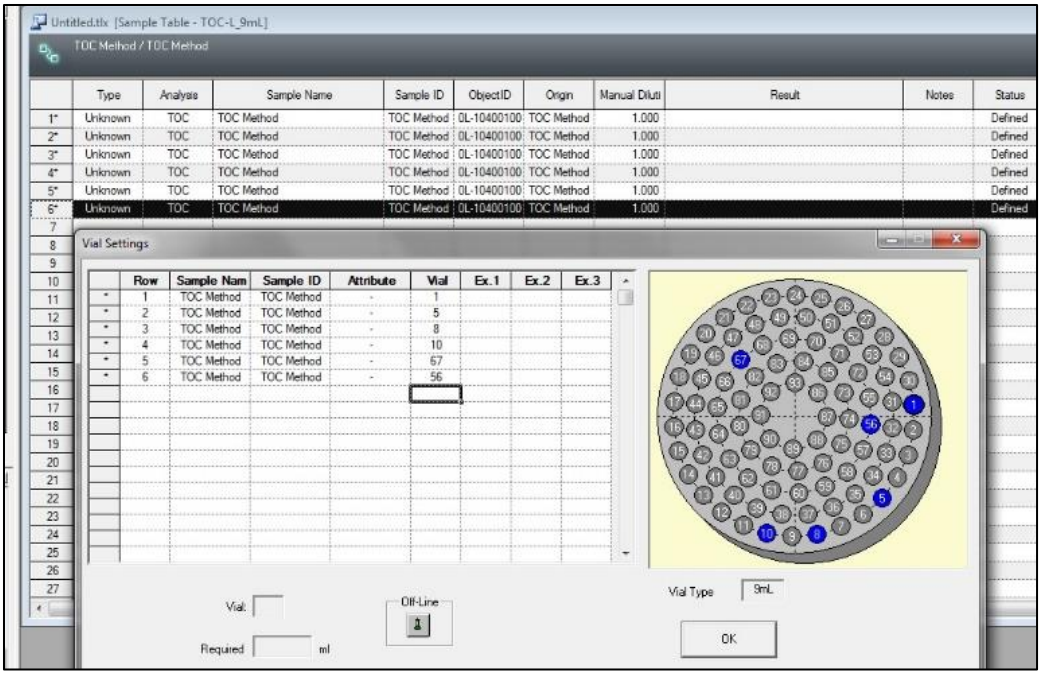




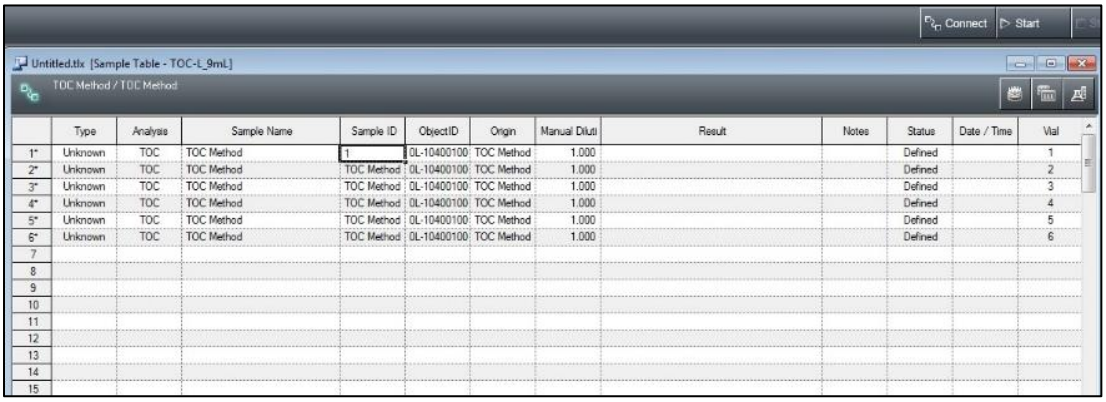
We can choose vials either consecutively by using the drag and drop option or one-by-one if required (shown in the next three figures).

