SHIMADZU
Excellence in science

TOC-L Series
Quick Reference Guide

Click to begin…

SHIMADZU

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Instrument Start-Up

1. Turn on main power switch on the right hand side of instrument
2. Push power button on the front
3. Turn on carrier gas

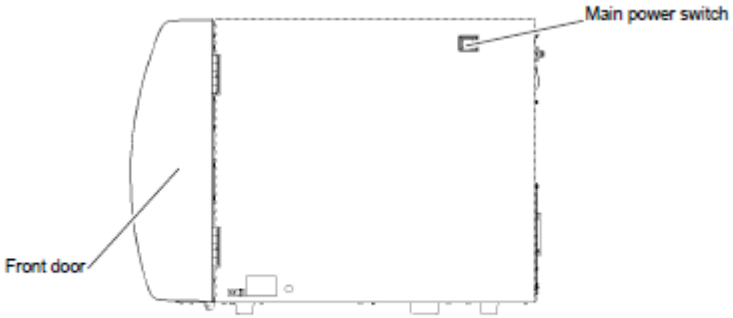


Figure 2.2 Right Side View

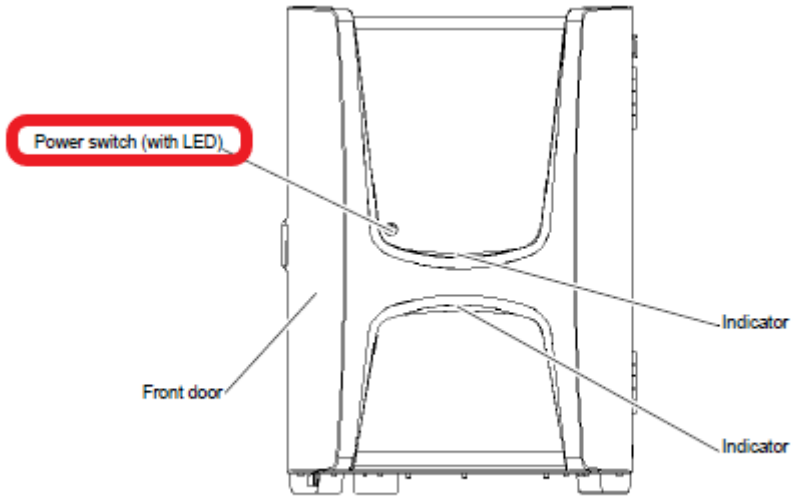


Figure 2.1 Front View

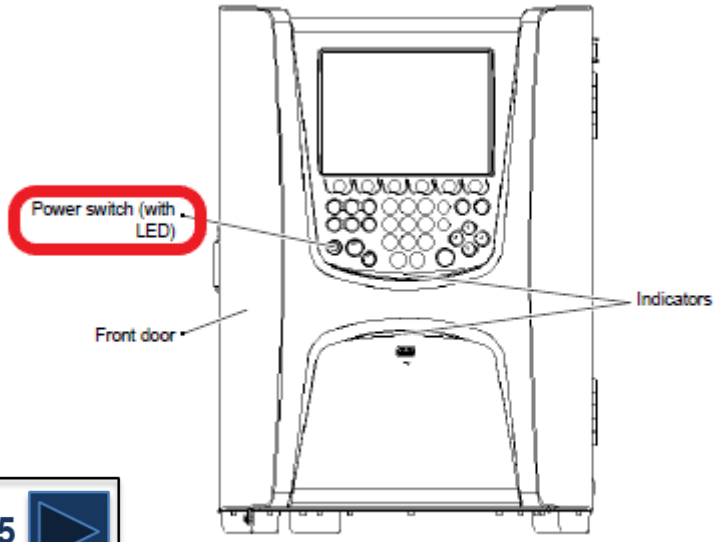


Figure 2.1 Front View

Section 1 / 5

Instrument Start-Up

Click on the ‘TOC-Control L icon to open the software

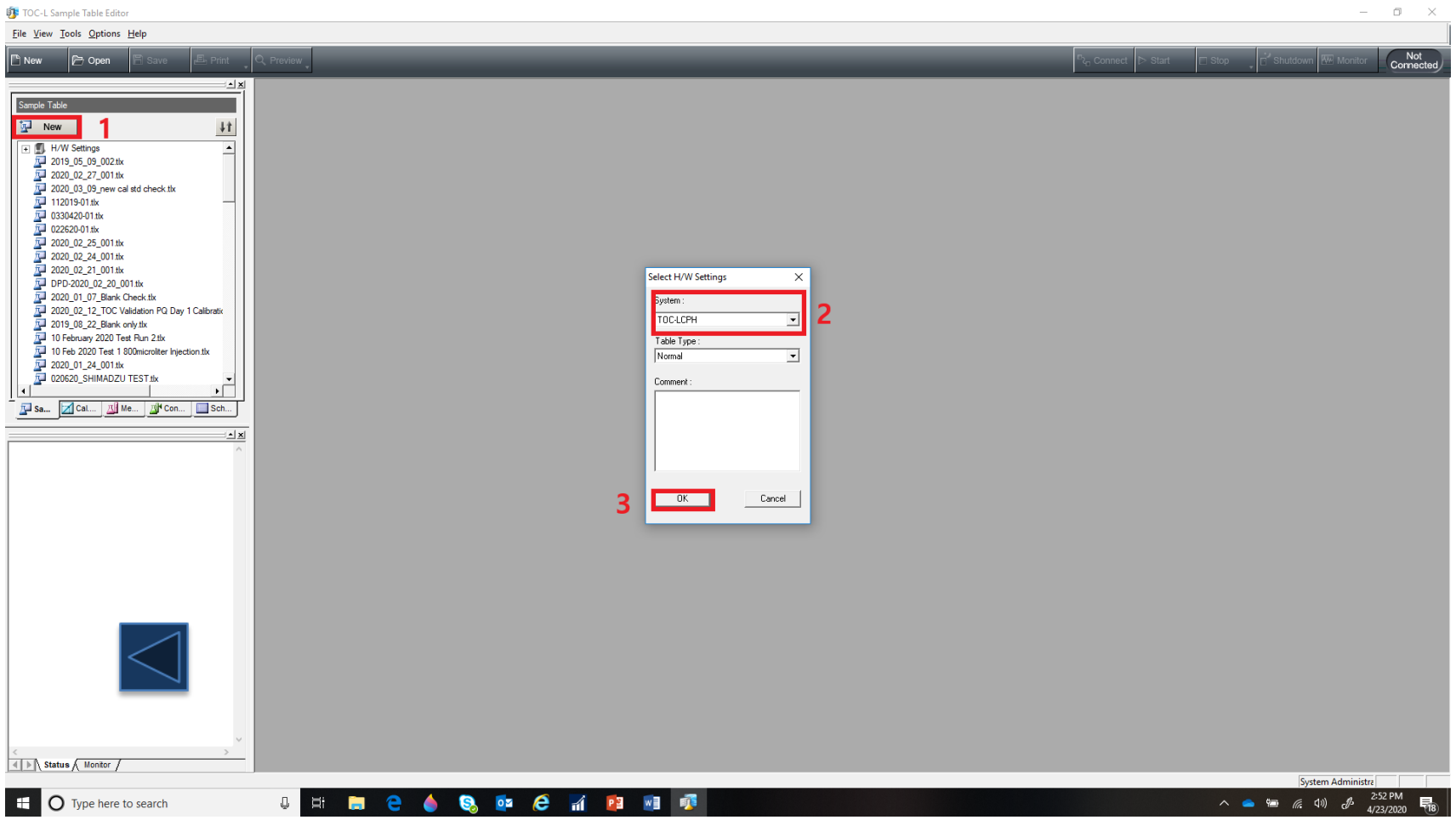
Click the ‘Administration’ tab to view Software Administration Functions

Click ‘Sample Table Editor’ to open the TOC Software.

Click ‘H/W Settings’ to view or create a hardware configuration.

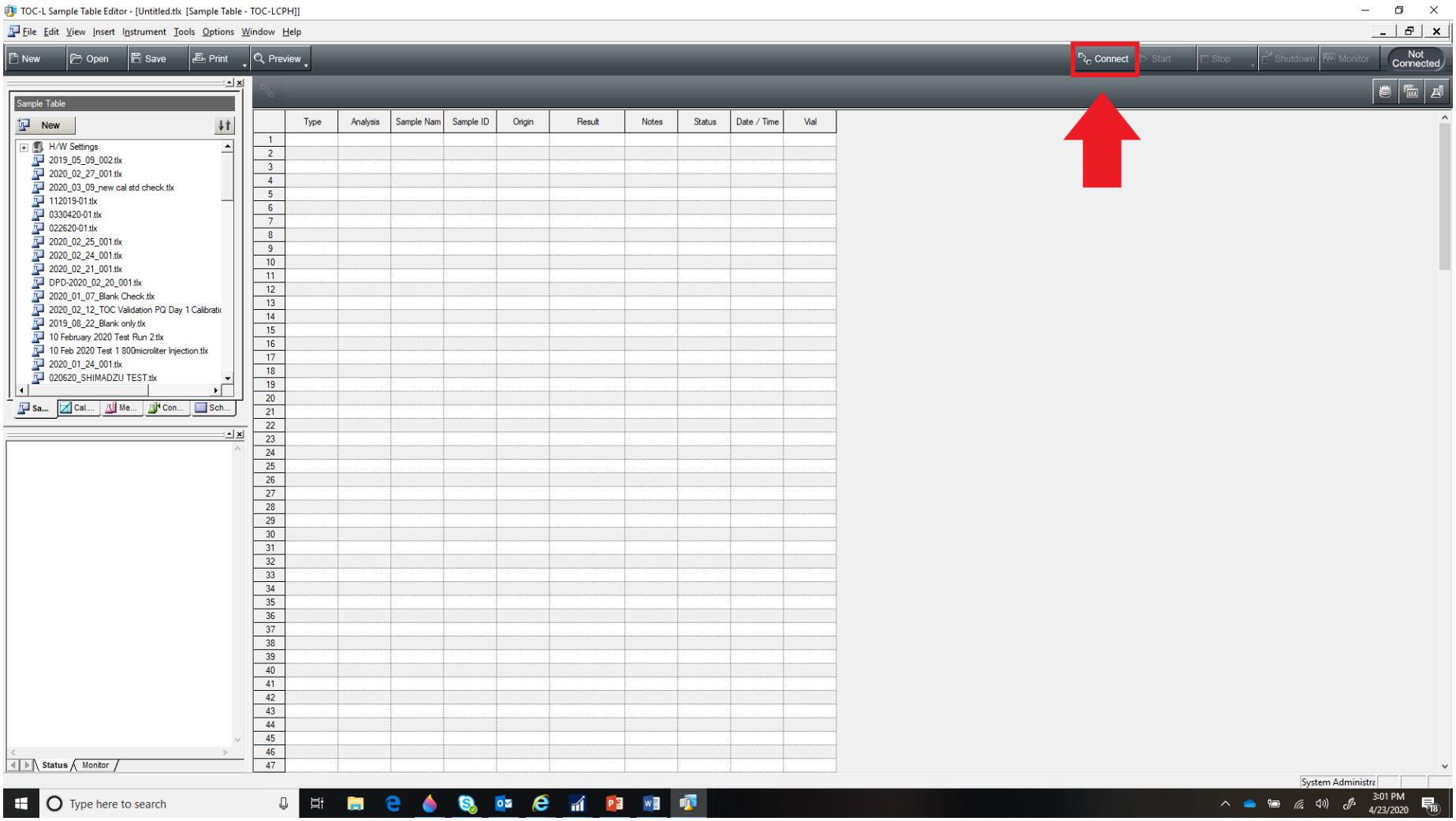
Instrument Start-Up

1. Open the TOC sample table editor and open a new sample table
2. Select the correct hardware configuration
3. Press ‘OK’



Instrument Start-Up

Press ‘Connect’ to start initializing the instrument



Instrument Start-Up

It Takes approximately 30-45 minutes for the instrument to stabilize and come to a ready status.

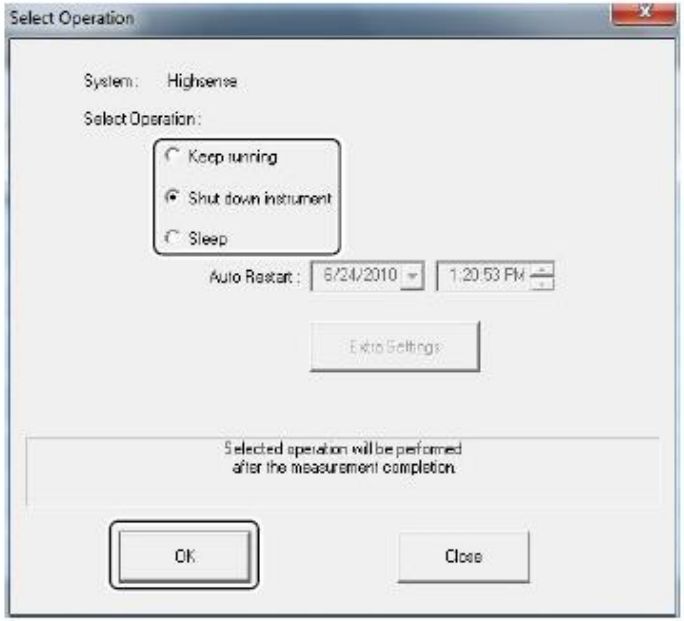
Push the ‘Monitor’ button to view the instrument status in real time. When all parameters are ok the instrument is ready to start running samples.

Instrument Shutdown

Click 

.

The Shutdown dialog box is displayed.



Select the appropriate shutdown process.

- Shut down instrument: The instrument power turns off after about 30 minutes (although the main power remains on).
- Sleep: The instrument enters a sleep state, and restarts automatically at a specified date and time.

After selecting shutdown and clicking the OK button, the power of the PC can be turned off without waiting for the instrument power to turn off.

After sleep is selected and the OK button is clicked, the power of the PC can be turned off after the connection between the PC and the instrument is broken.

It is always necessary to follow this procedure to shutdown the instrument to prevent melting of the injection block. This procedure shuts the furnace off and allows the instrument fans to cool the instrument for thirty minutes before the main power shuts off.

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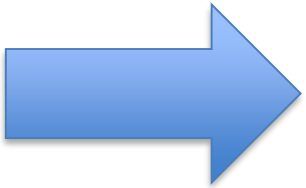
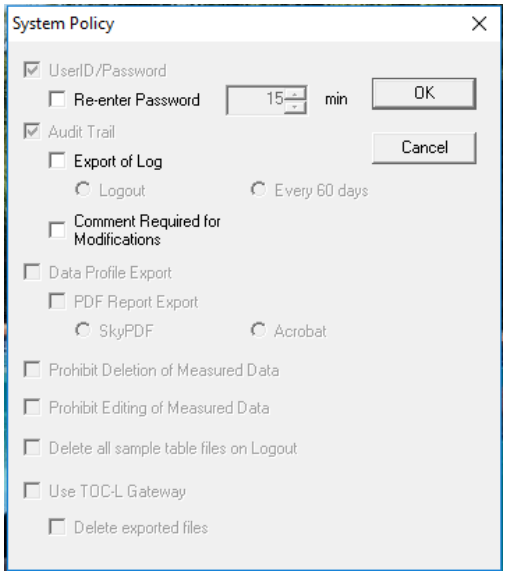
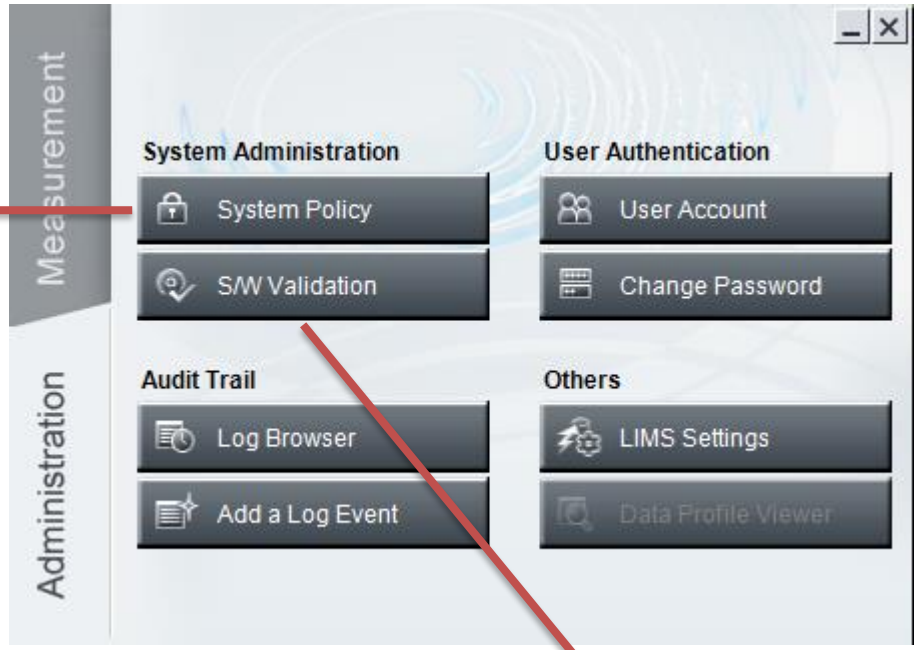
Software Administration Functions

Select the ‘TOC-Control L’ icon to open the software. Select ‘Administration’ to view the administration settings.



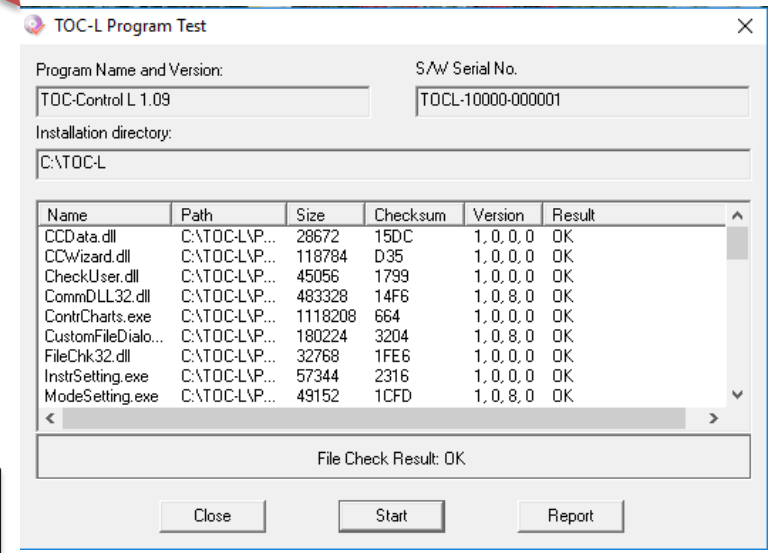
Software Administration Functions





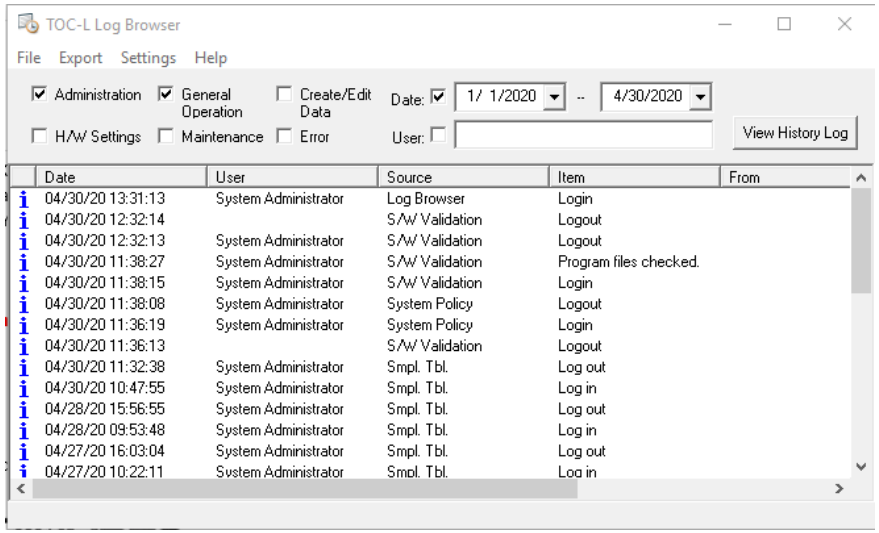
'System Policy' allows you to view the policies enacted when the software was installed

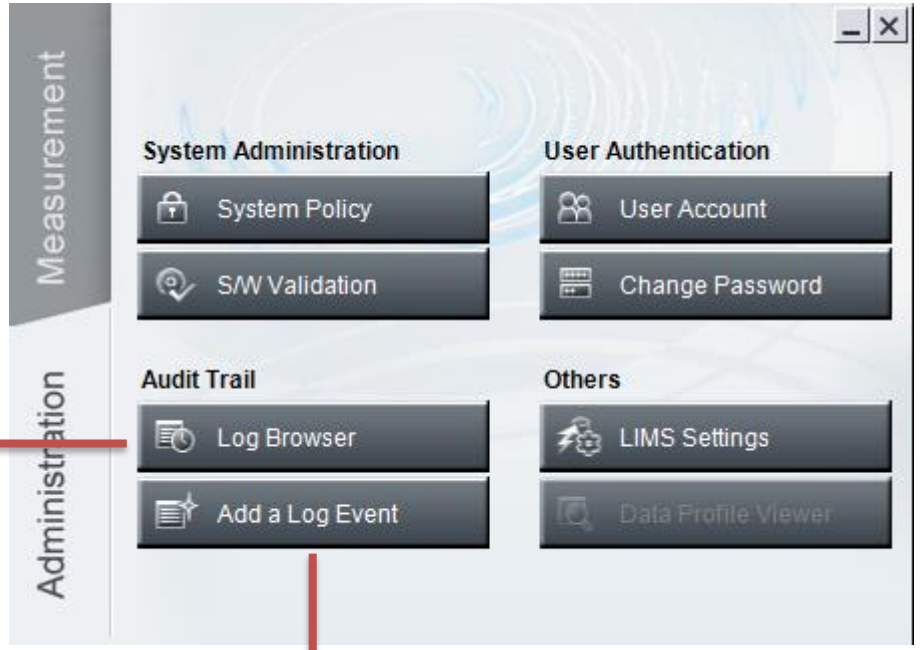
'S/W Validation' allows you to perform software program validation tests.