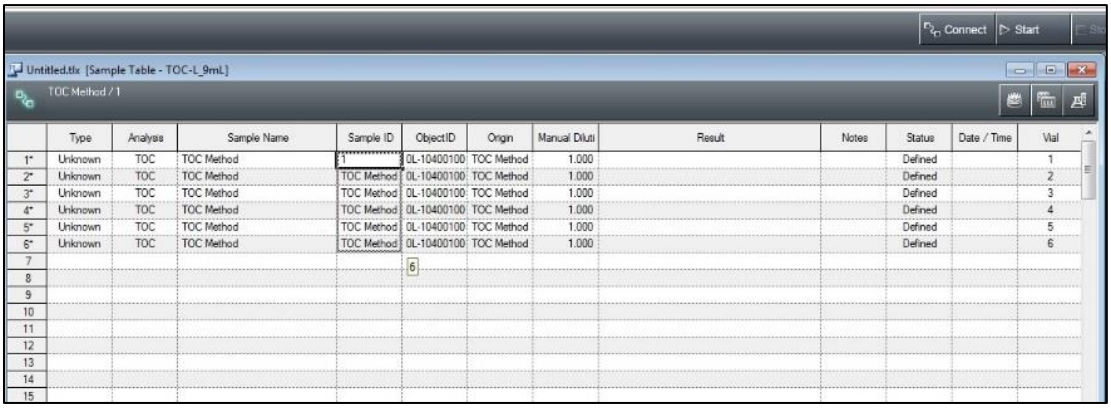




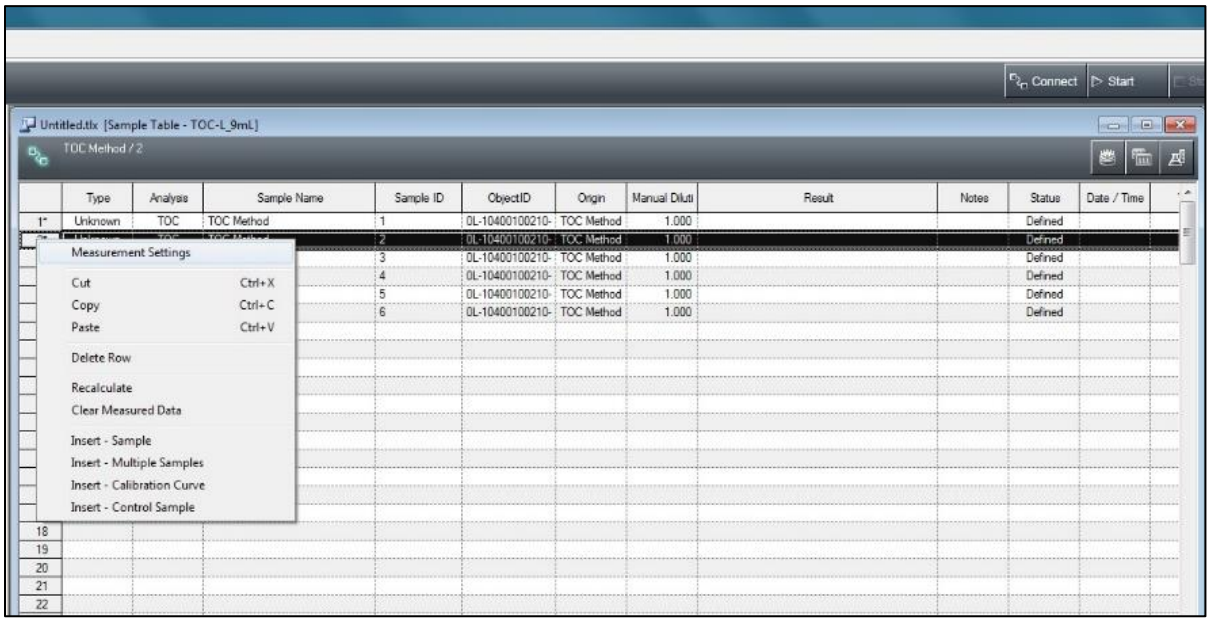


20. Below we can see our samples defined with their corresponding vials. It is possible to change the "Sample ID" of each sample on the sample table, itself, for the purposes of sample labelling.