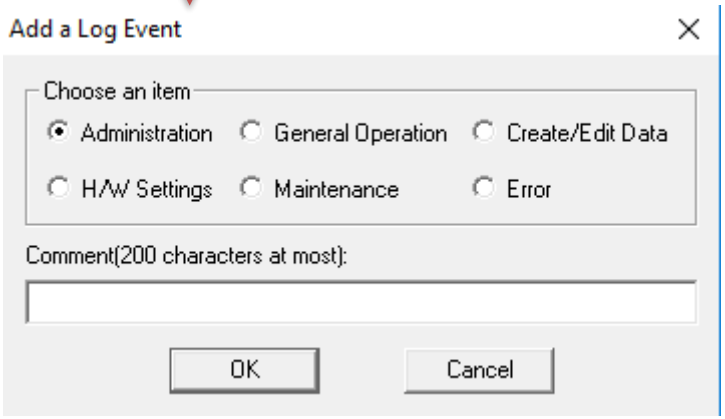
Software Administration Functions

If audit trail is enabled, log browser allows you to search the audit trails.

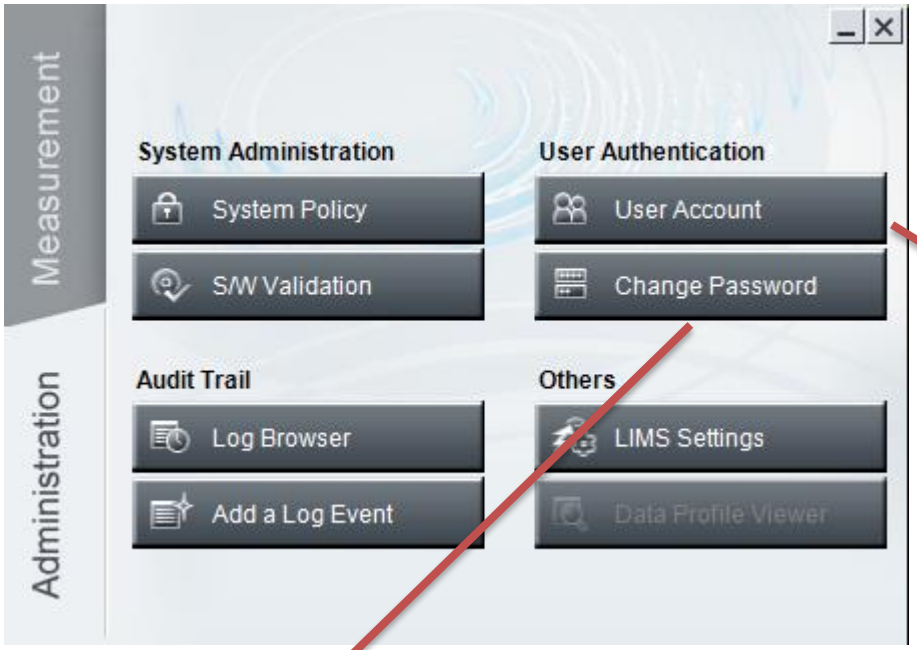




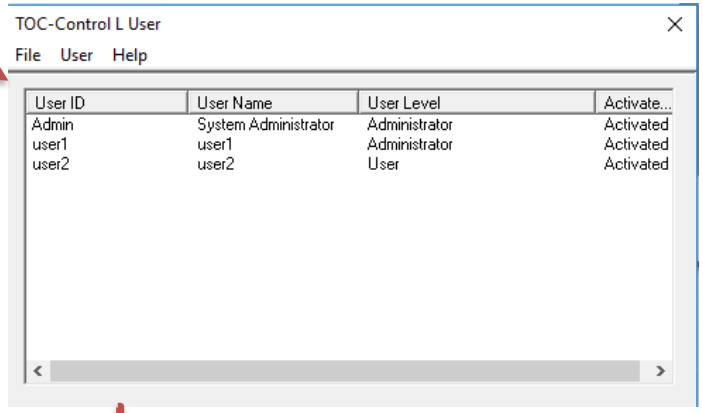
‘Add a Log Event’ allows you to manually add an event to the audit trail.

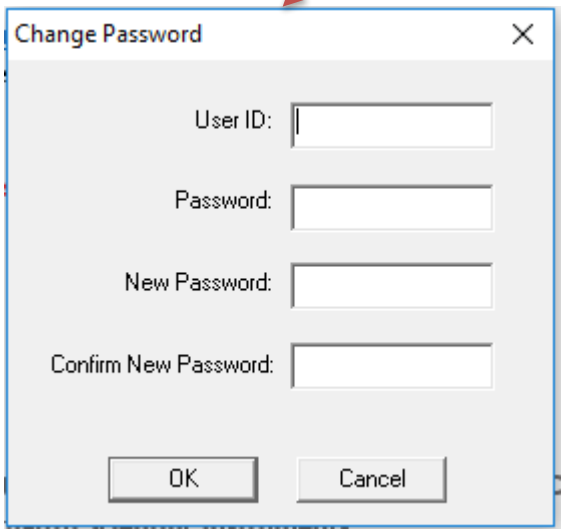


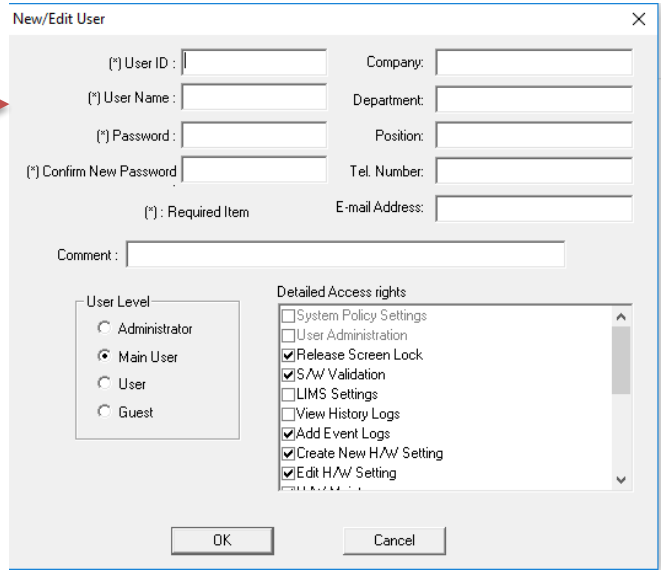
Software Administration Functions



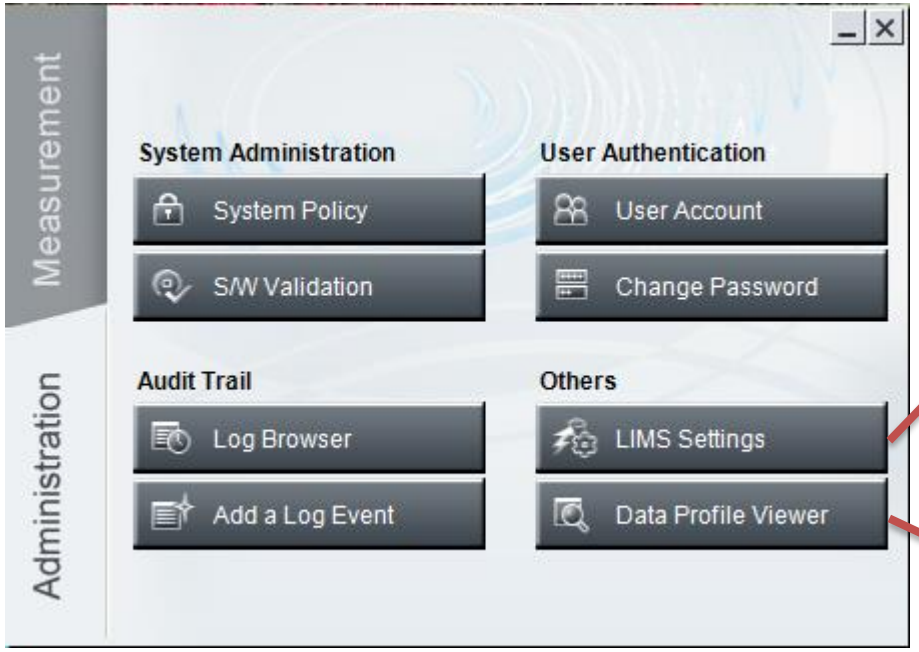
Shows a list of registered users and is where Administrators can add or delete user's. Different user levels can be assigned different rights within the software.

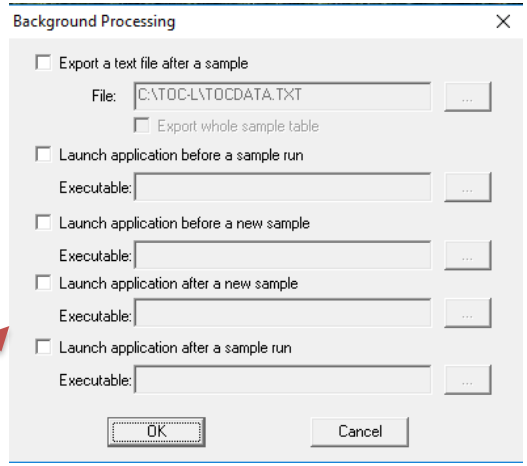






Software Administration Features

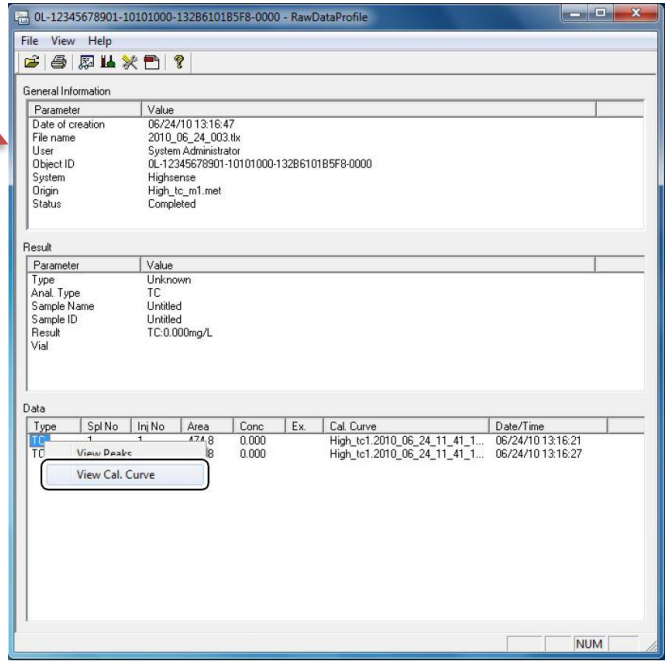




Allows you to customize LIMS Settings

The data profile can be exported from TOC-Control L software and displayed. Each row of the TOC-Control L sample table is output as an individual data profile.

Note: This function is only activated if Data Profile Export was selected in the System Policy dialog box.



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Section 5 / 5

Daily Checks

1. Verify there is enough water for analysis in the dilution water bottle. For low level analysis replace daily
2. Verify there is enough acid for analysis.
3. Check to make sure the drain pot is full and fill if necessary.

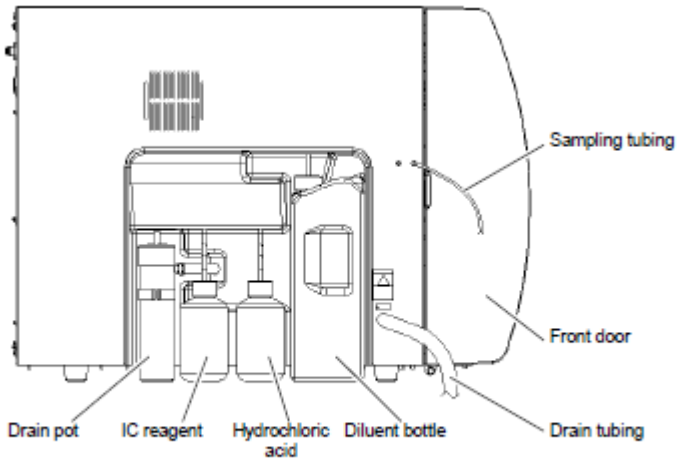


Figure 2.3 Left Side View

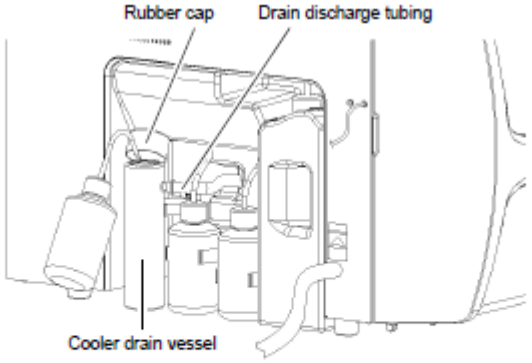


Figure 7.1 Drain Vessel Water Level

Verify that the water level in the drain vessel on the left side of the instrument is near the position of the overflow tube on the side of the drain vessel (within 10 mm) before using the instrument. Replenish as necessary with purified water (ion-exchange water).

Daily Checks

Check the level of water in the humidifier

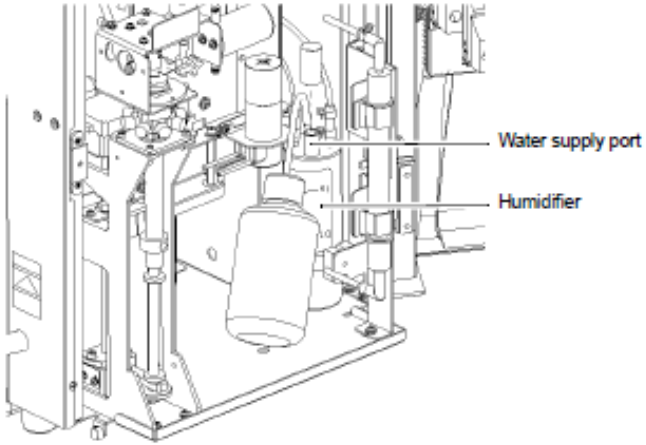


Figure 7.2 Humidifier Water Level

Verify that the water level in the humidifier is above the "Lo" mark. If the level is below the "Lo" mark, replenish by adding purified water through the water supply port on top of the vessel. Add water until the level reaches the "Hi" mark.

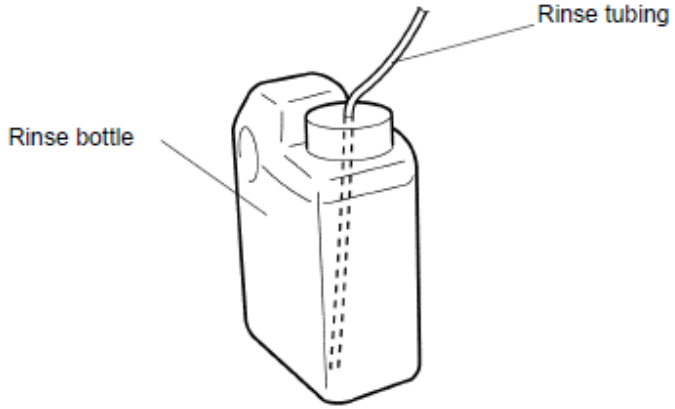
Perform the following inspections before conducting analysis with the ASI-L. If the following inspections are not properly conducted, air will be drawn into the ASI-L injection pump, preventing the delivery of rinse water.

- Check the rinse water level

  Verify that the water in the rinse bottle is above the 2-liter level mark. Add water if necessary.

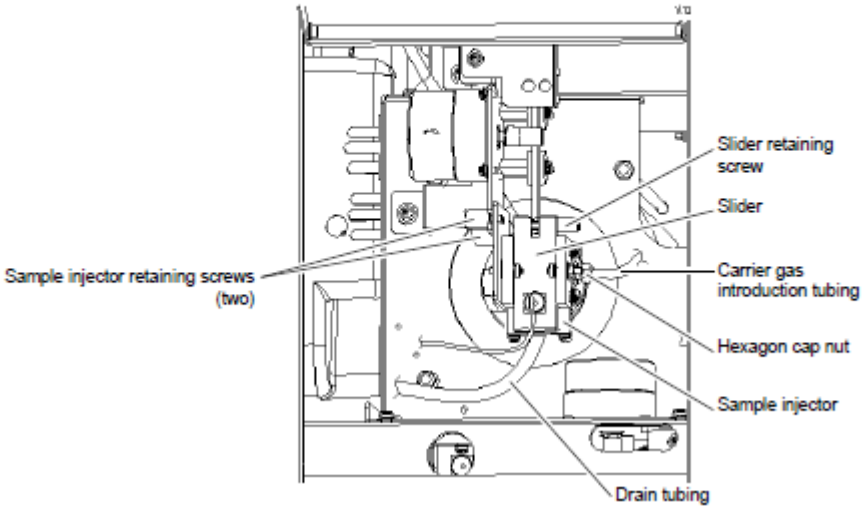
- Check the rinse tubing

  Verify that the tip of the rinse tubing reaches nearly to the bottom of the rinse bottle. Adjust the tubing position if necessary.

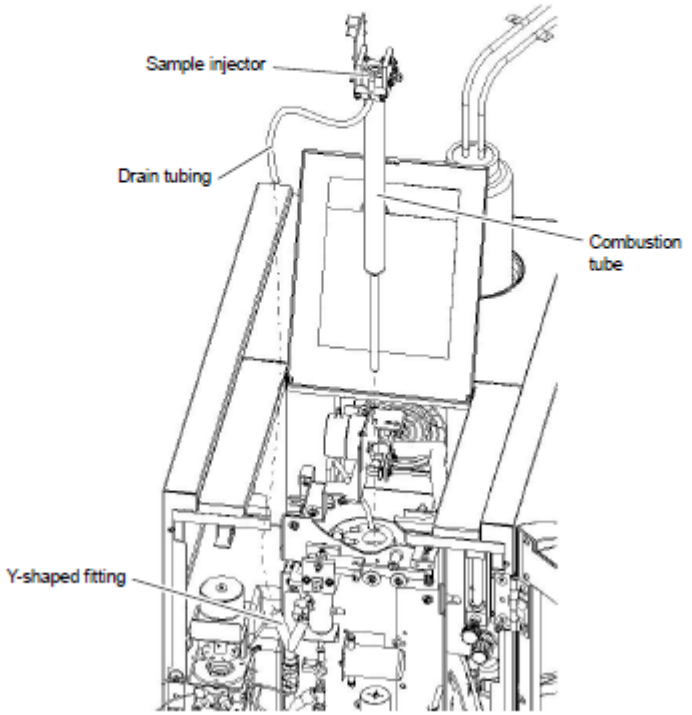


Replacing Catalyst

1. Open the door on top of the instrument, remove the slider retaining screws (knurled screws) from the sample injector, and pull the slider towards you and out of the sample injector.



2. Loosen the hexagon cap nut that joins the carrier gas introduction tubing (PTFE tubing) to the right side of the sample injector and then remove the tubing.
3. Loosen the hexagon cap nut on the PTFE coupling located on the cooling tubing inlet at the bottom end of the combustion tube.
4. Loosen the two knurled screws on the sample injector and remove the sample injector together with the combustion tube in the upward direction.



Replacing Catalyst

After the combustion tube has been removed the catalyst can be removed and washed or if it is consumed can be replaced with new. Quartz combustion tubes can be rinsed with DI water and reused until they break.

Washing Catalyst

Standard catalyst can be washed and reused several times using the following procedure.
High sensitivity catalyst cannot be washed and reused.

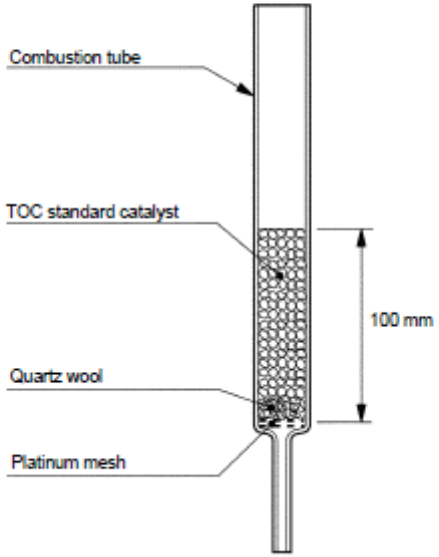
Procedure

1. Thoroughly wash the catalyst with tap water to remove any accumulated salts.
2. Neutralize the alkalis with dilute hydrochloric acid (5:1).
3. Thoroughly wash the catalyst with tap water to remove the hydrochloric acid.
4. Rinse with pure water (ion exchange water acceptable) and then dry.

Replacing Catalyst

Filling Combustion Tube with TOC Standard Catalyst

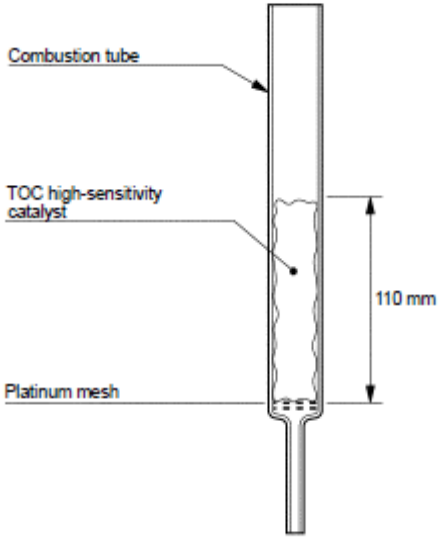
1. Insert two sheets of platinum mesh into the combustion tube.
2. Layer about 5 mm of quartz wool onto the platinum mesh using the catalyst filling rod.
3. Fill the TOC standard catalyst to a height of about 100 mm.



Filling Combustion Tube with TOC High Sensitivity Catalyst

Two cylindrical containers of catalyst are required for a single load.

1. Place two sheets of platinum mesh in the combustion tube.
2. Fit together the combustion tube opening and the opened end of the cylindrical catalyst container.
3. Use the catalyst filling rod to push catalyst into the combustion tube from the other opened end of the cylindrical catalyst container.
4. Transfer the contents of the two cylindrical catalyst containers to achieve a catalyst layer height of 110 mm in the combustion tube.