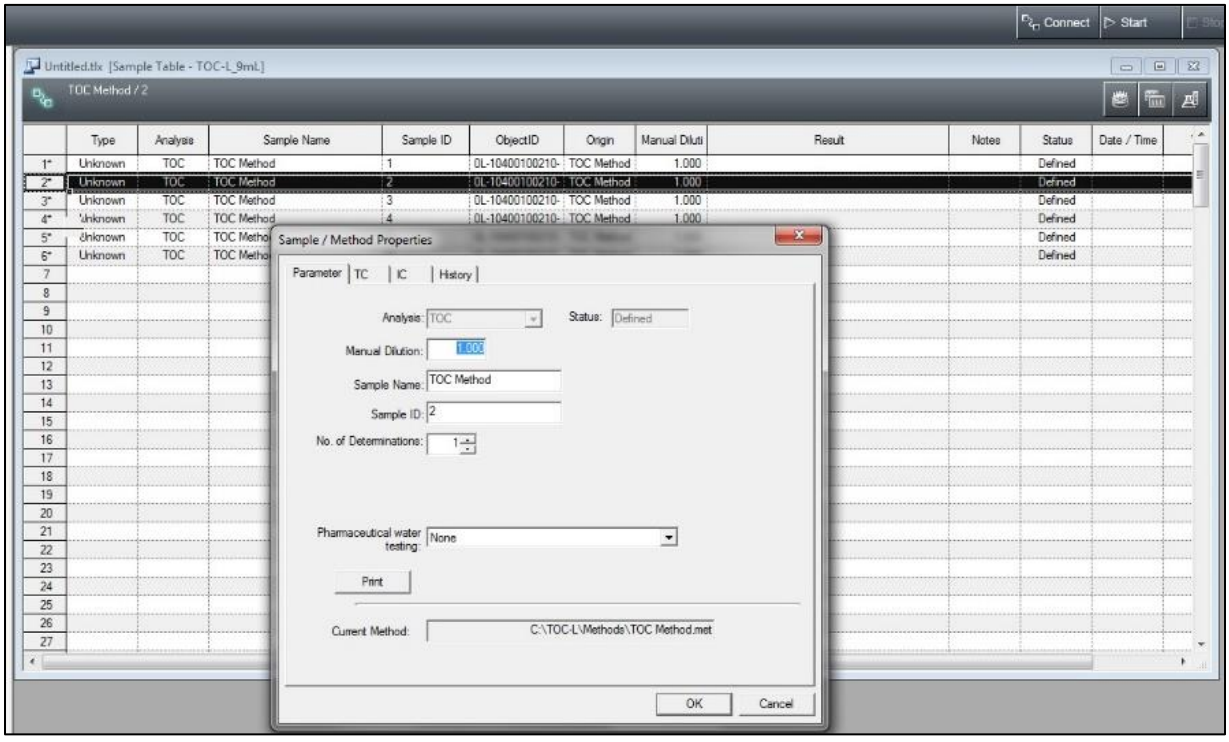




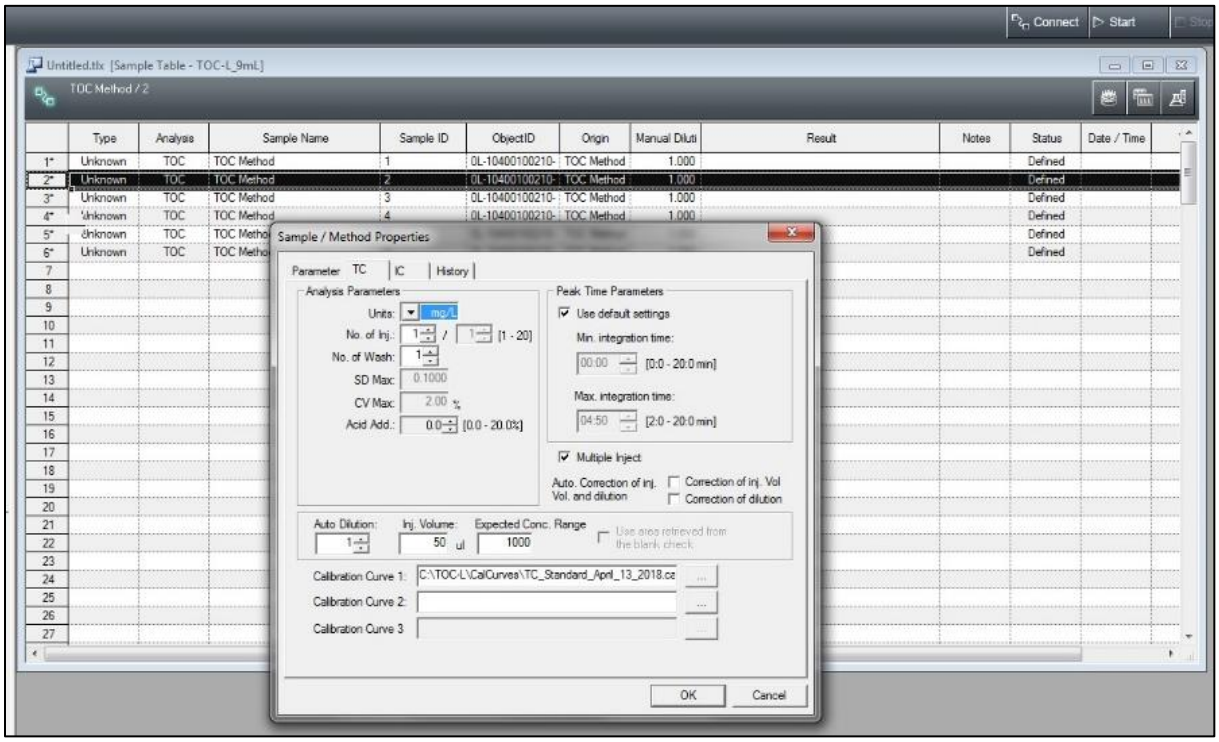
21. To change the other settings, right-click on the row that you would like and select "Measurement Settings".

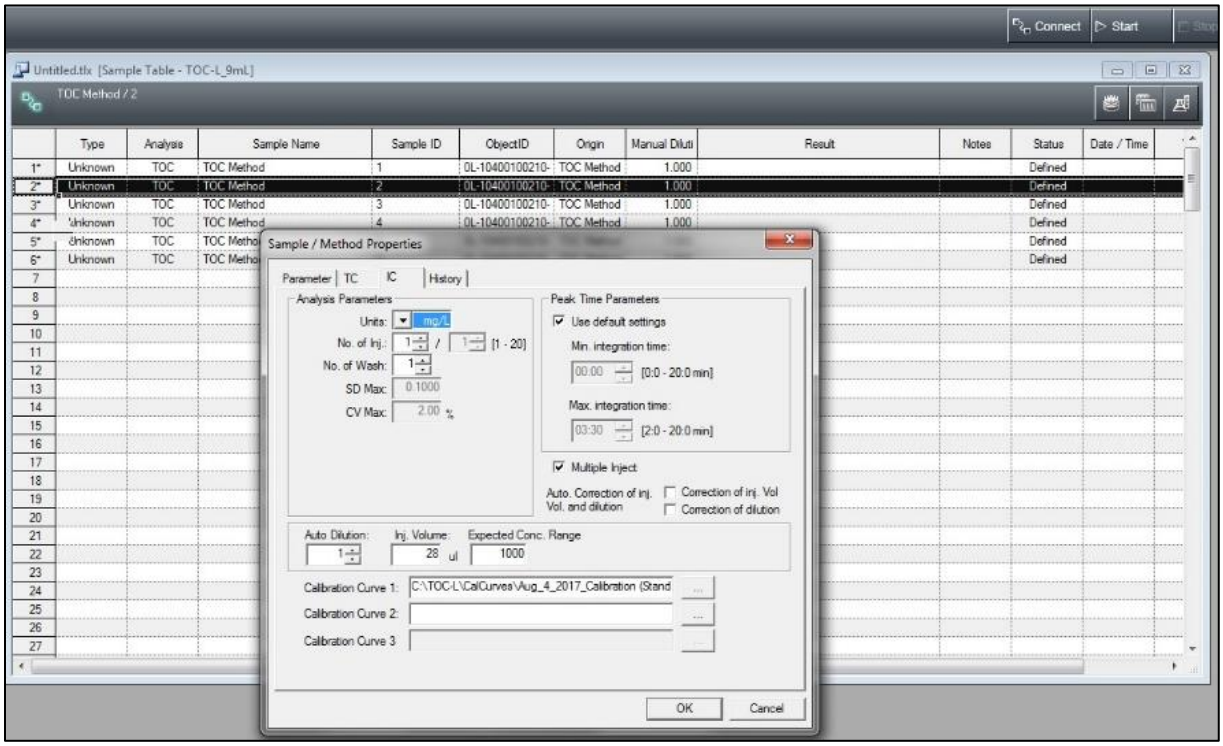


In the first tab (parameters) sample name, manual dilution ratio, sample ID and number of determinations can be changed.

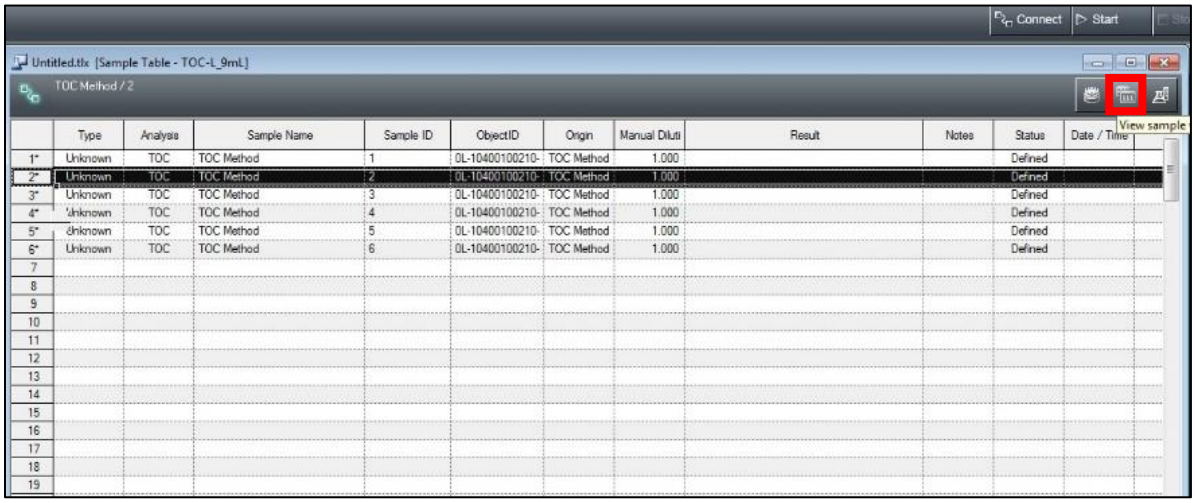


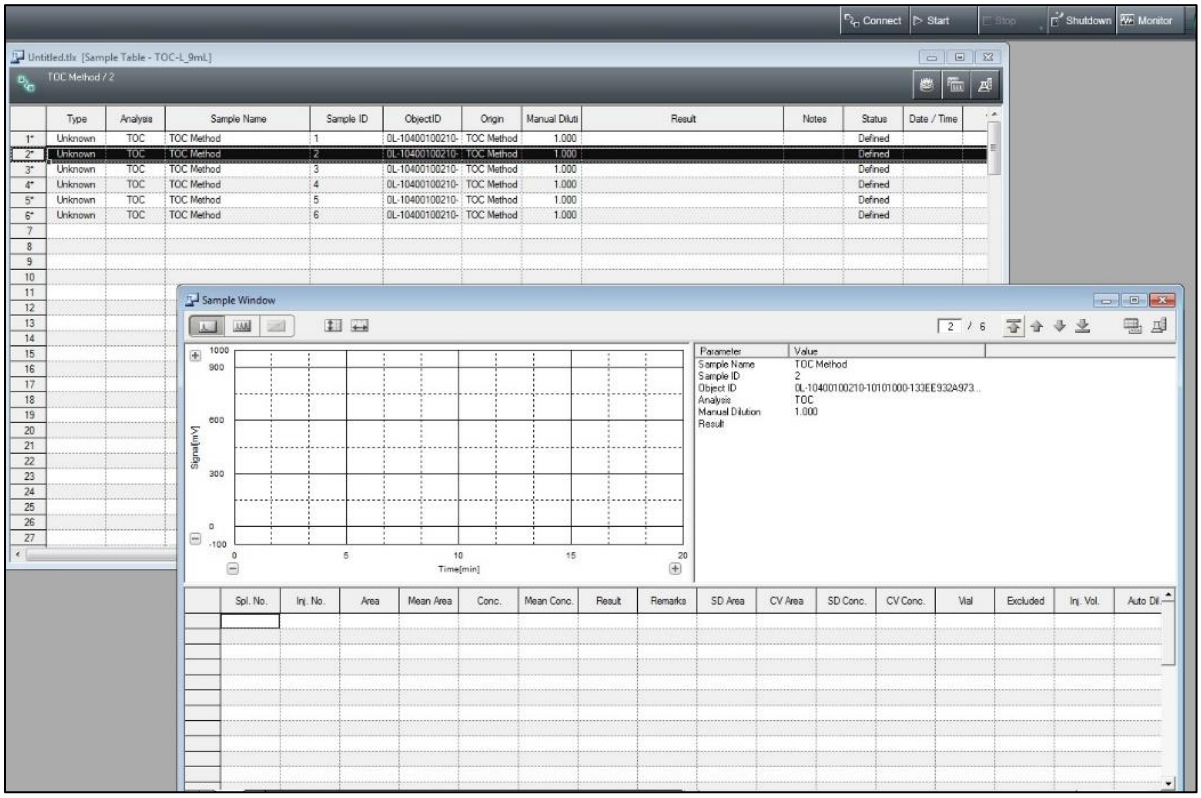
In the next tab, the settings related to TC measurements can be changed. These settings are selected based on your calibration when defining each sample. The same thing can be done in the IC tab shown in the second picture below.