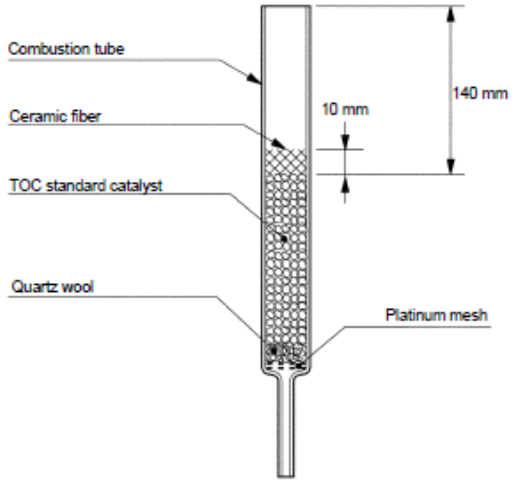
Replacing Catalyst

Filling Combustion Tube with Catalyst for TN Analysis

1. Insert two sheets of platinum mesh into the combustion tube.
2. Layer about 5 mm of quartz wool onto the platinum mesh using the catalyst filling rod.
3. Introduce the TOC standard catalyst into the combustion tube to a height of 140 mm from the top end of combustion tube.
4. Introduce 0.2 (± 0.1) g of ceramic fiber to cover the catalyst.
5. Lightly press down on the ceramic fiber using the catalyst filling rod to attain a thickness of 10 mm.

Note:
- The thickness of the ceramic fiber must be uniform, covering the entire surface of the catalyst, or a poor TN detection rate may result.
- The filling heights of the catalyst and ceramic fiber influence the TN detection rate, so it is important that the dimensions are close to those indicated below.

Catalyst: 140 mm from the top end of combustion tube
Ceramic fiber: 10 mm



Replacing Catalyst

Securely insert the top end of the combustion tube filled with catalyst into the hole on the bottom of the sample injector.

Fit the combustion tube into the hole in the center of the electric furnace and then insert the bottom end of the combustion tube into the PTFE coupling of the cooling tubing inlet. At this point, securely connect the drain tubing, which protrudes out of the sample injector, to the Y-shaped fitting.

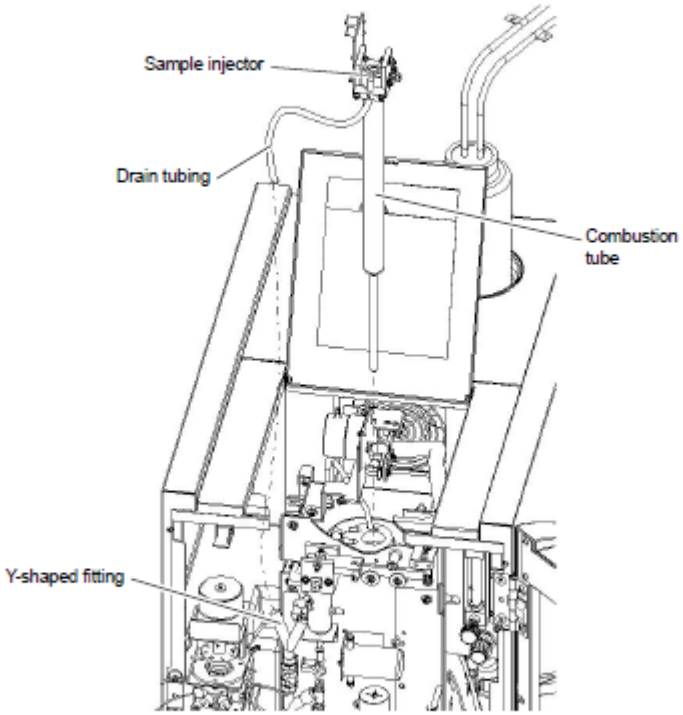
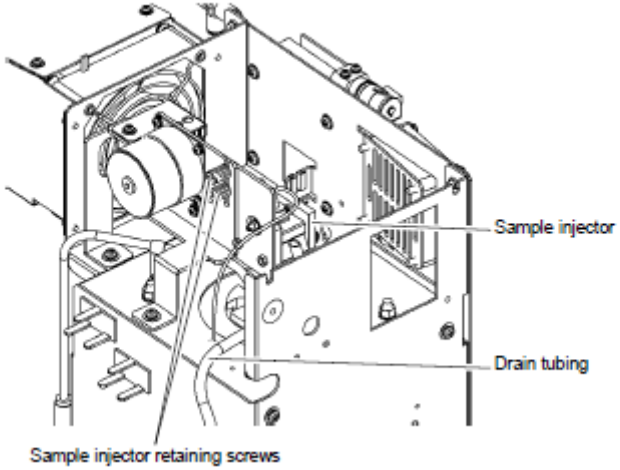


Figure 3.13 Installing the Combustion Tube

Secure the sample injector to the instrument body using the knurled screws.

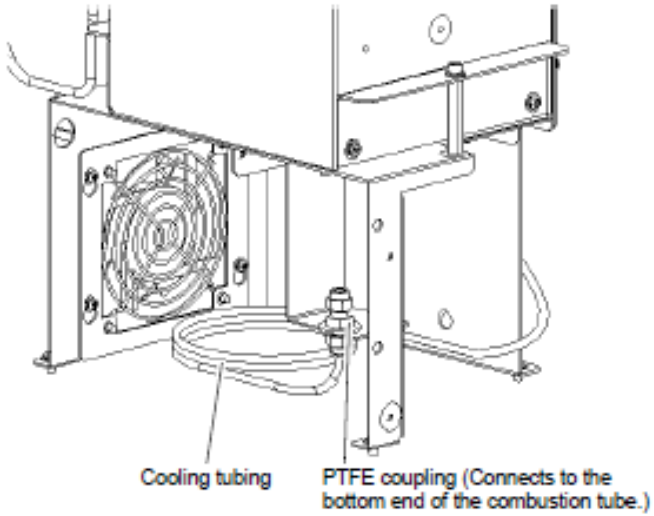
Reattach the slider and carrier gas introduction tubing to their original positions.



Replacing Catalyst

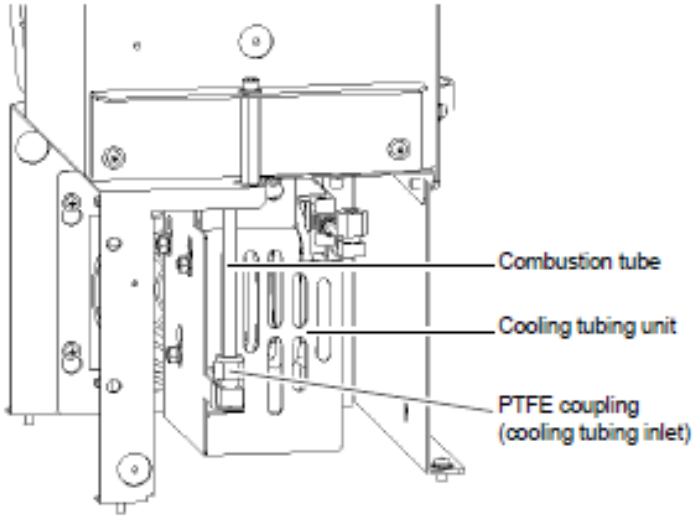
TOC-LC*N

Connect the bottom of the combustion tube to the PTFE coupling of the cooling tubing inlet. Carefully tighten the hexagon cap nut while holding the PTFE coupling in place with your finger.



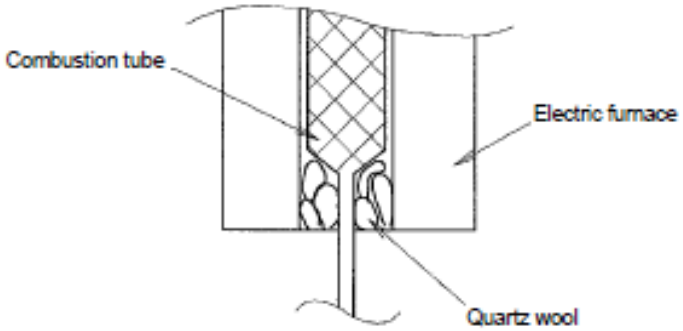
TOC-LC*H

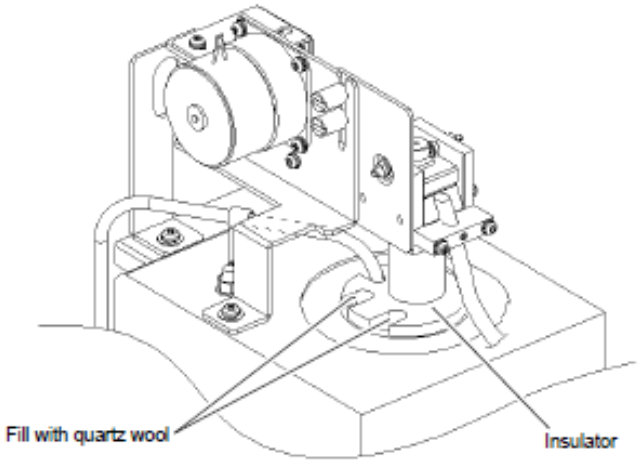
Connect the bottom of the combustion tube to the PTFE coupling of the cooling tubing inlet. Carefully tighten the hexagon cap nut while holding the PTFE coupling in place with your finger.



Replacing Catalyst

In order to prevent heat from escaping from the furnace, use the provided quartz wool to fill the gap between the bottom of the electric furnace and the combustion tube as well as the two notches in the insulator on top of the electric furnace.





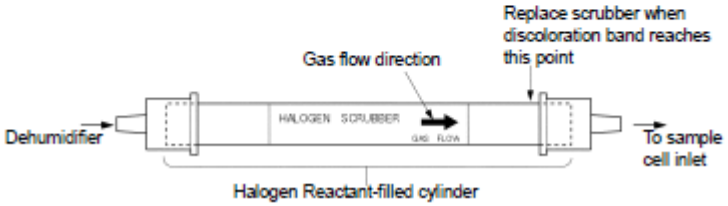
Replacing the Halogen Scrubber

Unclip the upper and lower clips that secure the halogen scrubber.

Disconnect the Viton connectors and remove the halogen scrubber. Release the white plastic clamp that secures the Viton connector while twisting it in the vertical direction.

Remove the film at both ends of the new halogen scrubber and connect it to the Viton connectors.

Secure the halogen scrubber with the two clips in its original position.



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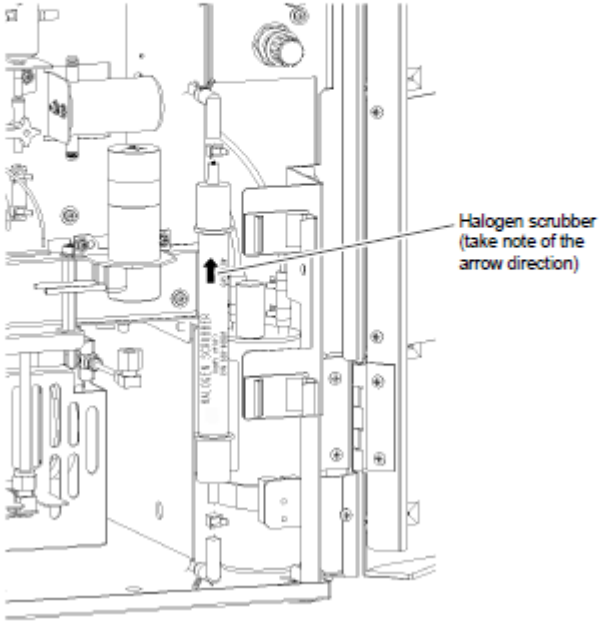
The baseline may fluctuate immediately after replacement. Allow the instrument to run for a short time until the baseline stabilizes. Stabilization normally occurs within one hour.

Used halogen scrubbers should be disposed of according to local industrial waste disposal regulations. It must be clearly disclosed using an industrial waste manifest that the halogen scrubber contains copper.



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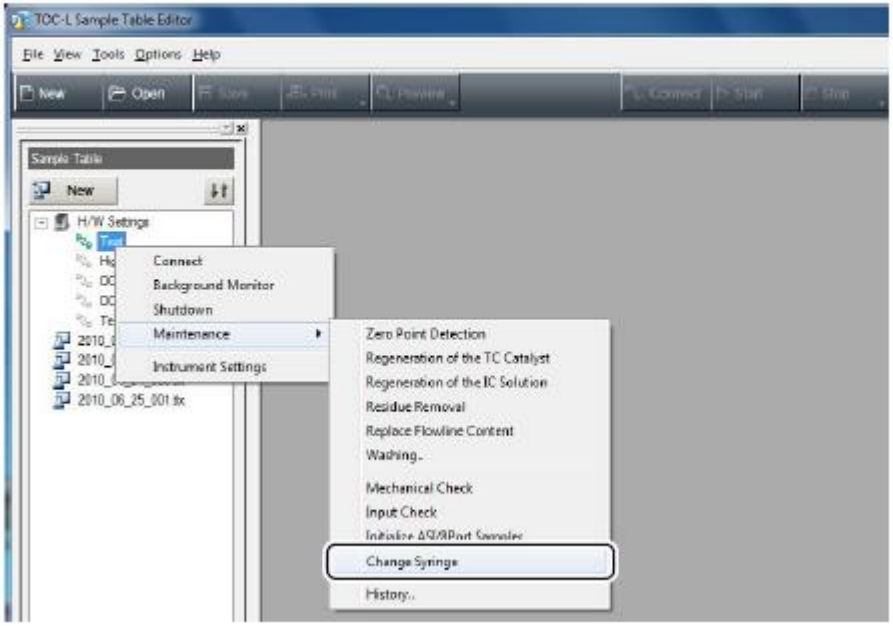
Replacing the Syringe / Plunger Tip

Open the Sample Table tab of the file viewer. Right-click on the connected system configuration icon, and click Maintenance – Change Syringe.



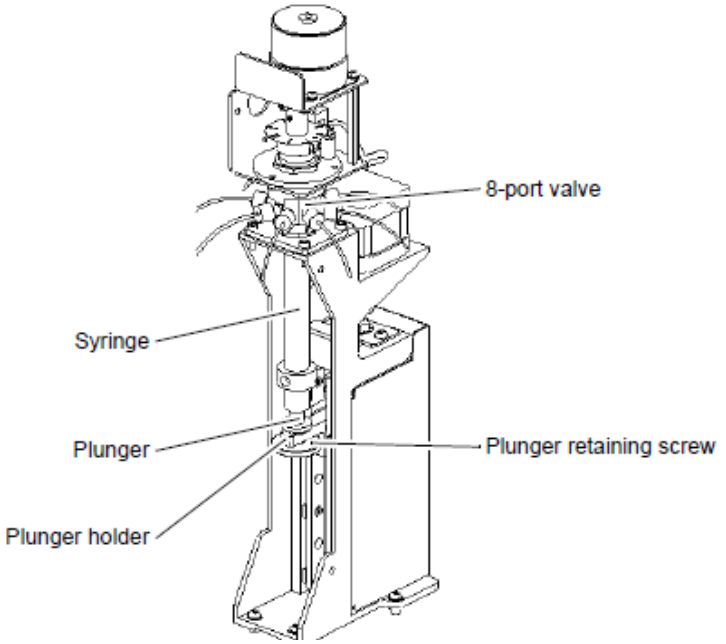
The Syringe Change window is displayed.

Click Preparation Start.

The syringe replacement preparation process begins. The progress bar blinks while the operation is in progress.

Disconnect the sparge gas line from the syringe, remove the plunger retaining screw, and remove the syringe.

Reverse the above procedure to install the new syringe and connect the sparge gas line. At this point, the plunger retaining screw is not completely tightened. Tighten the retaining screw so that the plunger can still be slightly moved forwards and backwards as well as left and right.



The plunger is secured to the plunger holder with the plunger retaining screw through a spring washer. Be careful not to lose the spring washer.

Replacing the Syringe / Plunger Tip

Click  to move the syringe to its uppermost position.

Once the syringe reaches the top position, hold the plunger vertical and tighten the plunger retaining screw to secure it to the plunger holder.

Click Finish Replacement. The syringe pump zero point detection operation is executed automatically.

Click Close.

Replacing the Syringe / Plunger Tip

Remove the syringe

Use a sharp knife to make two or three incisions at the bottom of the plunger tip

Grasp the plunger tip with a pair of pliers and remove it from the plunger. The O-ring that is held in place by the plunger tip also comes off. DO NOT reuse this O-ring.

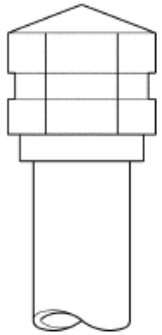
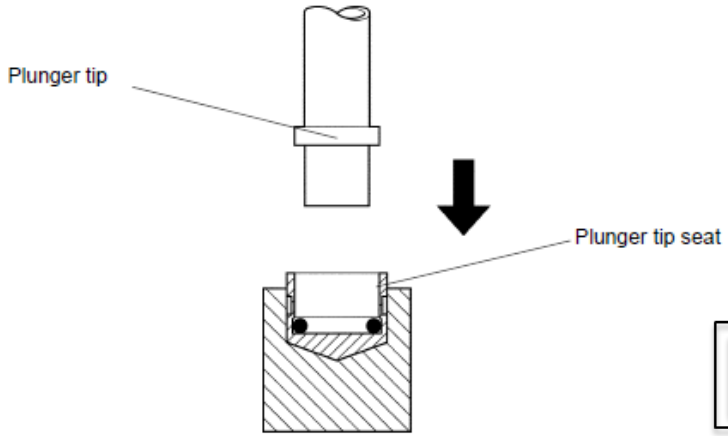


Figure 7.8 Removing the Old Plunger Tip

Pushing down on the plunger, press the leading edge of the new plunger tip straight into the plunger tip seat.



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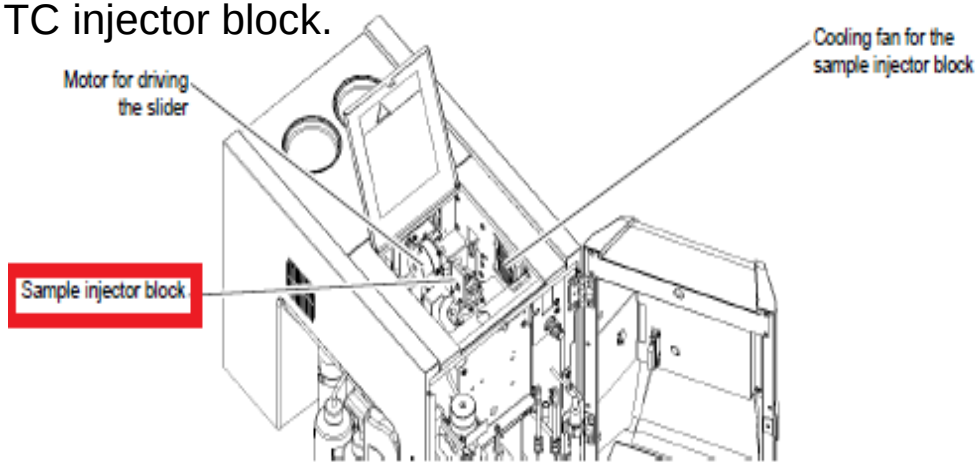
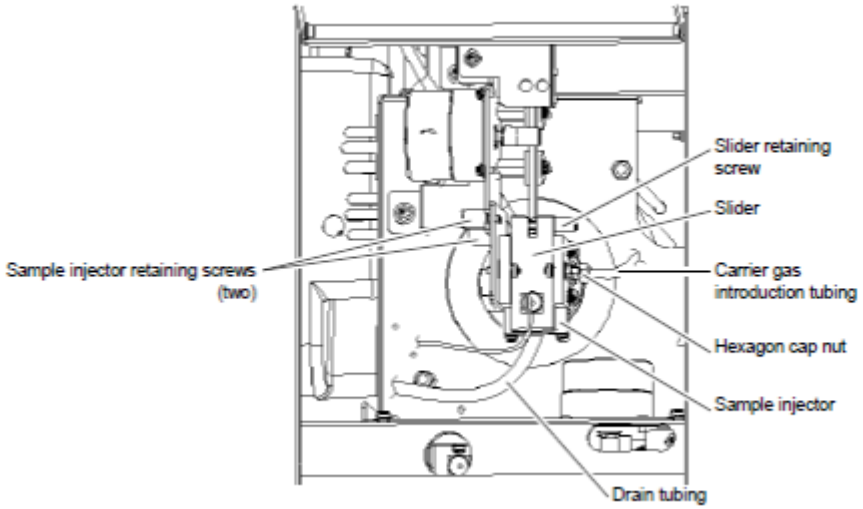
Section 3 / 3

Replacing the TC Slide Injection O-rings

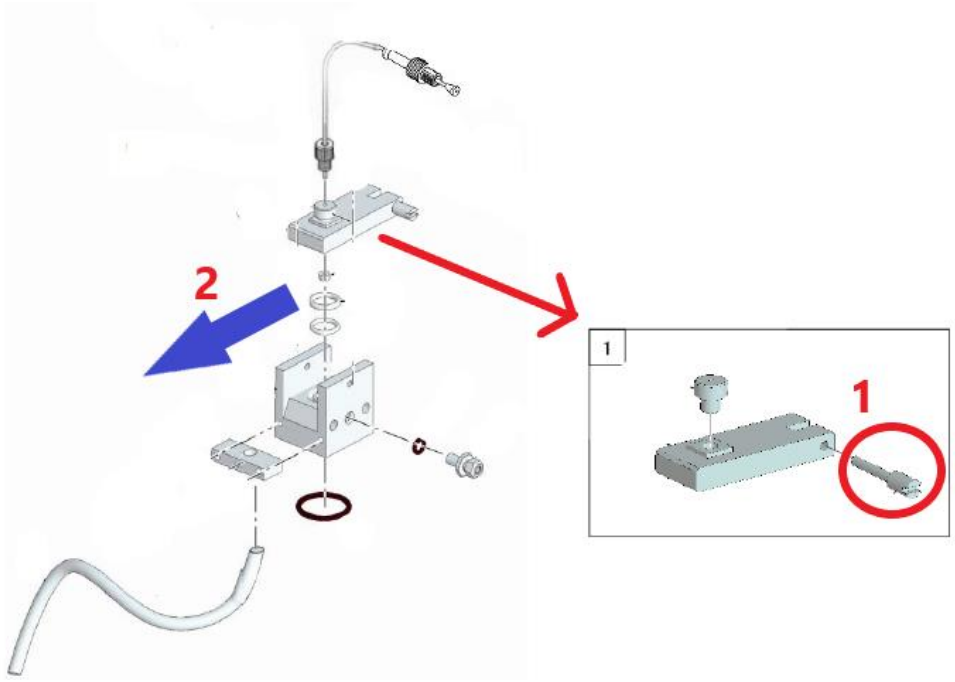
Open the lid on the top of the TOC and locate the TC injector block.