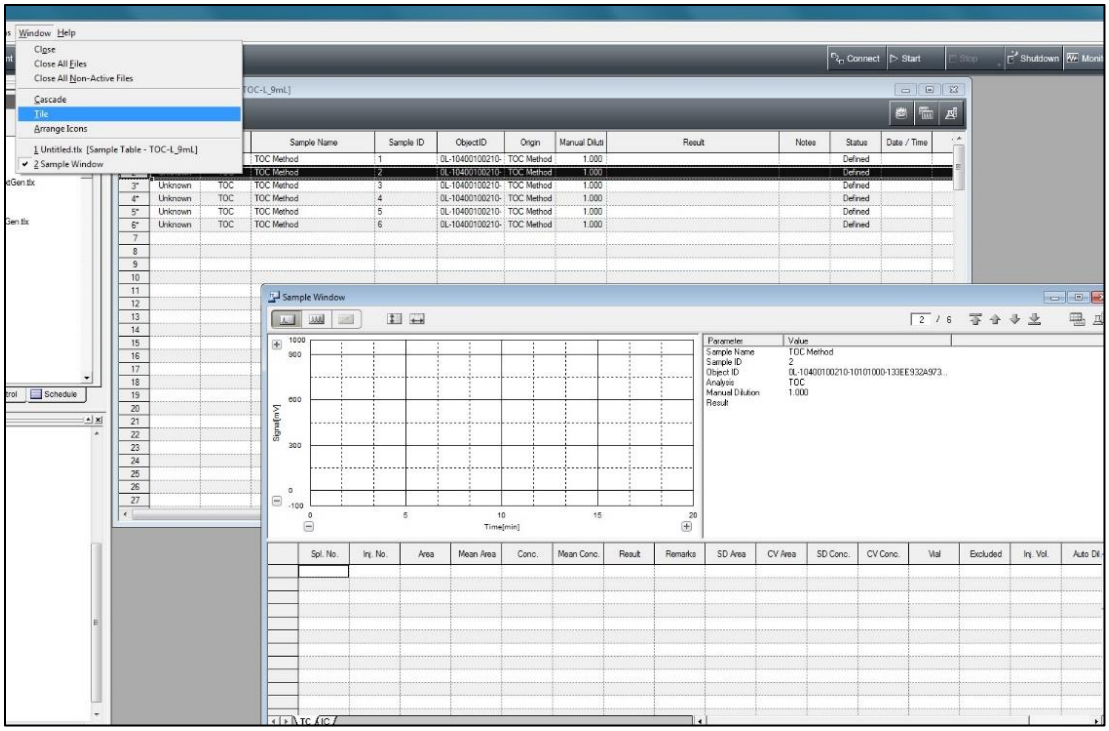


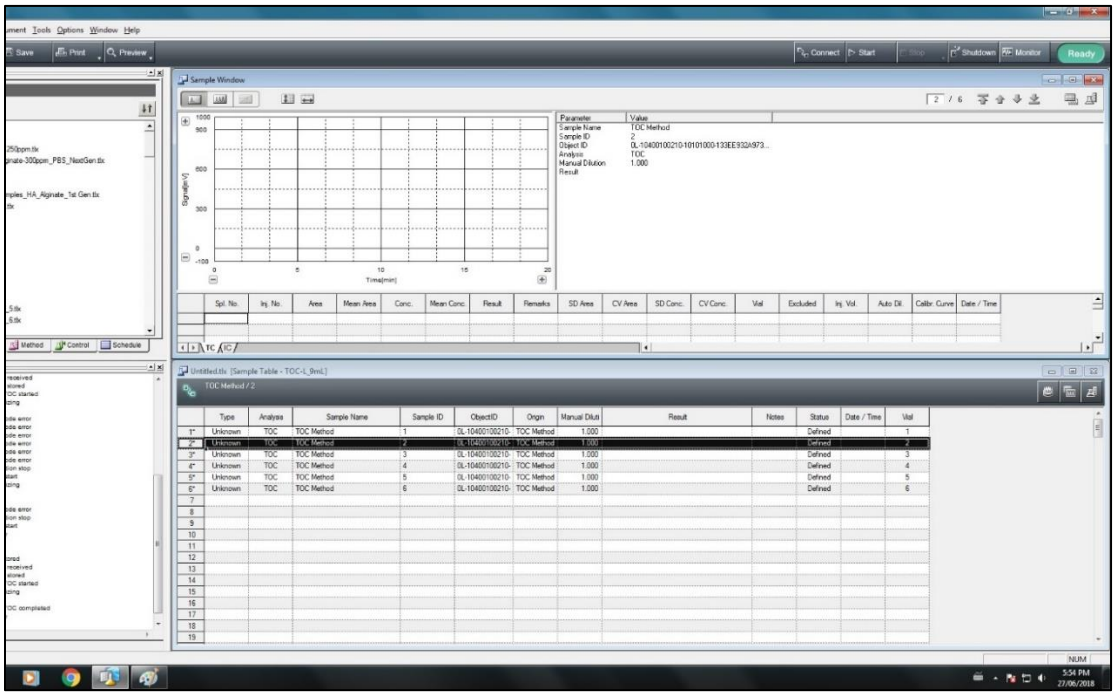


After choosing all the proper settings click on “Sample Window” icon (second icon on the top right corner of the sample table) to see your results. To see both the sample table and your results windows choose the tile option from the drop-down menu (shown in the third and fourth pictures below).

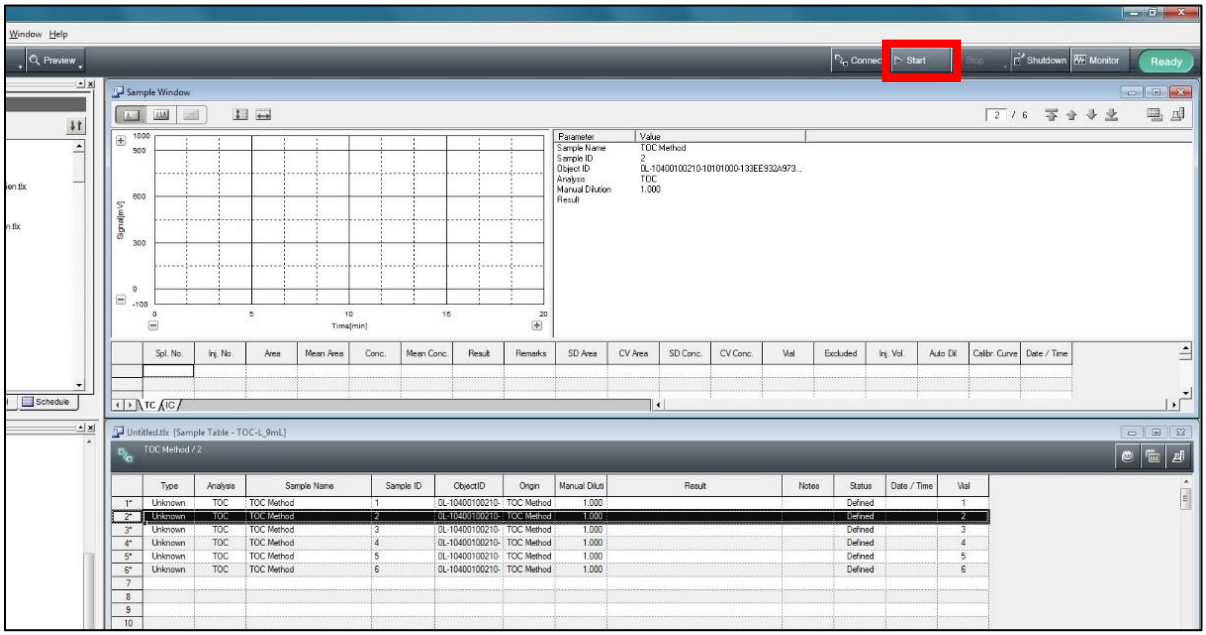




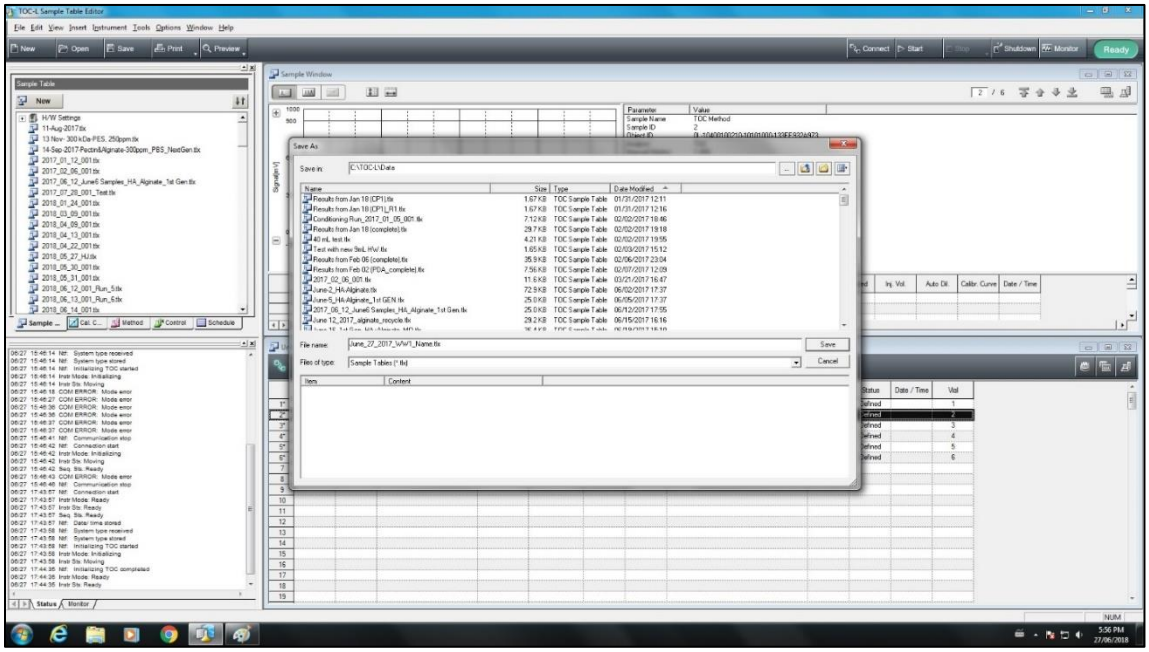

Window Help
Close
Close All Files
Close All Non-Active Files
Cascade
Tile
Arrange Icons
1 Untitled.tlk [Sample Table - TOC-L_9mL]
2 Sample Window
Sample Window
Sample Name
Sample ID
ObjectID
Origin
Manual Dilut
Result
Notes
Status
Date / Time
Parameter
Value
Spl. No.
Inj. No.
Area
Mean Area
Conc.
Mean Conc.
Result
Remarks
SD Area
CV Area
SD Conc.
CV Conc.
Val
Excluded
Inj. Vol.
Auto Dil.