1. Remove the thumb screw connecting the Teflon slider to the motor drive.

2. Pull the Teflon slider forward and out of the sliding injection block.

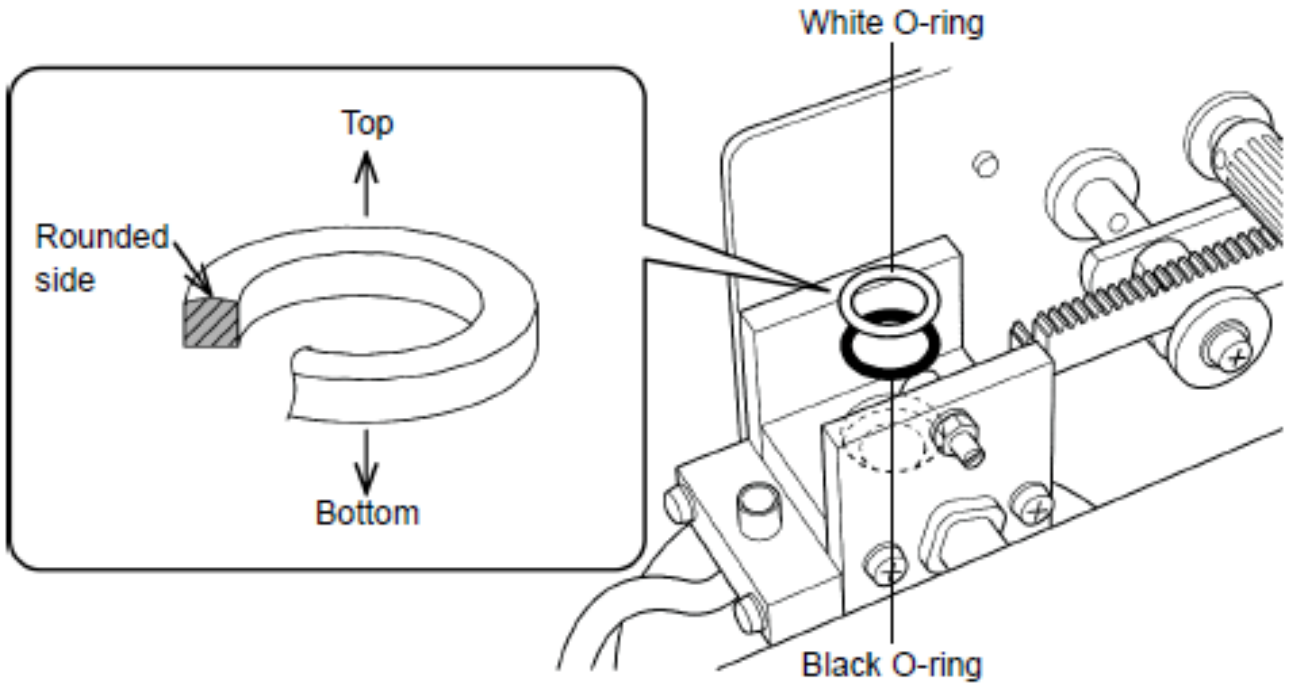


Section 1 / 3

Replacing the TC Slide Injection O-rings

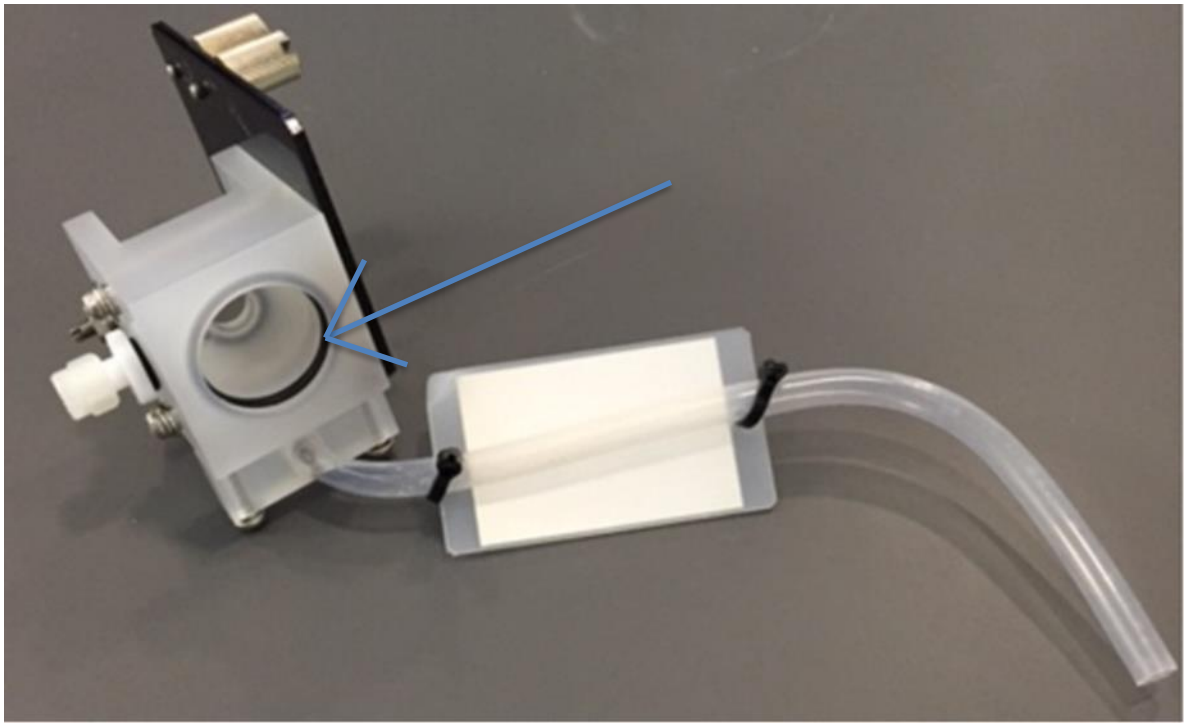
Two types of o-rings are stacked between the injection block and the slider of the sample injector. If a gas leak develops in this area, replace both o-rings.

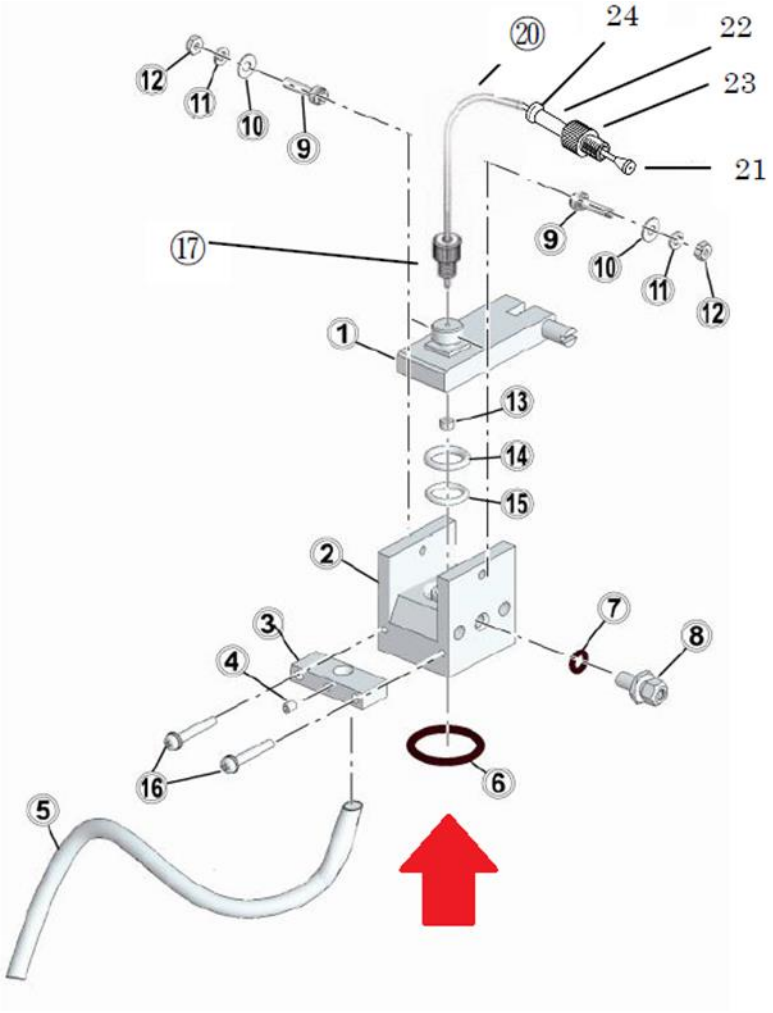
- White Upper O-ring: O-ring, PTFE (P/N S638-15025)
- Black Lower O-ring: O-ring, 4DP10A (P/N S0936-11209-84)



Replacing the TC Slide Injection O-rings

If a gas leak develops between the injector block and the combustion tube, replace the o-ring there as well.





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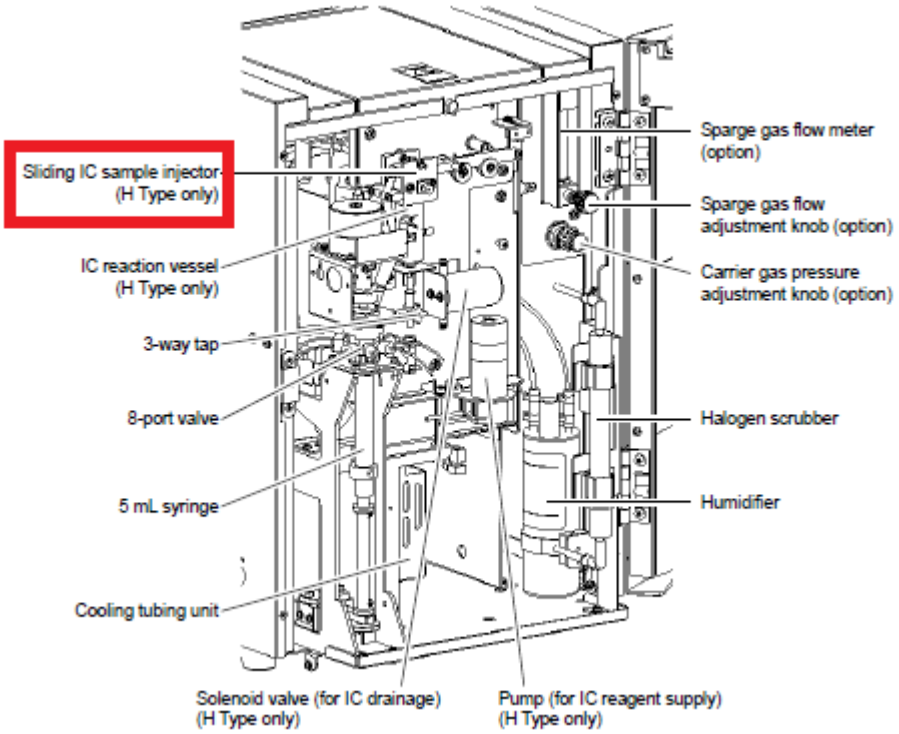
Section 3 / 3

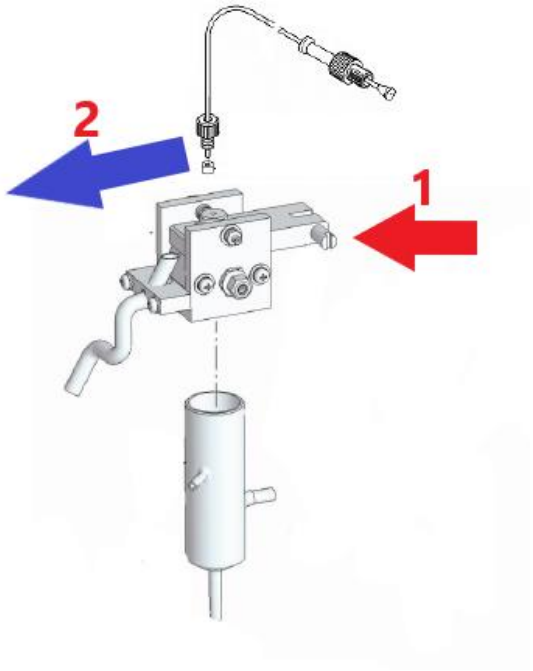
Replacing the IC Slide Injection O-rings

Open the front door of the TOC and locate the sliding IC sample injector



1. Remove the thumb screw connecting the PTFE slider to the motor drive.

2. Pull the PTFE slider forward and out of the sliding injection block.

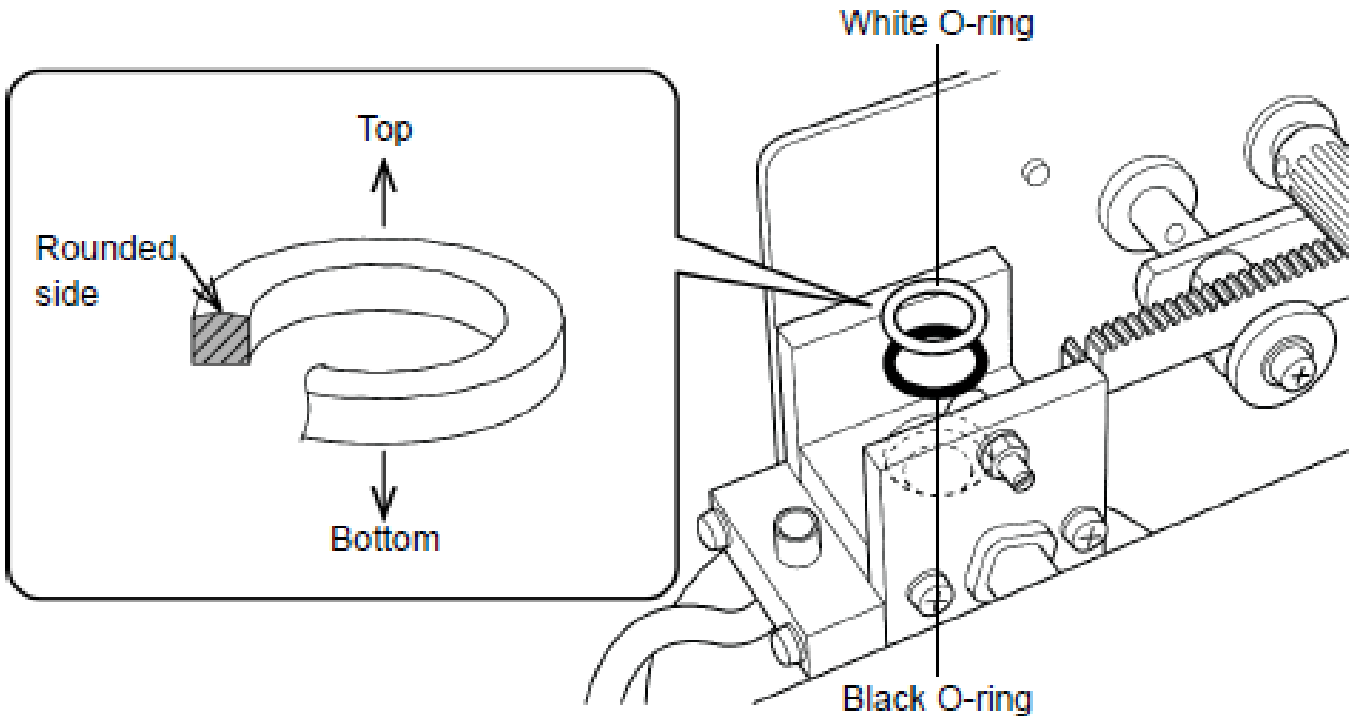


Section 1 / 2

Replacing the IC Slide Injection O-rings

Two types of o-rings are stacked between the injection block and the slider of the sample injector. If a gas leak develops in this area, replace both o-rings.

- White Upper O-ring: O-ring, PTFE (P/N S638-15025)
- Black Lower O-ring: O-ring, 4DP10A (P/N S0936-11209-84)



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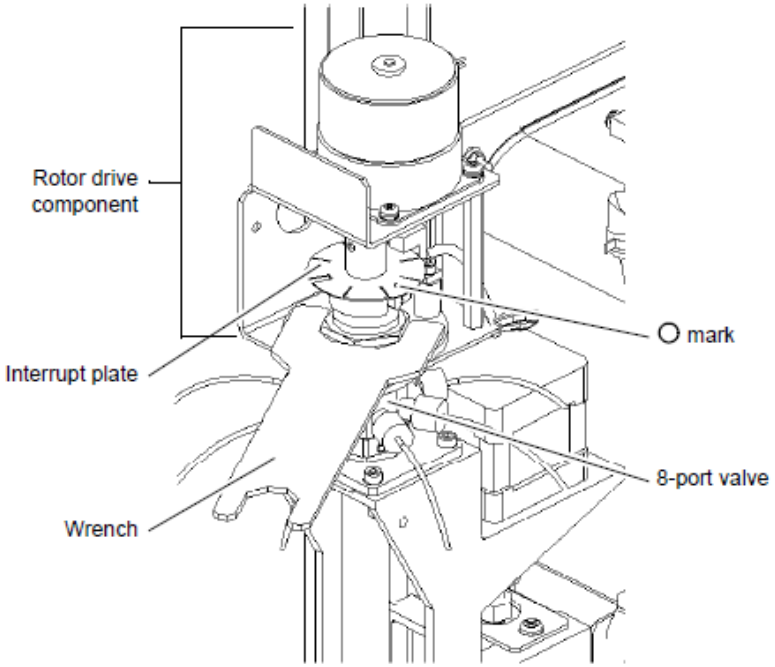
Replacing the Rotor in the 8-port Valve

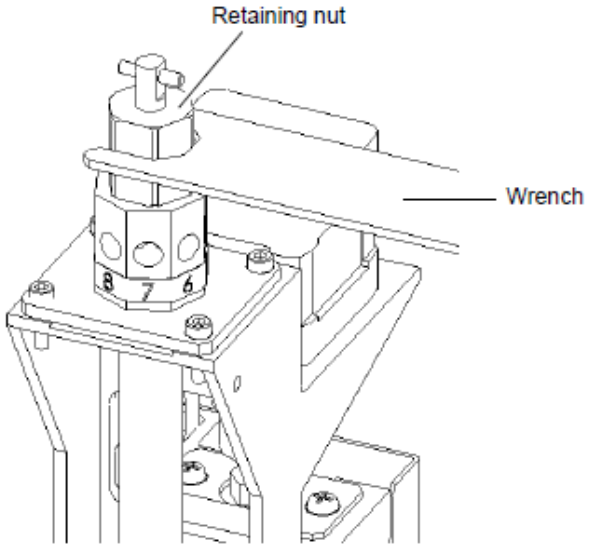
The ○ mark on the 8-port valve interrupt plate indicates the orientation of the rotor hole (stream direction). Take note of which port number the ○ mark is pointing towards.

Remove the nut using the provided wrench.



Pull up to remove the rotor drive component. Hang it on the screw on the right side of the plunger.

Loosen the retaining nut using the provided wrench.



Replacing the Rotor in the 8-port Valve

Remove the spring-equipped rod.

Grasp the rotor with pliers and pull it out.

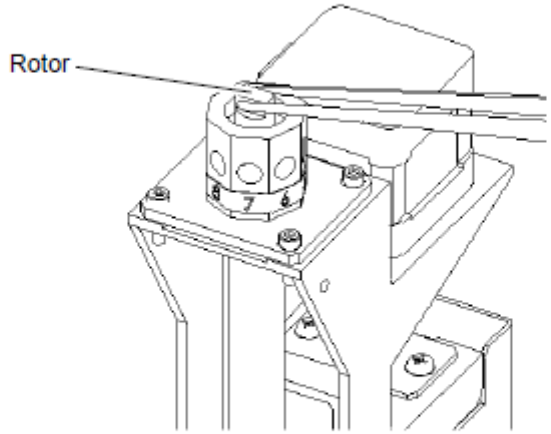
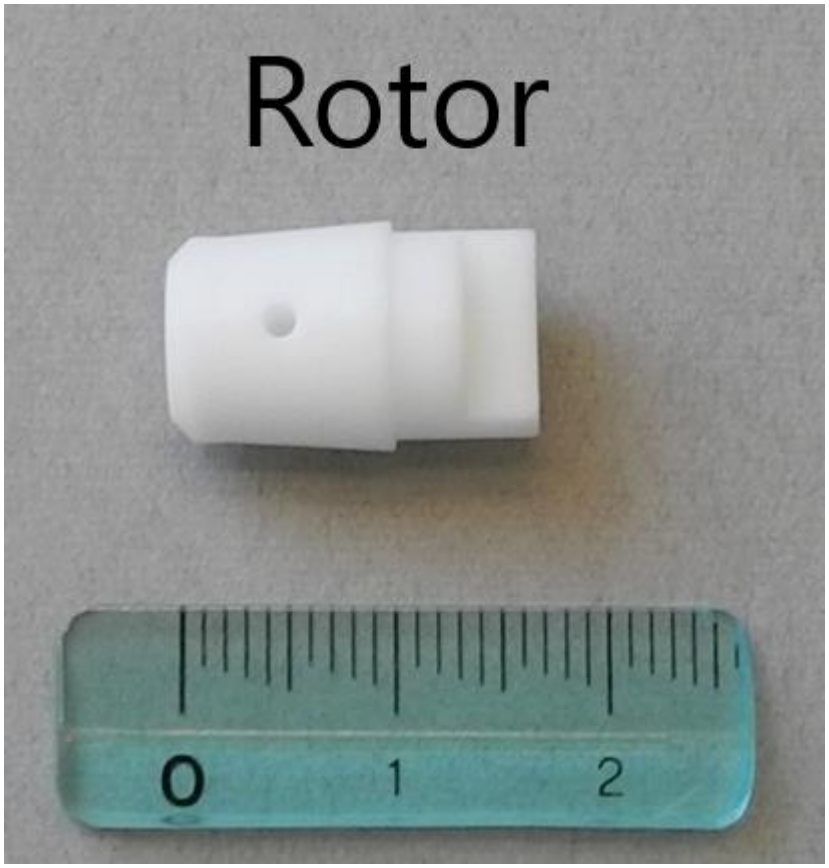


Figure 7.13 Removing the Rotor

Insert a new rotor, taking care that the rotor hole matches original orientation.

Reinsert the spring-equipped rod and tighten the retaining nut.

Replace the rotor drive component, and tighten the nut.



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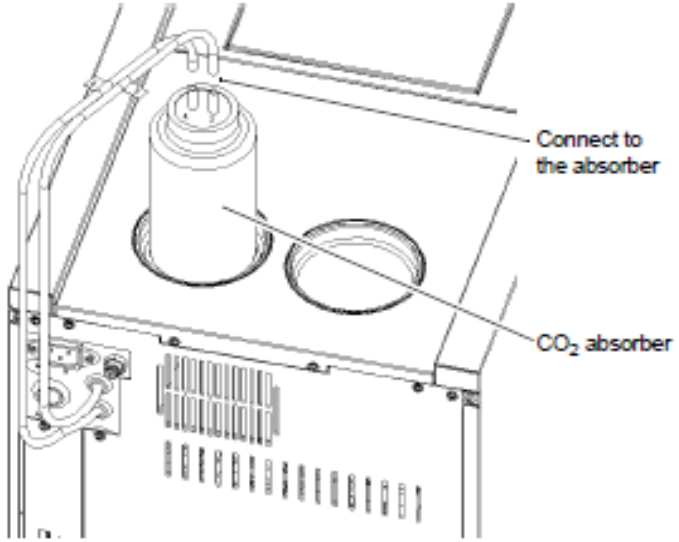
Section 2 / 2

Replacing the CO2 Scrubber

Disconnect the flexible tubing from the old CO2 scrubber and dispose of the scrubber per your local waste disposal regulations. Follow instructions below for installing the new scrubber.

1. Cut the tips of the 2 rigid tubes in the lid of the CO_2 absorber with a knife.
2. Connect the flexible tubing that exits the rear panel of the instrument (near the top) as follows:
   Tubing labeled "L": Connect to tube labeled "L" on CO_2 absorber.
   Tubing labeled "S": Connect to tube labeled "S" on CO_2 absorber.
   Note: The flexible tubing should not be bent.
3. Place the CO_2 absorber in the holder on the upper rear of the instrument.



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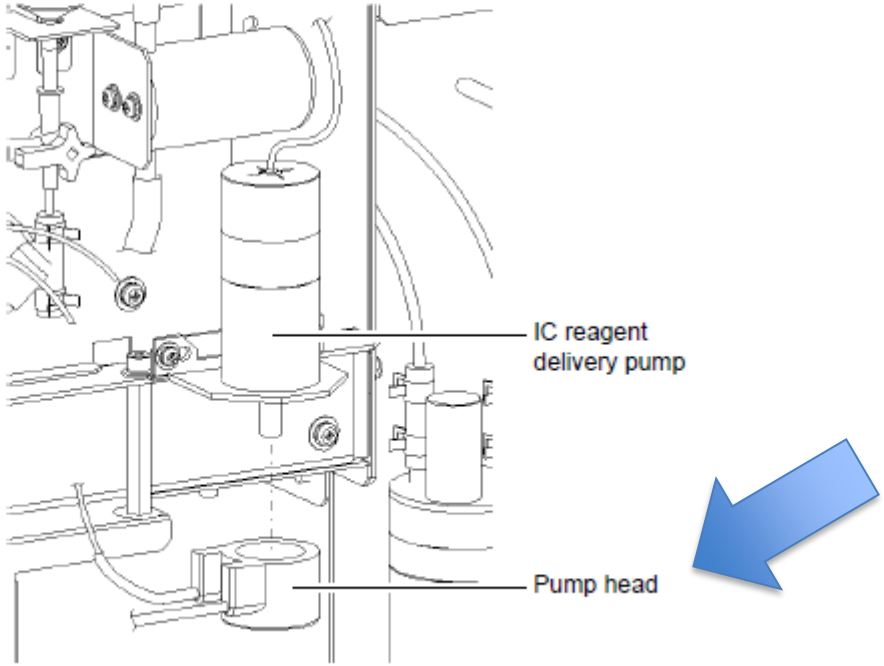
Replacing the IC Vessel Pump Head

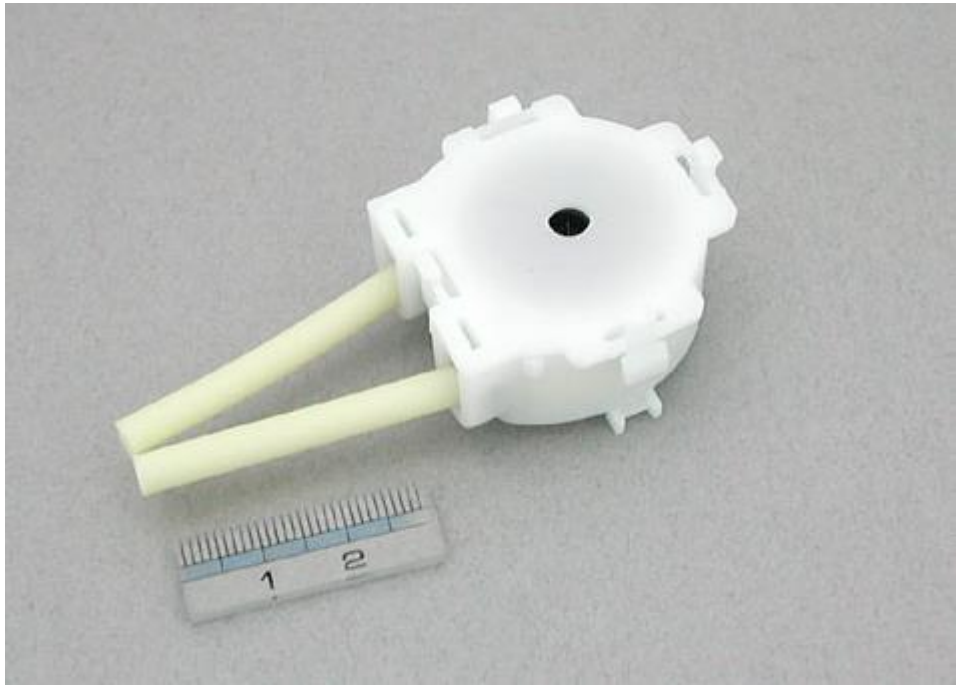
Grasp the front and rear stops of the pump head by hand. Pull the white pump head from the black pump body.

Disconnect both of the yellow rubber tubes on the left side of the pump head from the PTFE tubing.

Connect the new rubber tubing to the PTFE tubing of the pump head.

Replace the pump head by following the above procedure in reverse.





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Replacing the ASI Rinse Pump Head

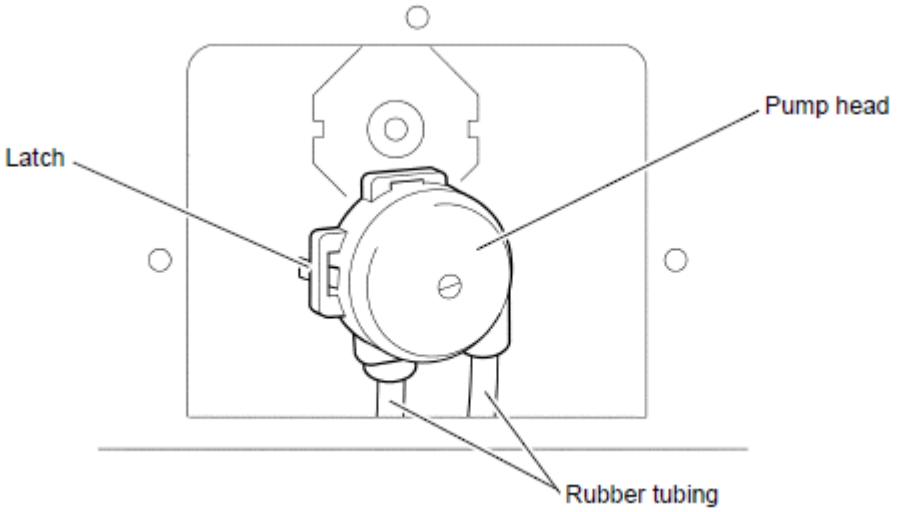
Remove the rectangular pump access cover on the back left side of the ASI-L.

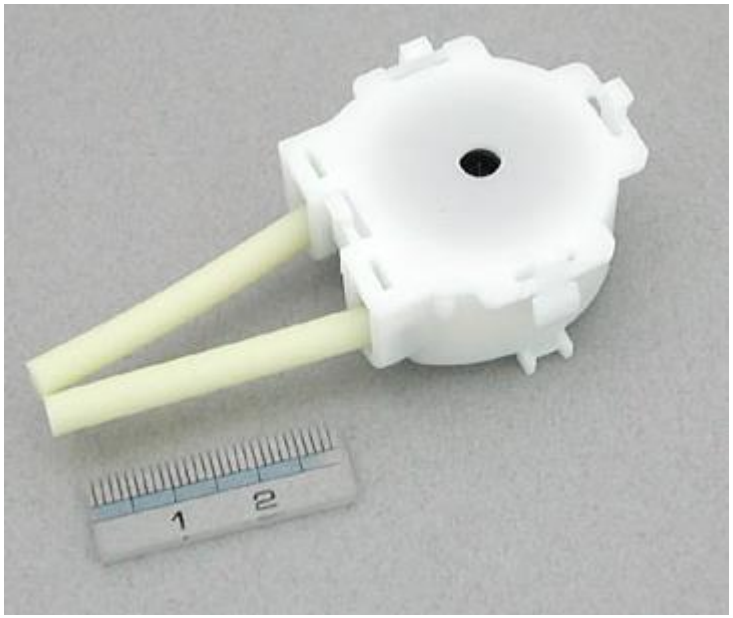
Grasp the latch on the pump head by hand and pull the white pump head from the black pump body.

On the left side of the pump head, disconnect both of the yellow rubber tubing from the PTFE tubing.

Connect the new rubber pump head tubing to the PTFE tubing.

Replace the pump head by following the above procedure in reverse and then replace the pump access cover.





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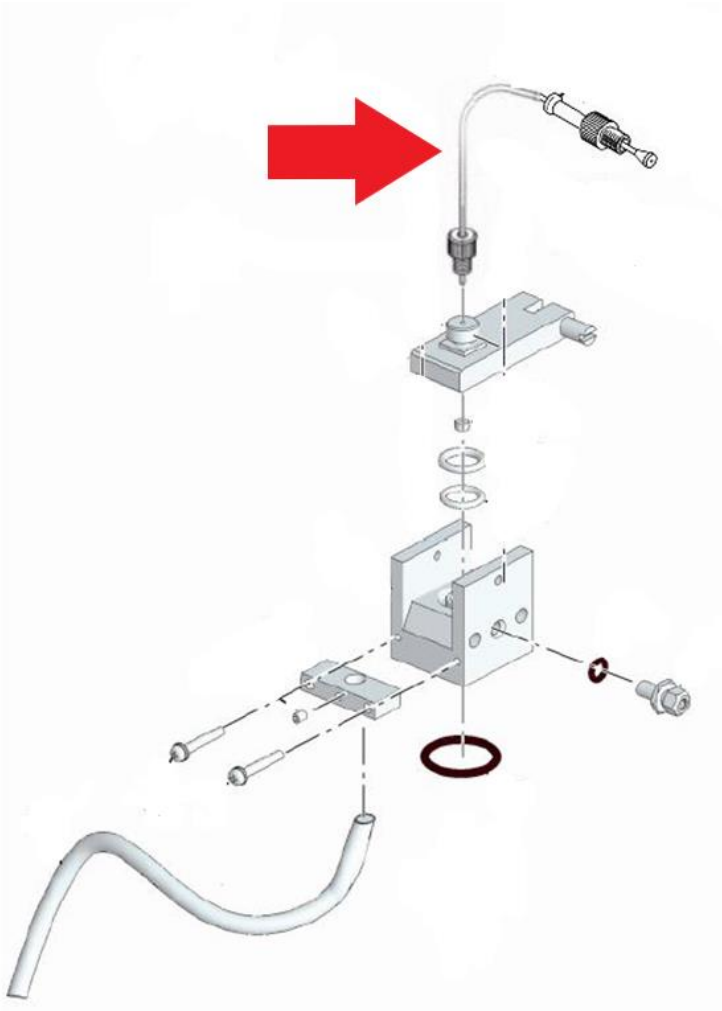
Replacing the TC Injection Tubing

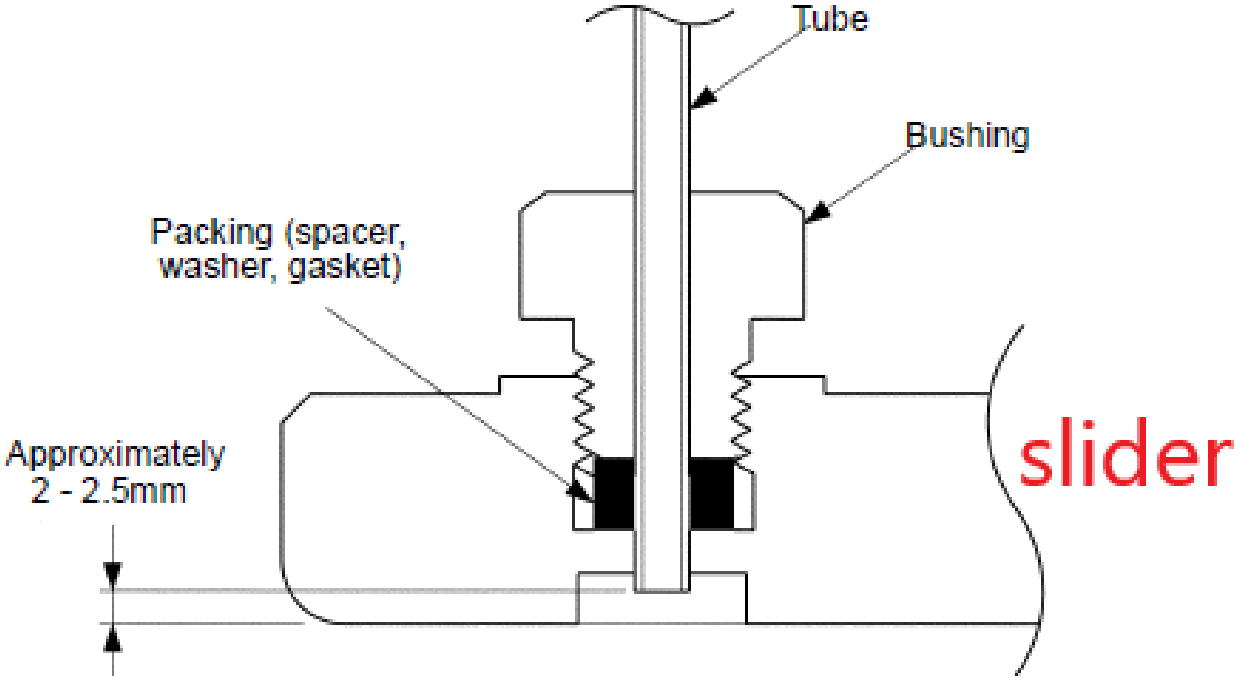
The TC injection tubing should be checked and replaced periodically. This tubing connects from port 7 on the valve to the slider on top of the injection block. If the tubing looks pinched or kinked it should be replaced.

When replacing the tubing it is important to make sure it is installed correctly in the slider and that it is injecting sample straight down onto the catalyst bed. If the sample injection sprays outward and hits the side of the combustion tube it will result in poor reproducibility



Replacing the TC Injection Tubing

Salt or other substances adhering to the tip of the tubing after rinsing can allow large droplets of sample to remain on the tip of the tubing after injection. A deformed tip on the sample injection tubing (a beard-like protrusion, for example) may cause the sample to be injected obliquely. In either case, reproducibility is adversely affected and the sample injection tubing should be replaced. Another option available only if there is sufficient length of tubing remaining is to use a sharp cutting tool at a 90° angle to cut off 2 - 3 mm from the tip of the currently used sample injection tubing.



Replacing the TC Injection Tubing

The manner in which sample is discharged from the TC or IC sample injection tubing has a large influence on reproducibility. The sample should be injected as vertically as possible onto the TC catalyst or into the IC reaction vessel. If the sample is scattered or injected obliquely, detach the sample injection tubing from the slider, and remove any obstruction that may be adhering to the opening at the tip of the sample tubing.

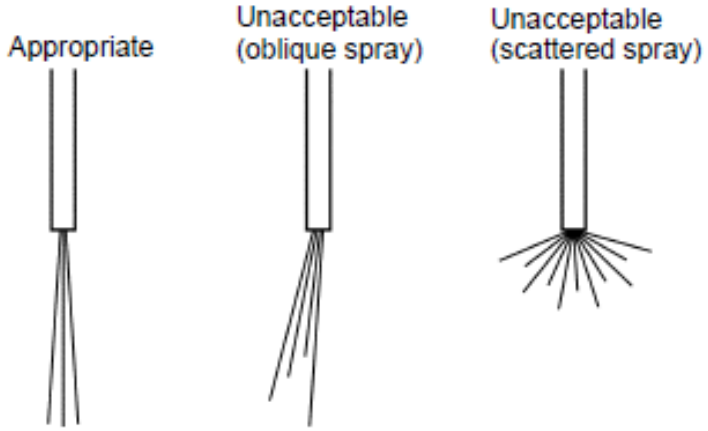


Figure 7.22 Sample Injection Patterns from Sample Tubing

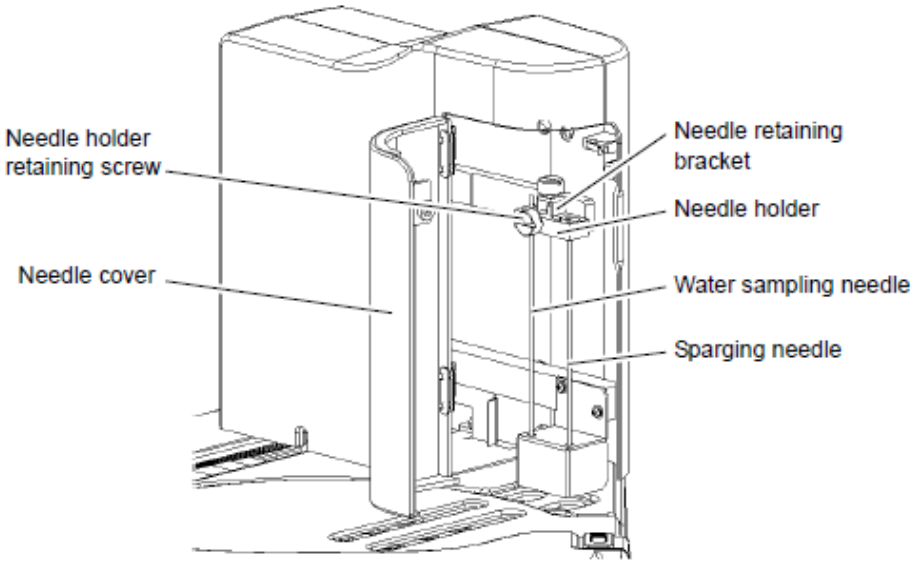
Ensure that the tip of the tubing does not protrude beyond the bottom surface of the slider. The O-ring or other seal parts will be scratched if the tubing protrudes, resulting in gas leaks. The sample is not properly injected unless the tip of the tubing extends into the depression in the bottom of the slider.

Check the state of the injection for TC analysis by looking through the TC combustion tube while it is attached to the instrument. The sample injection tubing can be detached from the slider, and water can be injected for inspection using the functions available in the Instrument>Maintenance>Mechanical Check screen.

Replacing the ASI Needle

Open the needle cover door towards the left.

Remove the needle holder retaining screw and then remove the needle holder from the arm.



Insert the needle into the opening in the needle holder.

Secure the needle by turning the retaining bracket and tightening the screw at the top of the needle holder.

Note: There are two openings; the left opening is for the sample tubing and the right opening is for the sparge tubing.

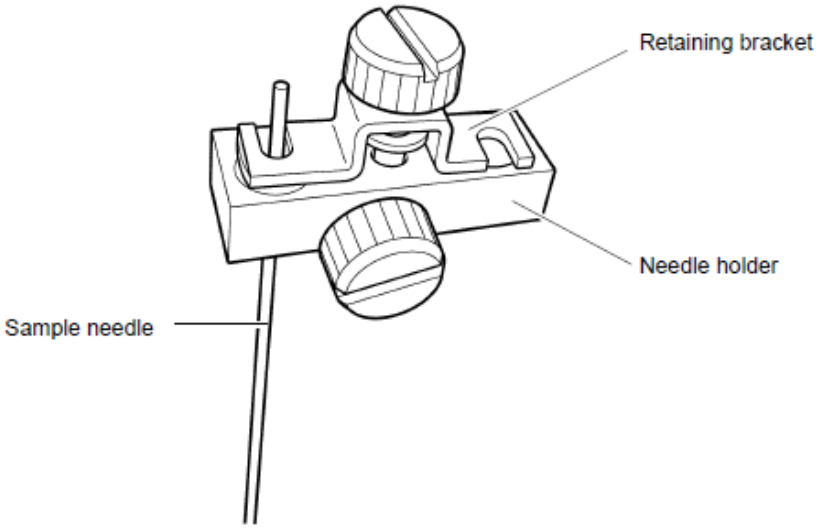


Figure 8.12 Installing the Needle

Feed the water sampling tubing through the hole in the needle cover and pull it inside the needle cover. When using the external sparge kit, pull the sparging tubing inside the needle cover in the same manner.

Fit the water sampling tubing or sparging tubing to the top of the needle and attach a retainer.