Sample Window
Ready
Sample Name
Sample ID
ObjectID
Origin
Manual Dilut
Result
Notes
Status
Date / Time
Val

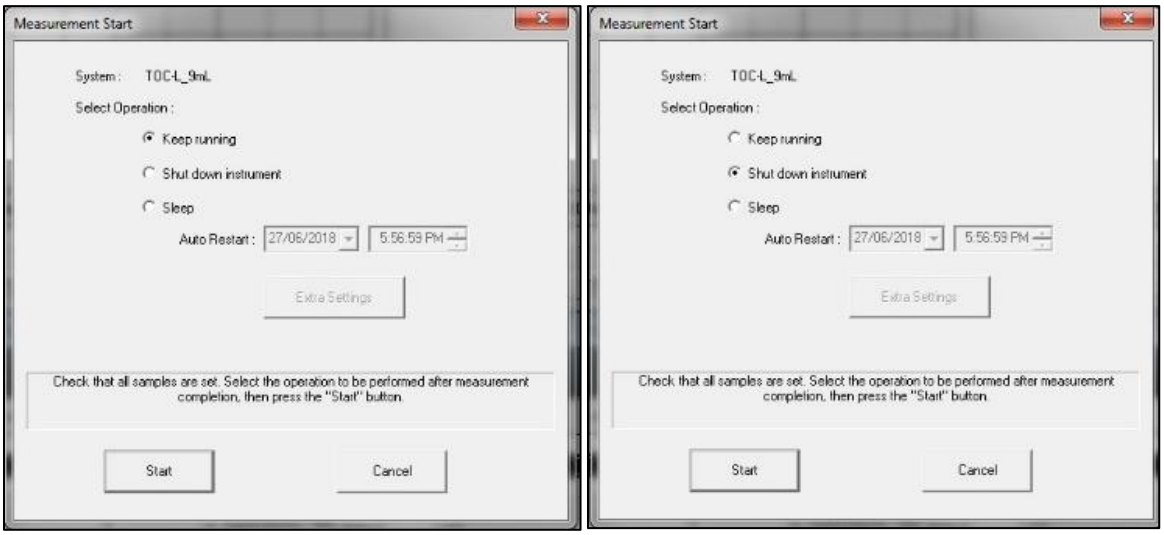
22. Now we have everything ready to start the run. Ensure all the water and acid levels on the instrument are enough considering the number of your samples, the number of injections and the type of measurements. Also make sure that the carrier gas pressure is high enough and the valve is open. Next, click on the "Start" button on the top right corner of the software.



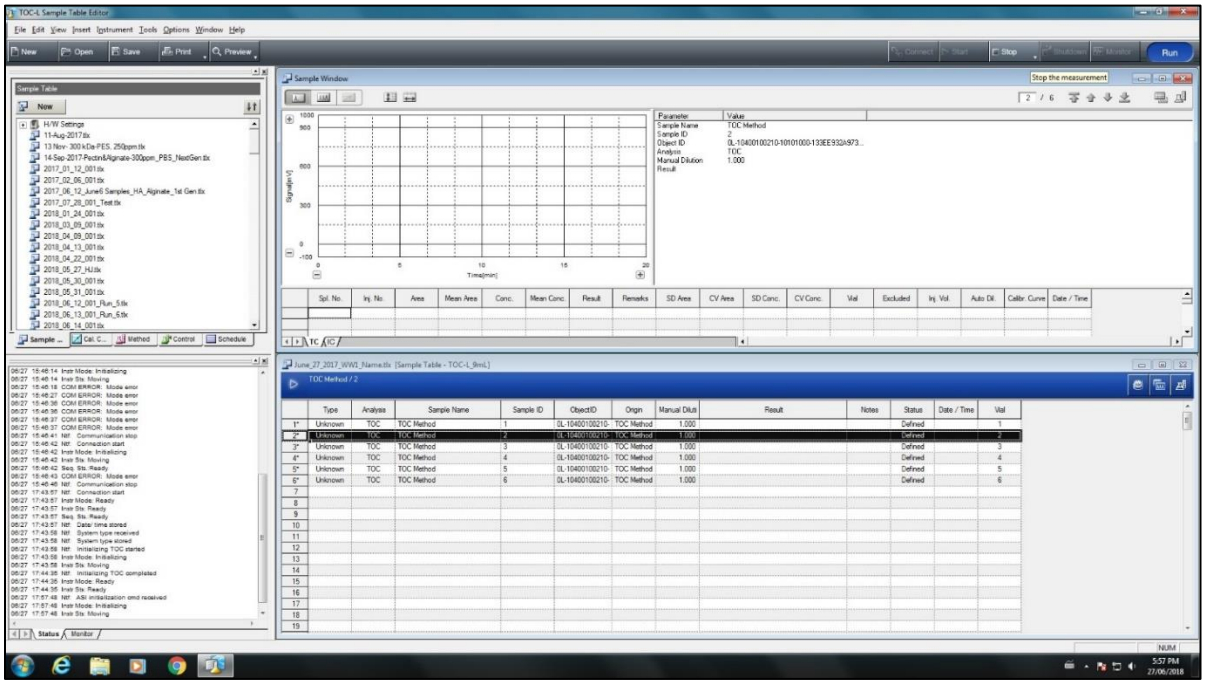
23. Next, the sample table must be saved under the proper name. Note that if you keep using the same sample table every time you do not need to save the sample table under different names each time.