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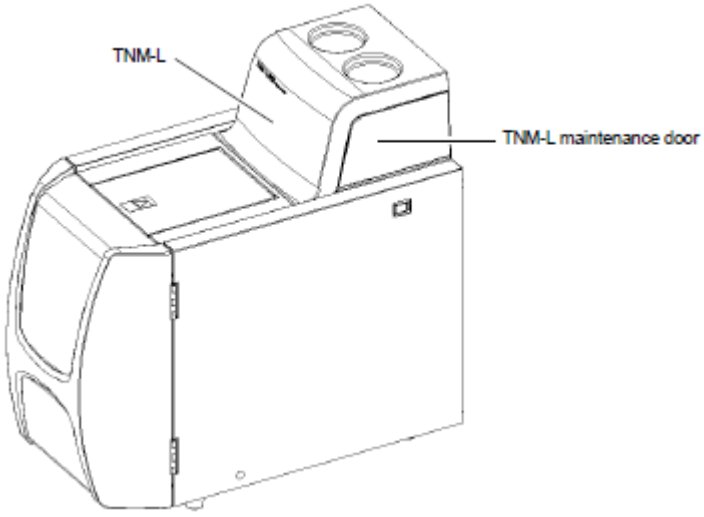
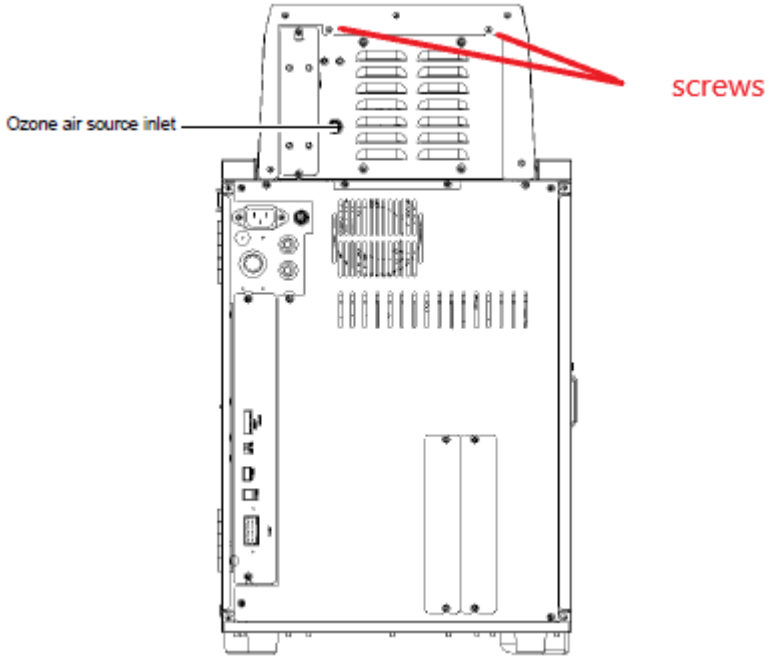
Replacing the Detector Gasket

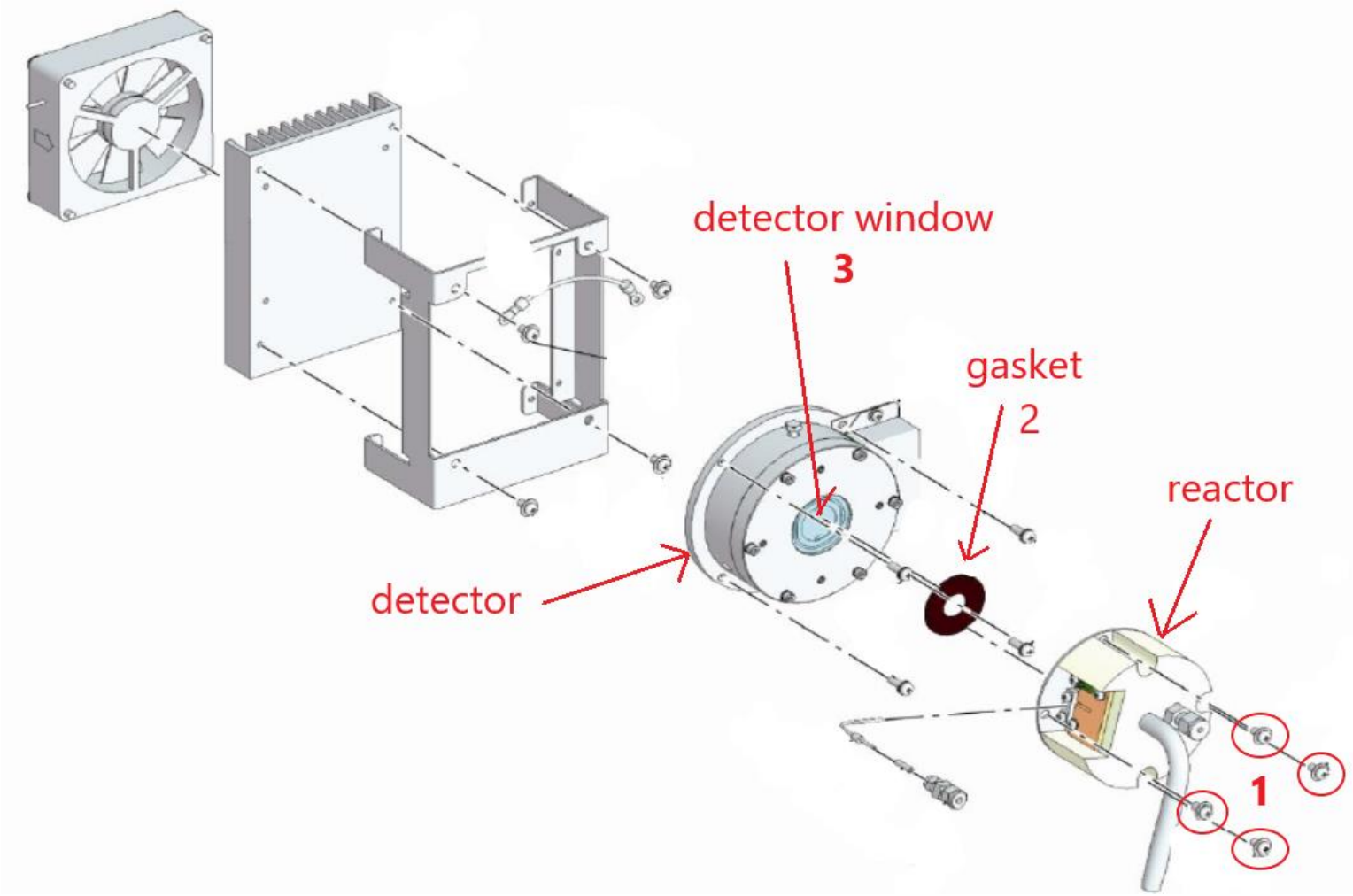
1. Locate the TNM-L unit mounted to the top of the TOC.
2. Loosen the two screws on the back of the TNM-L that hold the cover on.
3. Lift the cover up and remove.





Replacing the Detector Gasket

1. Remove theses four screws and pull the reactor forward and away from the detector
2. Remove the old gasket and replace with new making sure the flat side is facing the detector window
3. Clean the detector window with a Kim Wipe and some alcohol
4. Reconnect the reactor to the detector and replace the TNM-L cover



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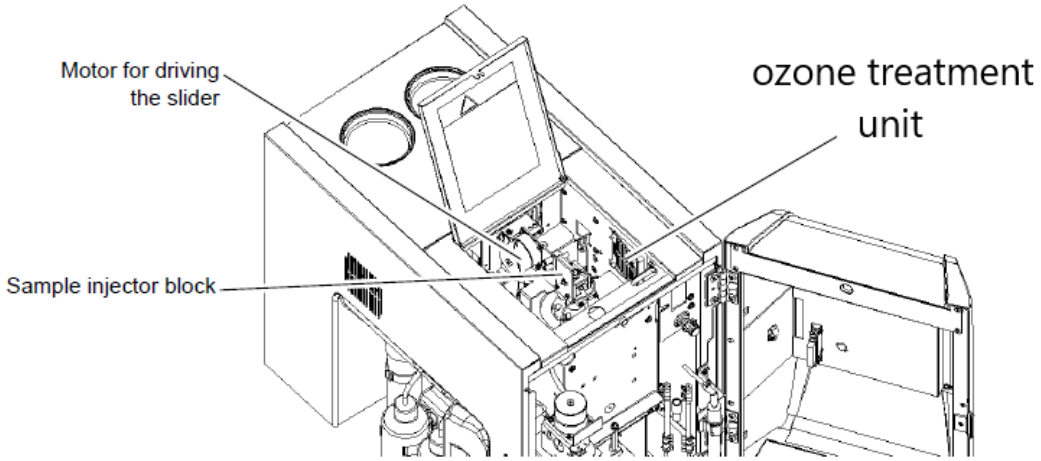
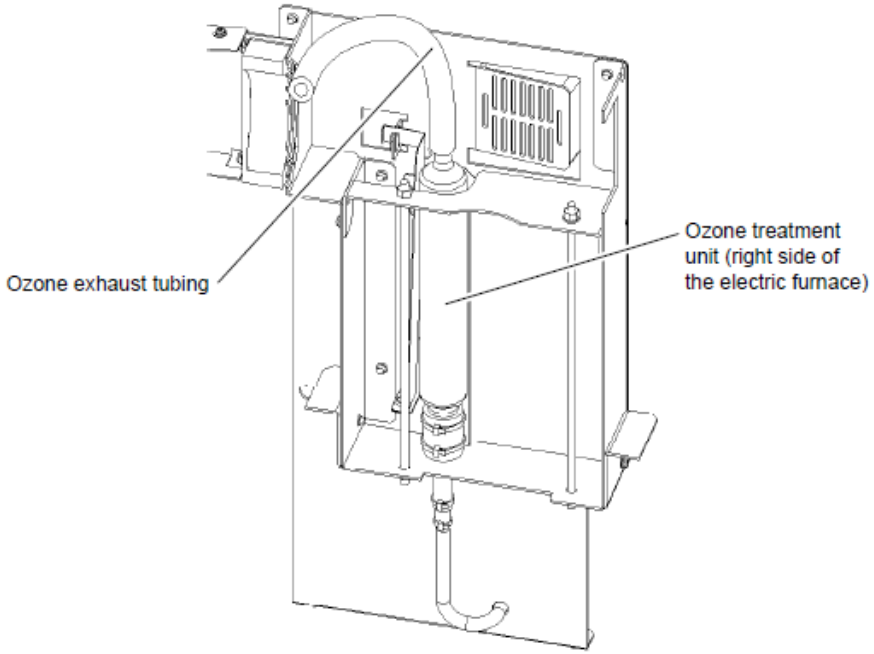
Section 2 / 2

Replacing the Ozone Killing Catalyst

The ozone treatment unit breaks down ozone gas produced by the ozone generator. The catalyst inside the ozone treatment unit must periodically be replaced. Replace the catalyst once a year for an instrument that runs 8 hours a day and 5 days a week.



Disconnect the tubing (Viton tubing) from each end of the ozone treatment unit and pull out the ozone treatment unit in the upward direction.

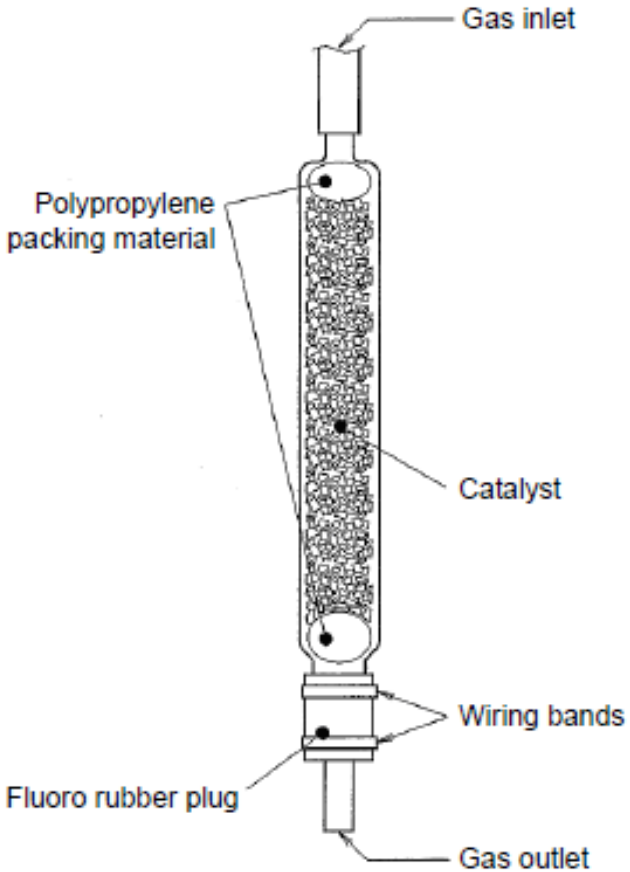


Section 1 / 3

Replacing the Ozone Killing Catalyst

Remove the larger diameter fluoro rubber plug and take out the used catalyst and polypropylene packing material.

Insert a small amount of new polypropylene packing material at the bottom of the vessel and then fill with catalyst to about 2 cm below where the vessel hollows. Insert another small amount of polypropylene packing material on top of the catalyst and then reattach the fluoro rubber plug. (The amount of catalyst is about 150 g.)



Ozone treatment unit

Filling with catalyst

Replacing the Ozone Killing Catalyst

Return the ozone treatment unit to its original position in the instrument and reconnect the tubing to each end of the unit.

CAUTION

Used catalyst should be disposed of according to local industrial waste disposal regulations. It must be clearly disclosed using an industrial waste manifest that the catalyst contains manganese dioxide.

Removing the Covers

Front View

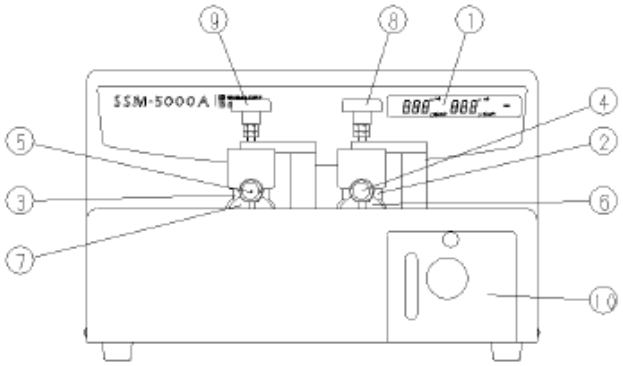


Figure 2.1 • SSM-5000A Front View

No.	Item	Description
1	Display window	Displays the TC furnace temperature, IC furnace temperature, and power supply ON/OFF status.
2	Sample port cover (IC side)	IC sample port cover. This green cover can be moved up, down, left, and right.
3	Sample port cover (TC side)	TC sample port cover. This blue cover can be moved up, down, left, and right.
4	Sample boat push rod knob (IC side)	Use this green knob to move the sample boat in and out of the IC furnace.
5	Sample boat push rod knob (TC side)	Use this blue knob to move the sample boat in and out of the TC furnace.
6	Sample port (IC side)	The sample boat is inserted and removed through the opening at the top of the IC port.
7	Sample port (TC side)	The sample boat is inserted and removed through the opening at the top of the TC port.
8	Sample port cover knob (IC side)	Turn this green knob counterclockwise to raise the IC sample port cover. Move the knob to the right to open the sample port.
9	Sample port cover knob (TC side)	Turn this blue knob counterclockwise to raise the TC sample port cover. Move the knob to the right to open the sample port.
10	Flow controller	Controls the carrier gas flow.

Top View

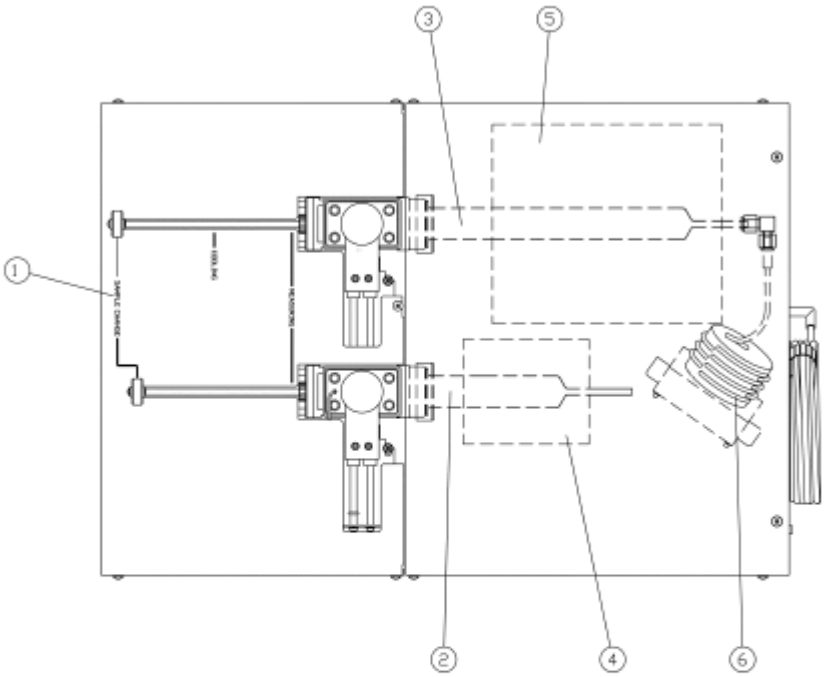


Figure 2.2 • Top View

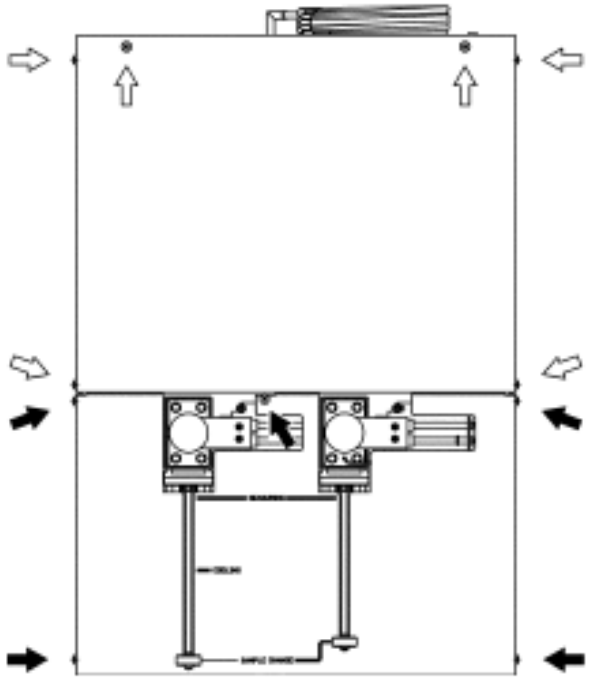
No.	Item	Description
1	Sample boat stop position indicators	Placement indicators used to move the sample boat into and out of the furnace.
2	IC reaction tube	Made of Pyrex glass.
3	TC combustion tube	Made of quartz glass and filled with an oxidizing catalyst.
4	IC furnace	Normal operating temperature is 200°C.
5	TC furnace	Normal operating temperature is 900°C.
6	Condensation coil	Cools the gases generated by sample combustion in the TC combustion tube.

Removing the Covers

Remove the screws and carefully remove the rear cover first.

CAUTION

Be careful as there is a wire harness connected to a fan on the left hand side of the cover that needs to be unclipped before the cover can be completely removed.



Next Remove the front cover:

- Move the sample pushrod knob to the 'Measuring' position
- With the sample port cover closed, slightly lift the rear of the front cover, pull it forward about 8cm and lift off.

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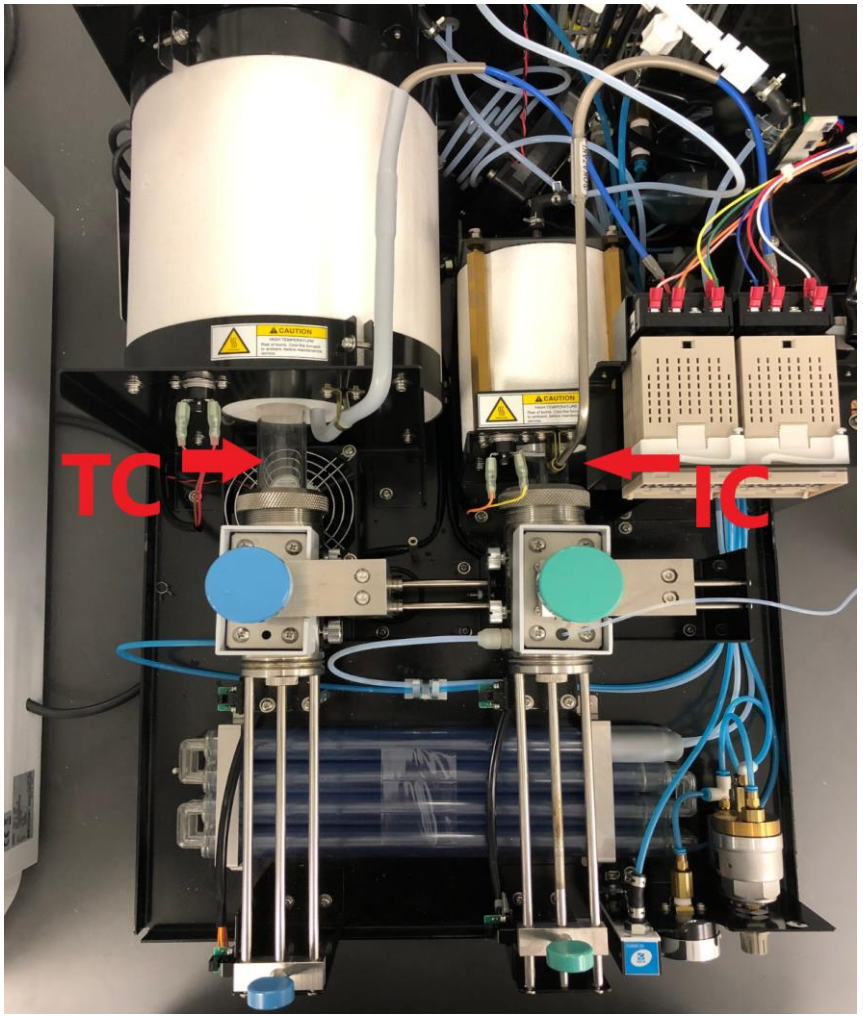
Section 2 / 2

Replacing the Catalyst

The following procedure describes removal of the TC combustion tube. This same procedure can also be used to remove the IC combustion tube. The only difference is the IC combustion tube is an empty quartz tube while the TC combustion tube is filled with catalyst.

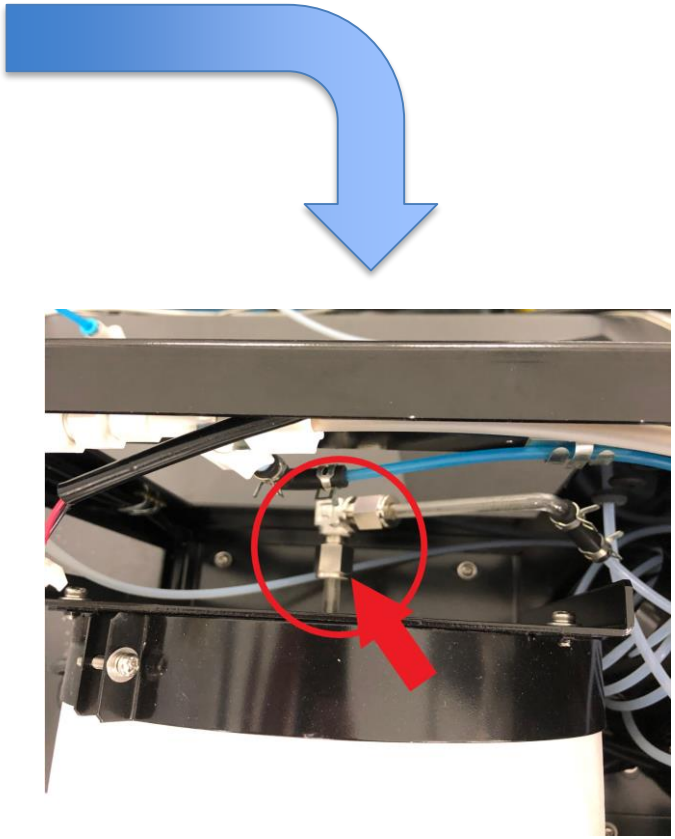


Replacing the Catalyst

1. Locate where the combustion tube comes out the back of the TC Furnace

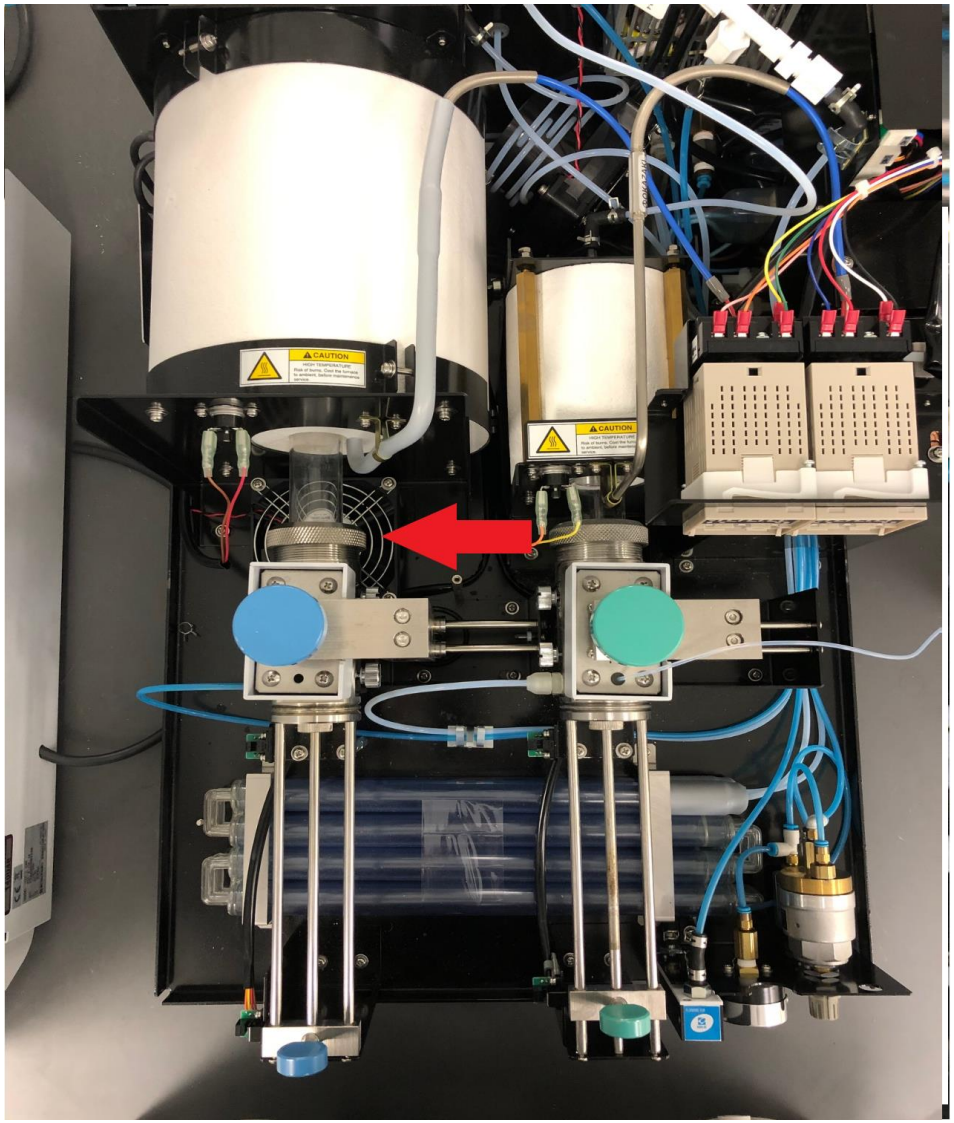
2. Find the stainless steel elbow fitting that connects the combustion tube to the glass shaped ‘L’ tube

3. Loosen the fitting on the side of the combustion tube and remove the fitting.



Replacing the Catalyst

Unscrew the metal ring to disconnect the sample port from the quartz combustion tube

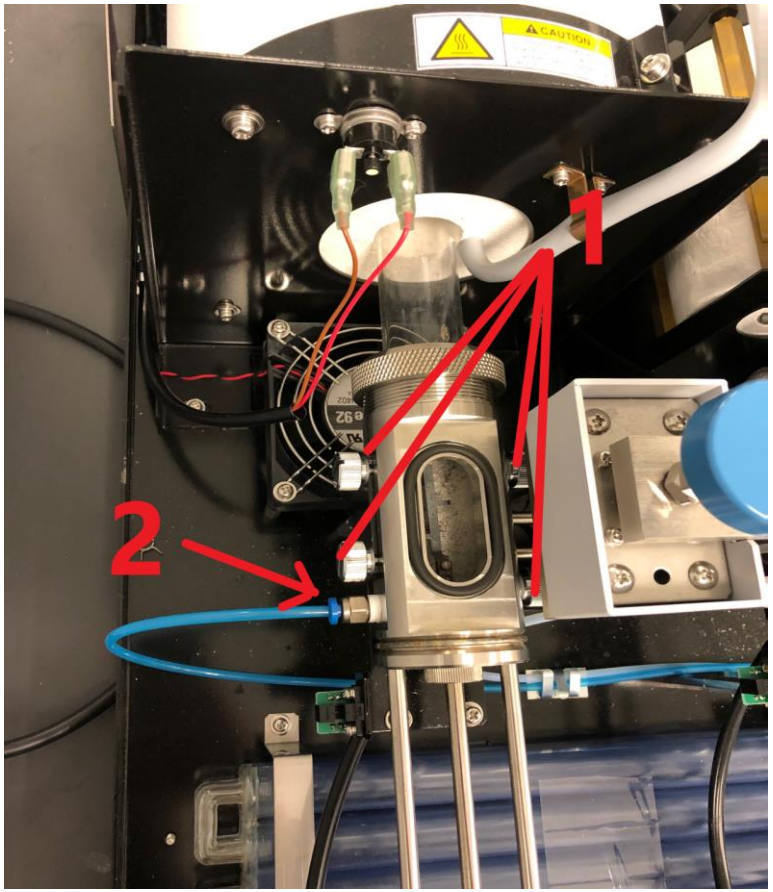


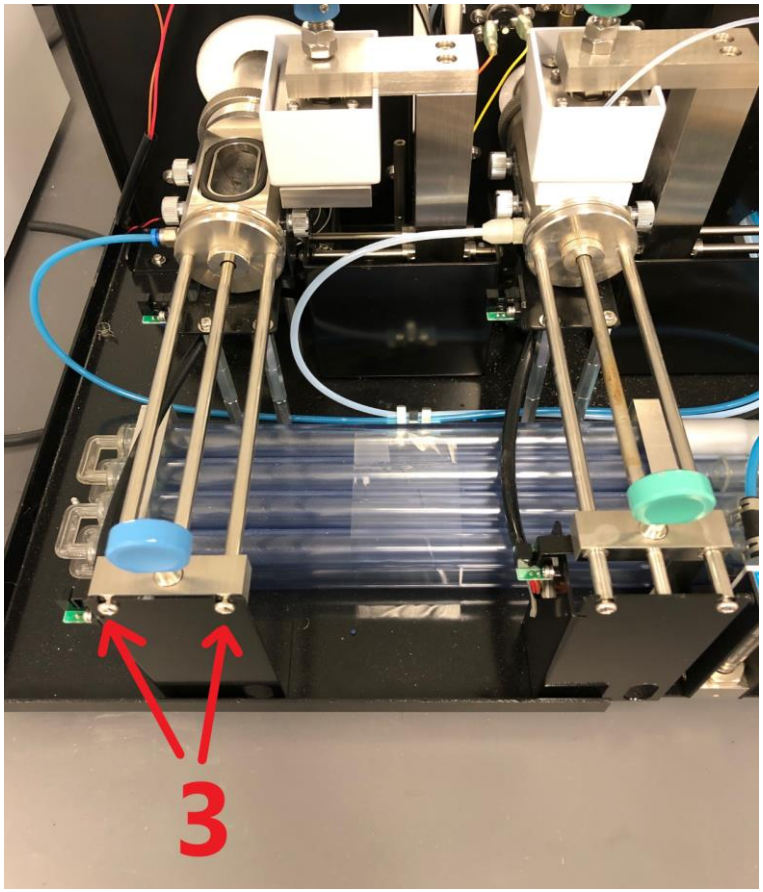
Replacing the Catalyst

1. Loosen the four thumbscrews on the sides of the sampling port

2. Remove the blue air line from the left hand side of the sampling port. To do this, push the blue collar of the fitting toward the sampling port while at the same time pulling the blue tubing away from the sampling port.

3. Loosen the two screws toward the front that holds the push rod to the frame.





Replacing the Catalyst

1. Remove the sampling port

2. Remove the combustion tube by carefully pulling the combustion tube forward until it is completely out of the furnace.

CAUTION

Make sure to pull the combustion straight forward until it is completely out of the furnace otherwise you risk breaking the end of the combustion tube.

Replacing the Catalyst

Prepare the catalyst mixture.

- 2 containers of cobalt oxide catalyst (25g each).
- 1 container of platinum catalyst (20g).

In a clean container, mix the catalysts to as uniform a mixture as possible.

Introduce approximately 45g of the catalyst mixture into the TC combustion tube.

To prevent clustering of either type of catalyst, rotate the TC combustion tube while filling it with the catalyst mixture.

Note: Do not allow pulverized catalyst dust to be transferred into the TC combustion tube. This dust will increase resistance to the airflow through the TC combustion tube, making measurement impossible.

Fold the catalyst support screens and insert them into the TC combustion tube.

Push the catalyst support screens into place with the catalyst filling rod supplied as a TOC-L accessory.

Use the catalyst packing rod to pack the catalyst in a manner that creates some resistance between the inside of the combustion tube and the outside of the mesh. This prevents the mixed catalyst from crumbling and falling through the mesh and possibly flowing upstream toward the detector.

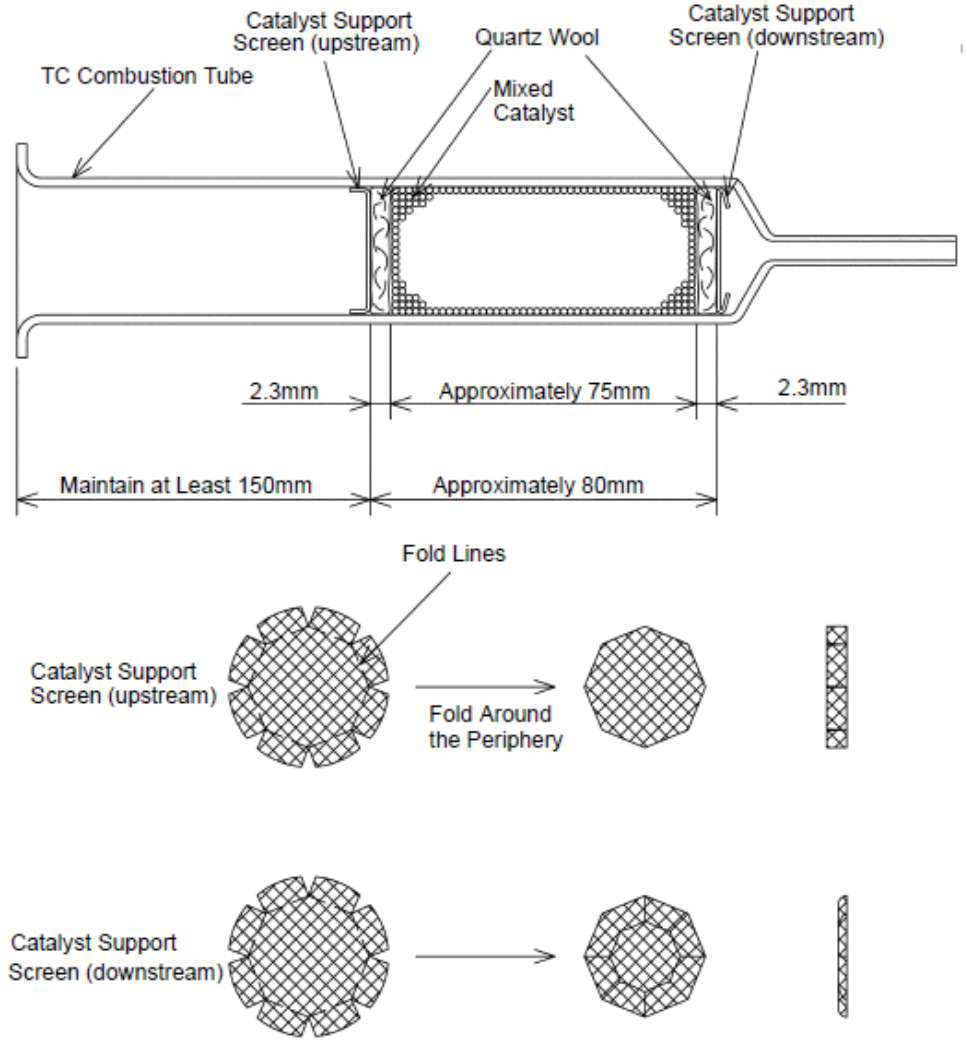
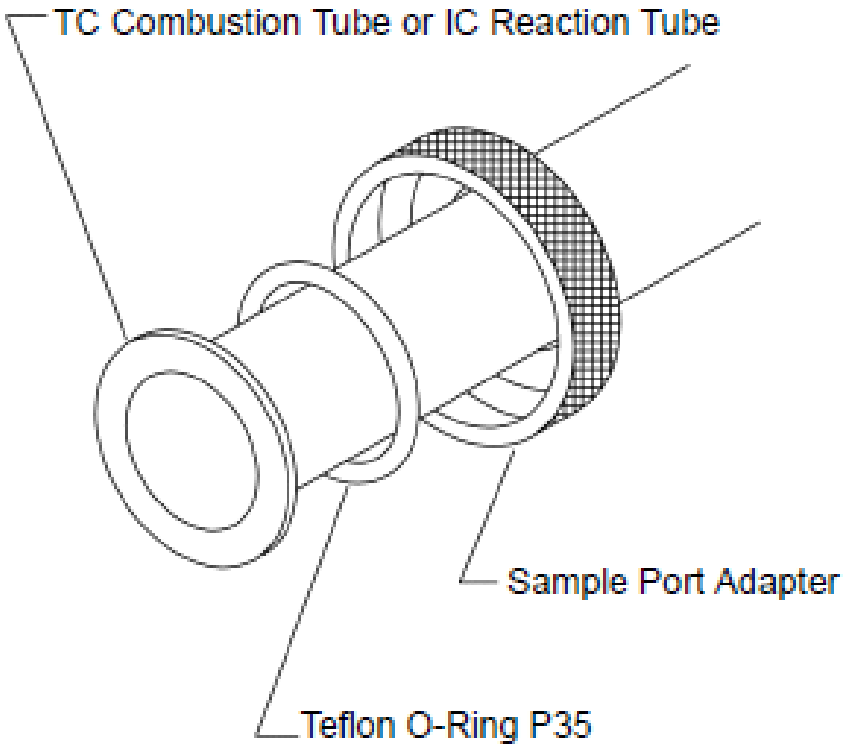


Figure 5.6 • Filling the TC Combustion Tube with Catalyst

Replacing the Catalyst

Place the white PTFE o-ring around the newly packed combustion tube. Then place the metal sample port adapter around the combustion tube.

Follow the instruction in reverse order to install the combustion tube into the furnace and attach the sample port to the combustion tube.



CAUTION

When tightening the metal sample port adapter to the new combustion tube only hand tighten. Over tightening could cause the combustion tube to break upon heating the tube as the quartz expands.

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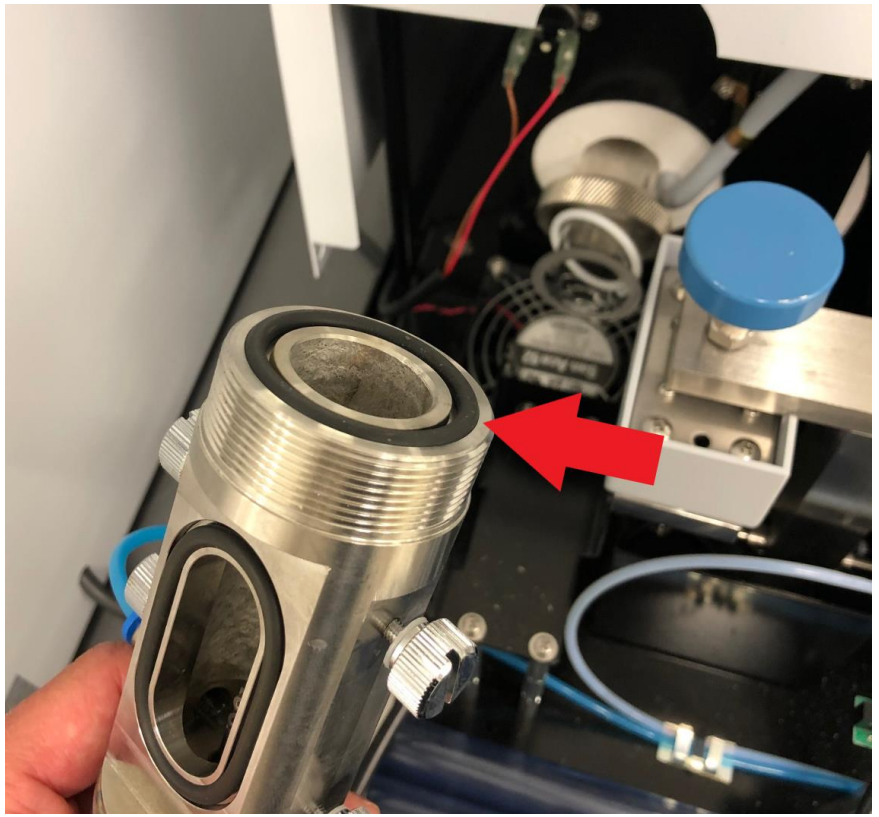
Section 7 / 7

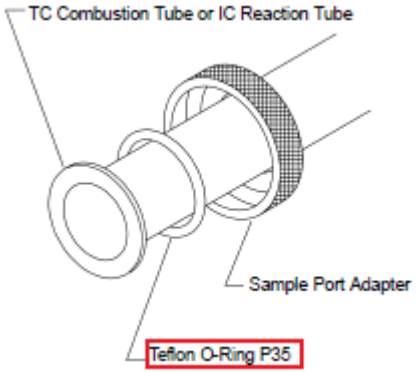
Replacing the O-rings

Remove the combustion tube and sample port following the instructions for replacing the catalyst.

Replace the white PTFE o-ring that is around the combustion tube.

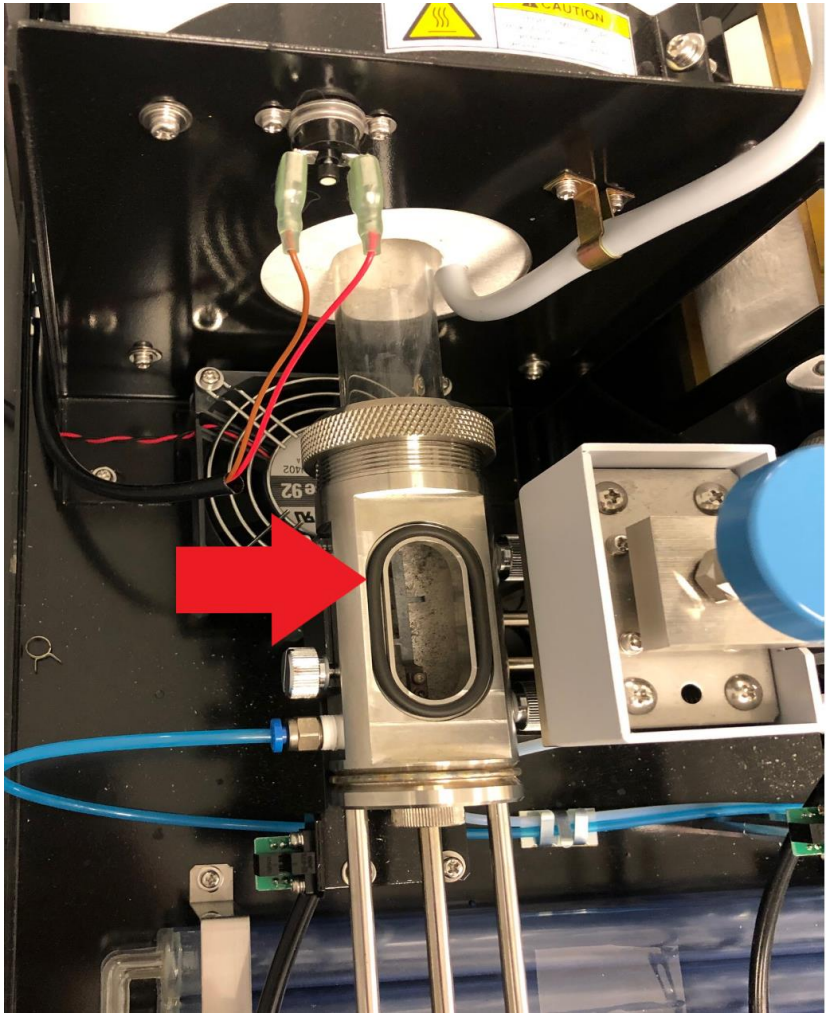
Replace the black viton o-ring the fits on the end of the sample port and seals to the combustion tube.





Replacing the O-rings

Loosen the blue knob for TC or green knob for IC and slide the sample port cover to the right. Remove the old o-ring and replace with new.