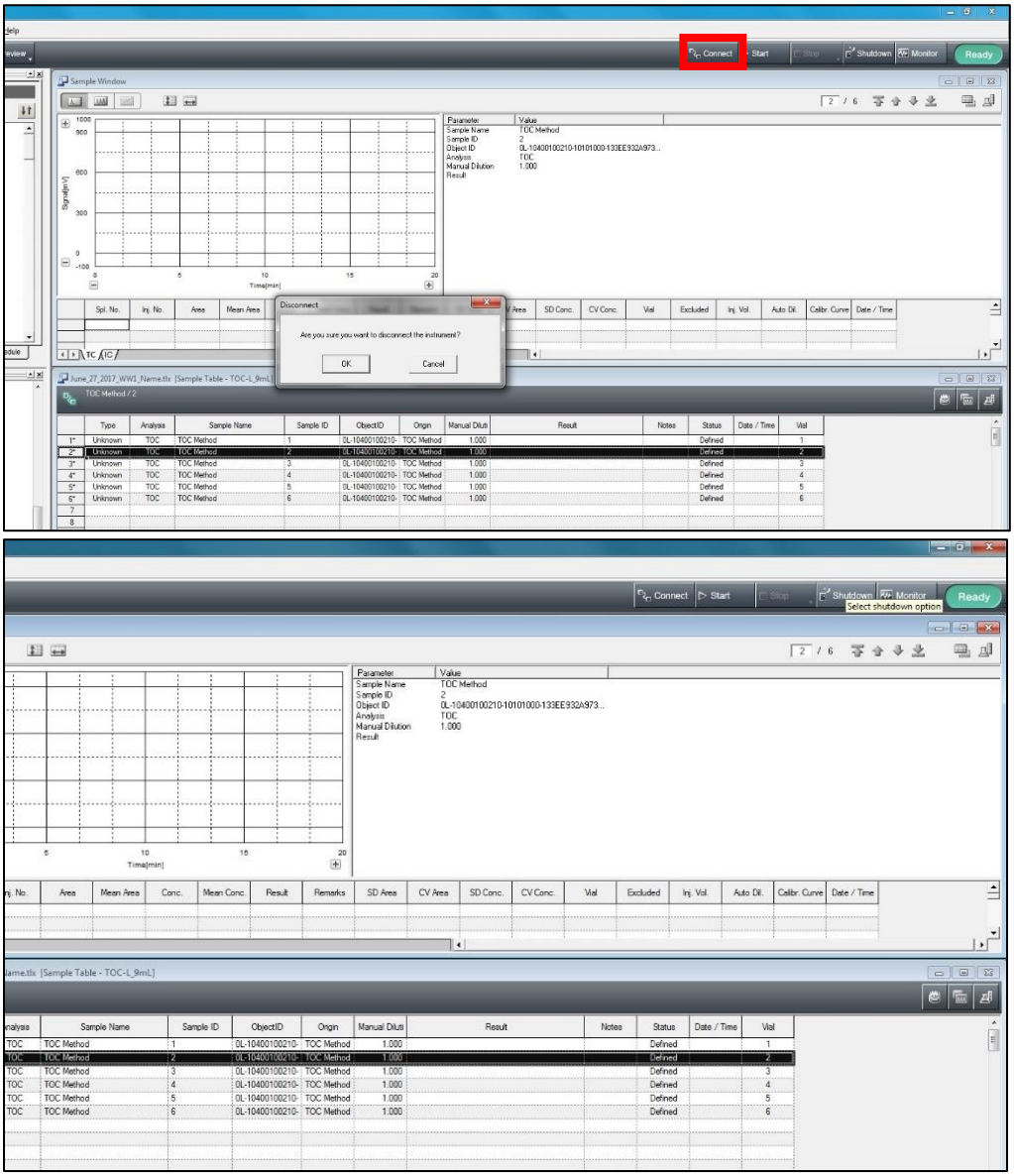
24. You will now need to choose whether you want to run more samples after your run or you want the software to shut-down the instrument, in the “Measurement Start” dialogue box.



25. After clicking “Start”, the green “Ready” icon will be changed to a blue “Run” icon. It is a good idea to stay and watch the instrument closely for the first run to see if it works as expected.



26. Also, if you would like to run the sample later or for you need to abort the communication between the software and the instrument you can select “Connect” in the upper right corner to disconnect the instrument. **Note that this does not mean that you are shutting the instrument down.** To shut down the instrument you need to have the instrument connected and use the shutdown button.



27. Next, once you select “OK”, the software will shut down the instrument, but the fan will keep running for 30 minutes. **Under no circumstance use the power button on the instrument to shut it down since it will turn off the fan and the heat from the furnace will melt the tubing and parts of the instrument, causing severe damage.**