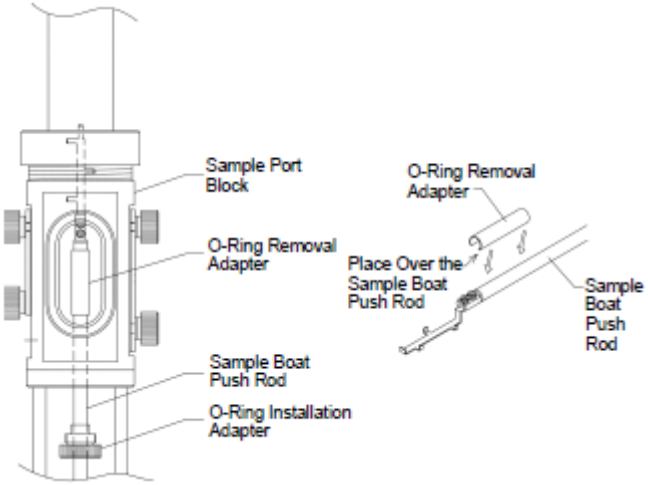
Replacing the O-rings

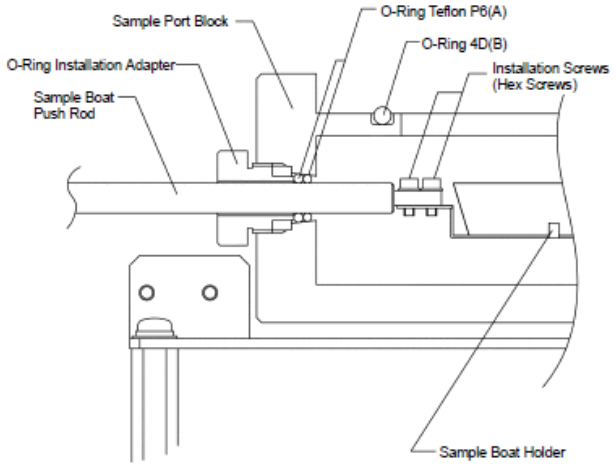
1. Remove the front cover.
2. Holding the knurled part of the O-ring installation adapter, with the sample boat push rod inserted, turn it counterclockwise to remove it.
3. Push the sample boat push rod knob about 4 to 5cm into the furnace from the SAMPLE CHANGE position.
4. Open the sample port cover, and fit the O-ring removing adapter over the sample boat push rod through the sample port opening.
5. Pull back the sample boat push rod knob to the SAMPLE CHANGE position. The two O-rings can now be removed from the sample port block.
6. Loosen the Phillips screw at the rear of the sample boat push rod knob, and pull the sample boat push rod from the knob.
7. Remove the O-ring installation adapter and two O-rings.
8. Verify that there is no foreign matter stuck to the surface of the sample boat push rod.
9. Reverse the procedure for removing the O-rings to put two new O-rings and the O-ring installation adapter on the sample boat push rod. Tighten the Phillips screw on the back of the sample boat push rod knob.
10. Holding the knurled part of the O-ring installation adapter, turn it clockwise to screw it into the sample port block until the sample boat push rod resists movement.
11. Verify that no gas leaks from the Teflon O-rings.

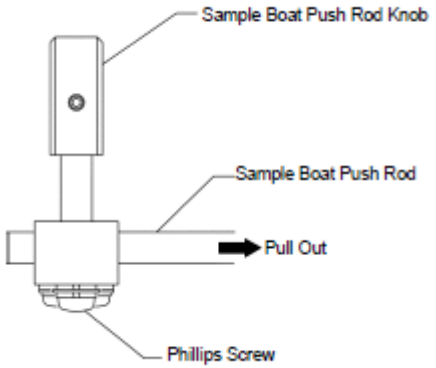
Note: Any foreign matter that is stuck to the surface of the sample boat push rod prevents the rod from sliding smoothly and may cause gas leaks due to accelerated wear or scratching of the Teflon O-rings.

If foreign matter is found on the surface of the sample boat push rod, clean it off or use a soft cloth to polish the surface of the rod.

If foreign matter is stuck to the part of the rod that enters the furnace and cannot be easily wiped off, wash it in approximately 1M hydrochloric acid or polish with polishing powder. Using polishing powder may reduce the diameter of the sample boat push rod, causing more frequent gas leaks.







TIP » *The surface of the TC sample boat push rod may become discolored due to the formation of an oxide film on the portion that is inserted into the furnace. This film does not interfere with analysis unless the surface of the sample boat push rod becomes roughened.*

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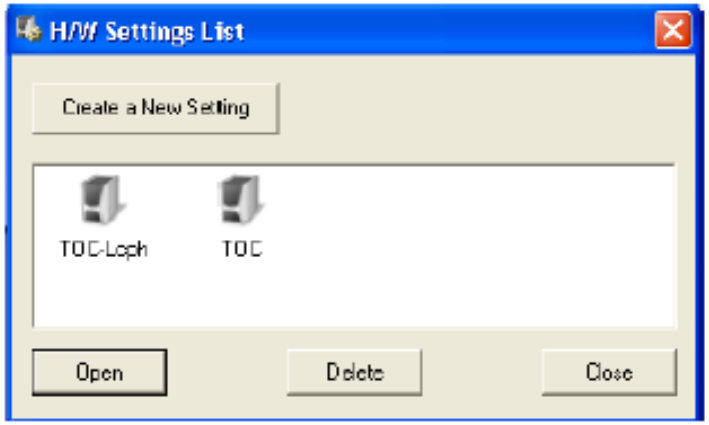
Section 3 / 3

Creating a Hardware Configuration

1. From the Desktop double click the TOC-Control L icon. If the Measurement tab is not open, select it and click the "HW Settings" button.

2. Click on "Create A New Setting".

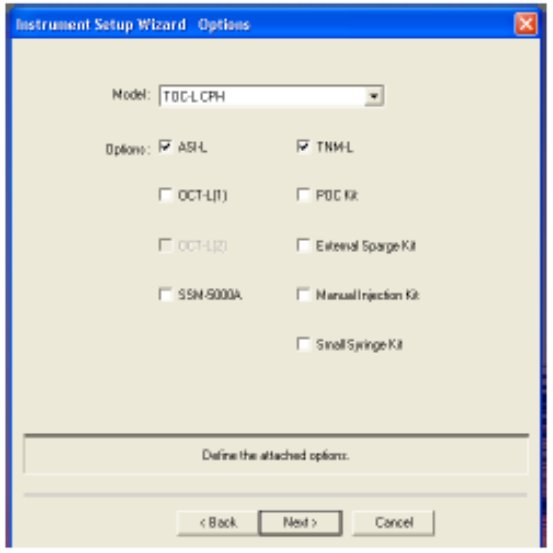


Section 1 / 4

Creating a Hardware Configuration

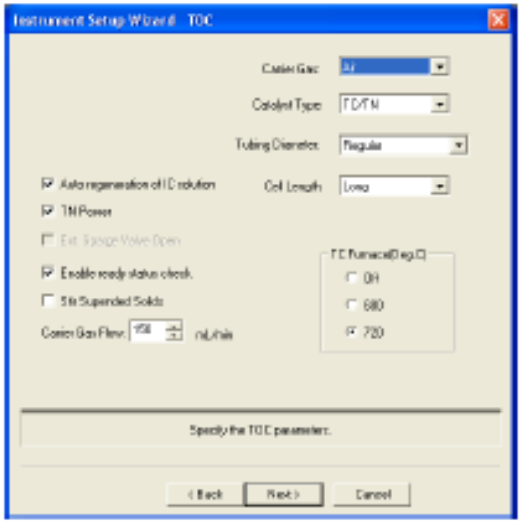
3. Enter a suitable system name and the instrument serial number (entering 10101000 for the serial number works if you have problems with the software not accepting other inputs). Click Next to continue.

4. Select the appropriate Instrument model and any options attached to the system. Click Next to continue.

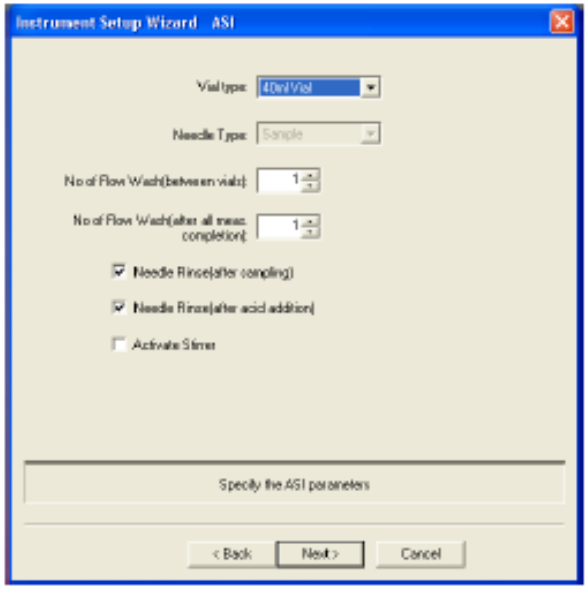


Creating a Hardware Configuration

5. Select the appropriate choices for carrier gas and catalyst type. Check appropriate boxes for TN Power, etc. and set flow rate to 150 ml/min. If a TNM-L is part of the system choose the 720 radio button. Click Next to continue.

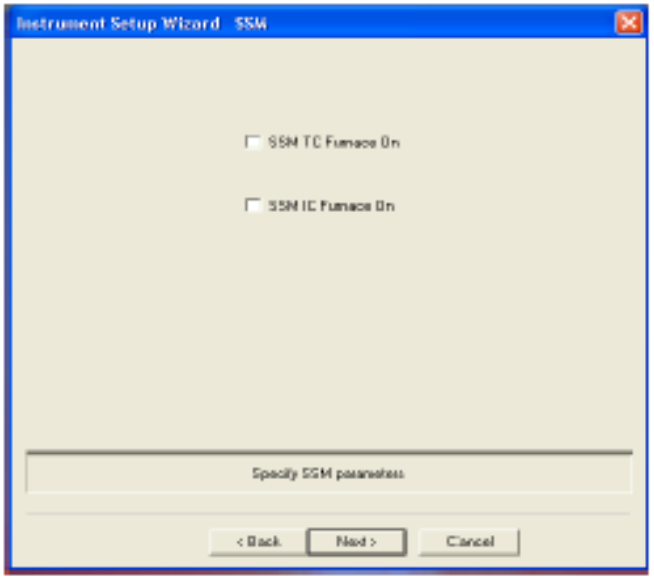


6. If an ASI autosampler is attached to the system select the appropriate vial size based on the included vial rack. Click Next to continue.

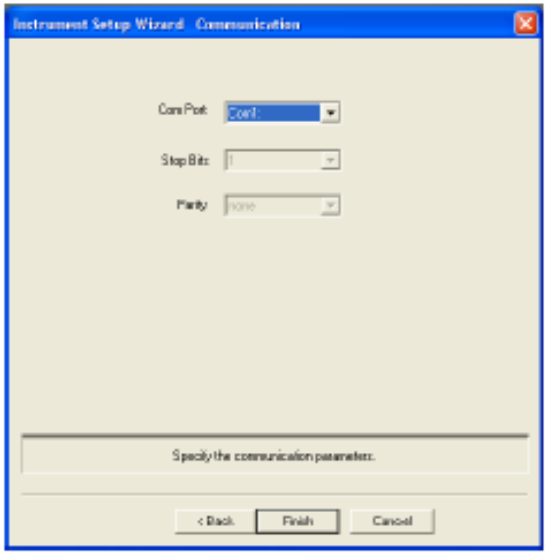


Creating a Hardware Configuration

7. If a SSM-5000 is part of the system check the TC/IC furnace boxes. This automatically heats the furnaces upon software connection to the instrument. Click Next to continue.



8. Confirm and select the appropriate Com port for instrument communication and click Finish.



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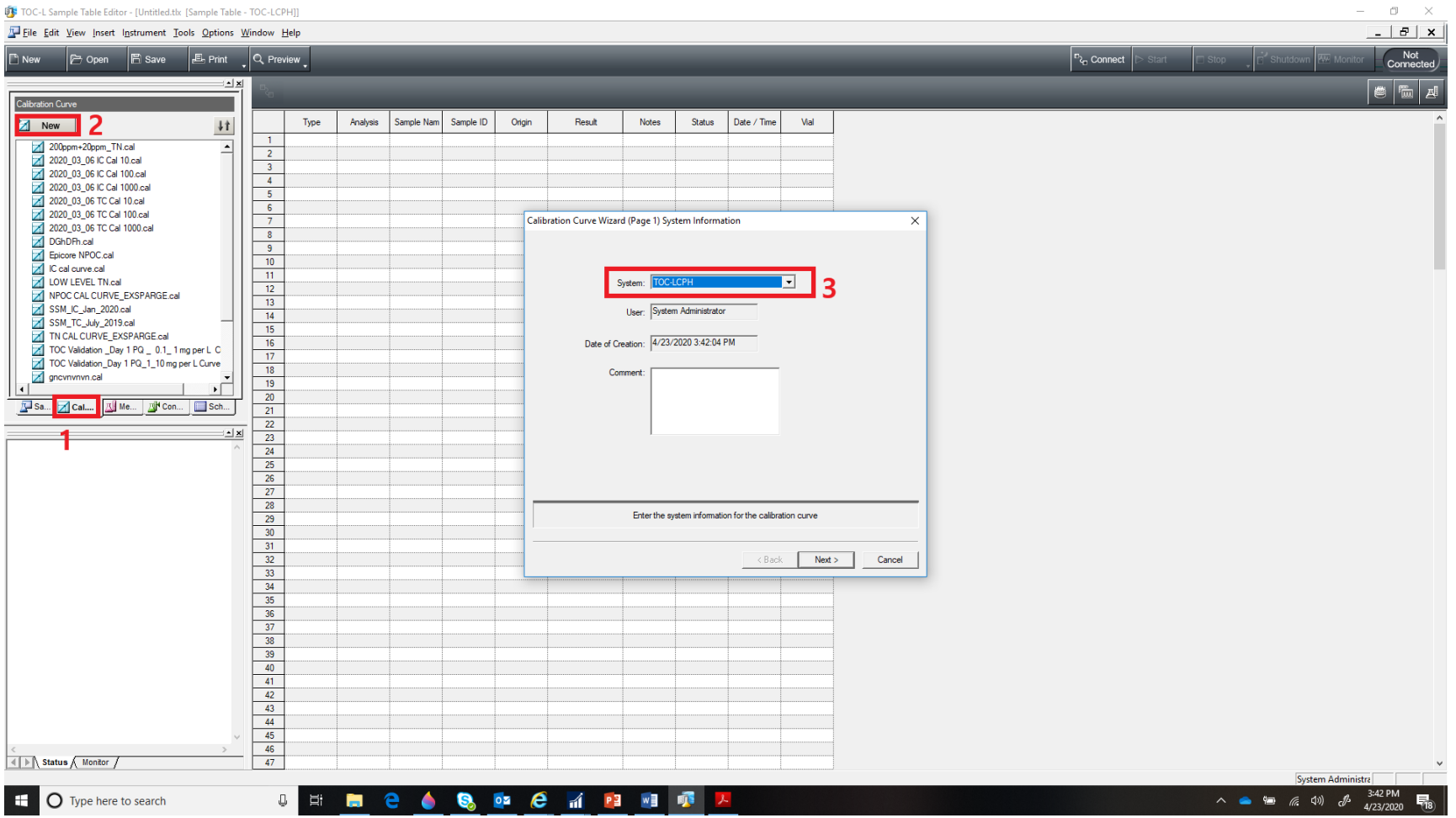
Section 4 / 4

Creating a Calibration Curve

1. Push the calibration curve list button
2. Push the ‘New’ button
3. Select the correct hardware configuration

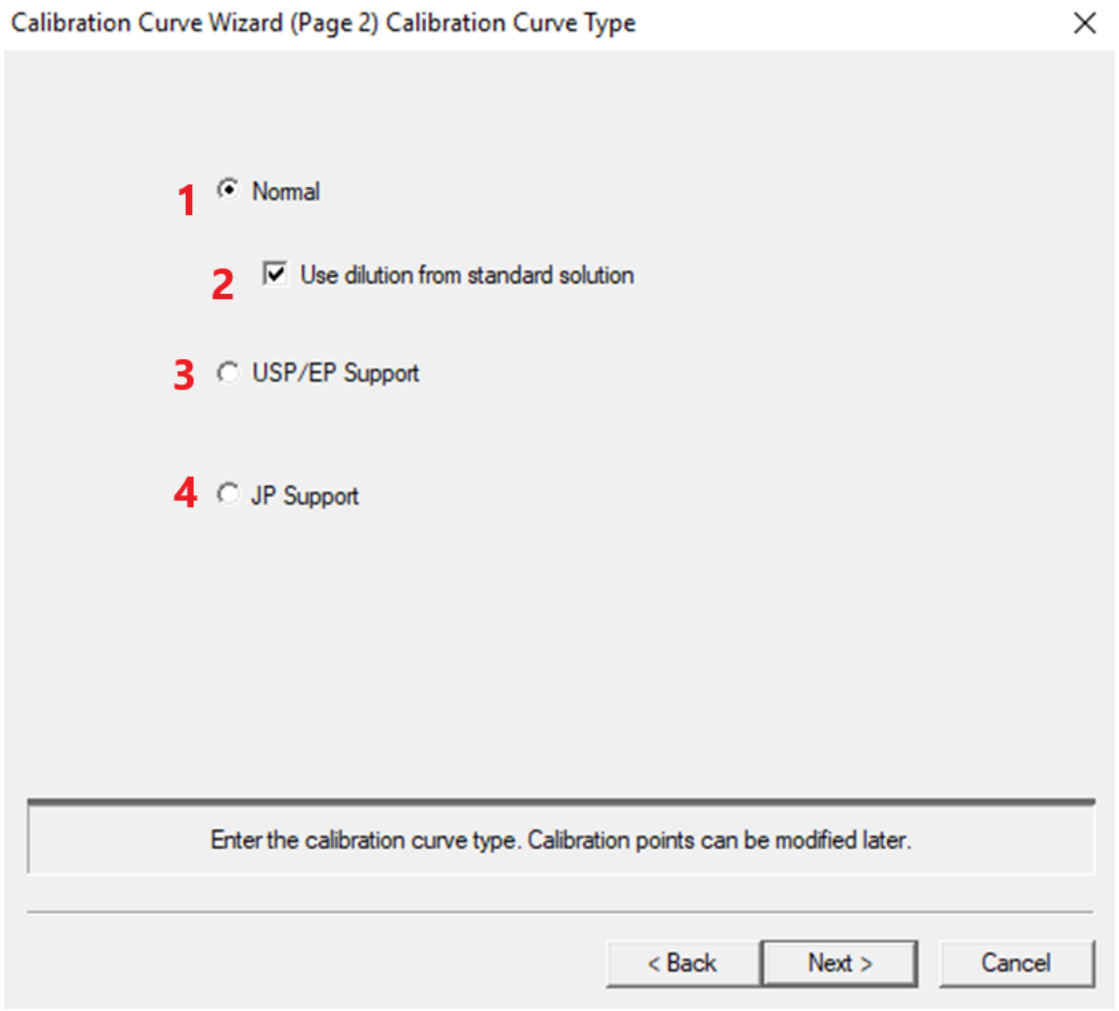


Section 1 / 7

Creating a Calibration Curve

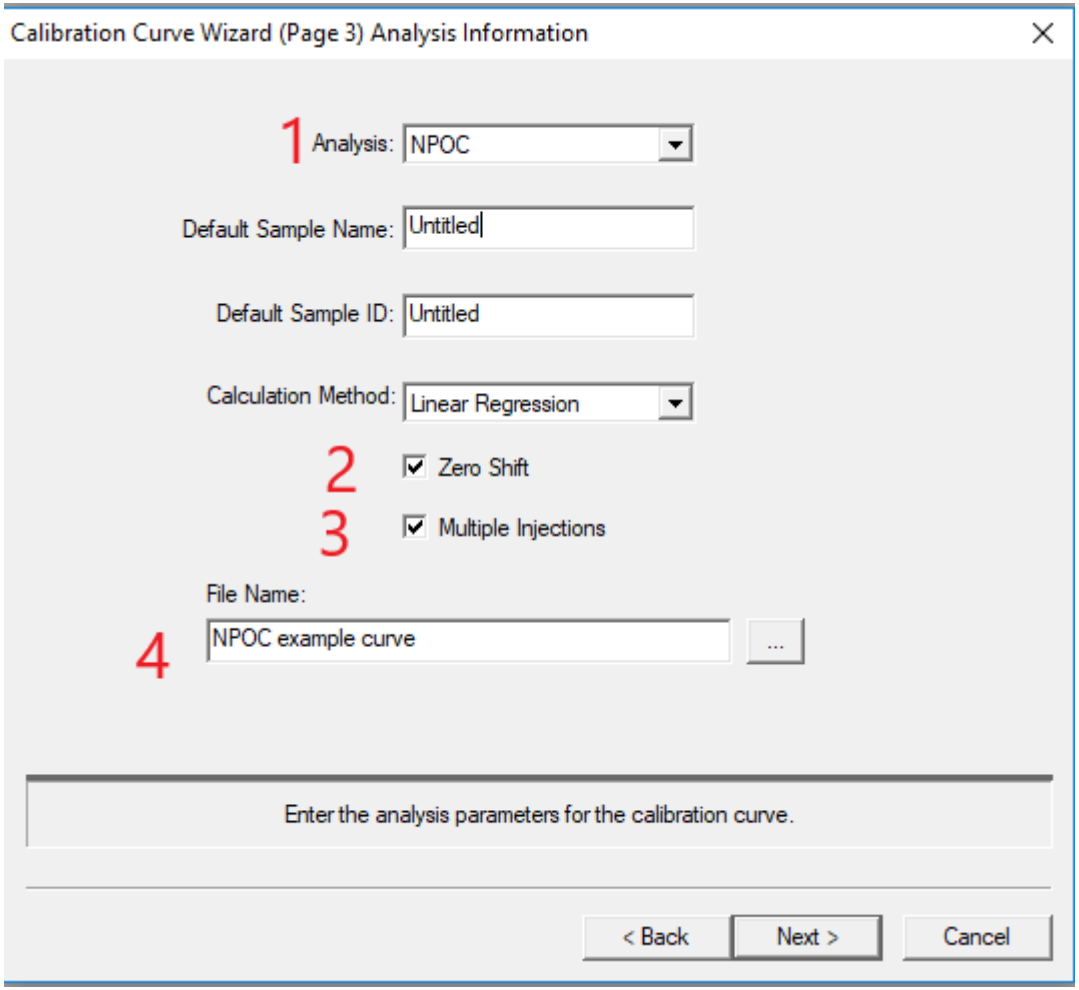
Select the curve type

1. Select ‘Normal’ to create a curve with individually prepped standard solutions.
2. Select ‘Use dilution from standard solution’ to create a curve where the instrument will auto-dilute a high standard to create a multi-point calibration curve.
3. Select ‘USP/EP Support’ to create a curve using the USP criteria.
4. Select ‘JP Support’ to create a curve using the JP criteria.



Creating a Calibration Curve

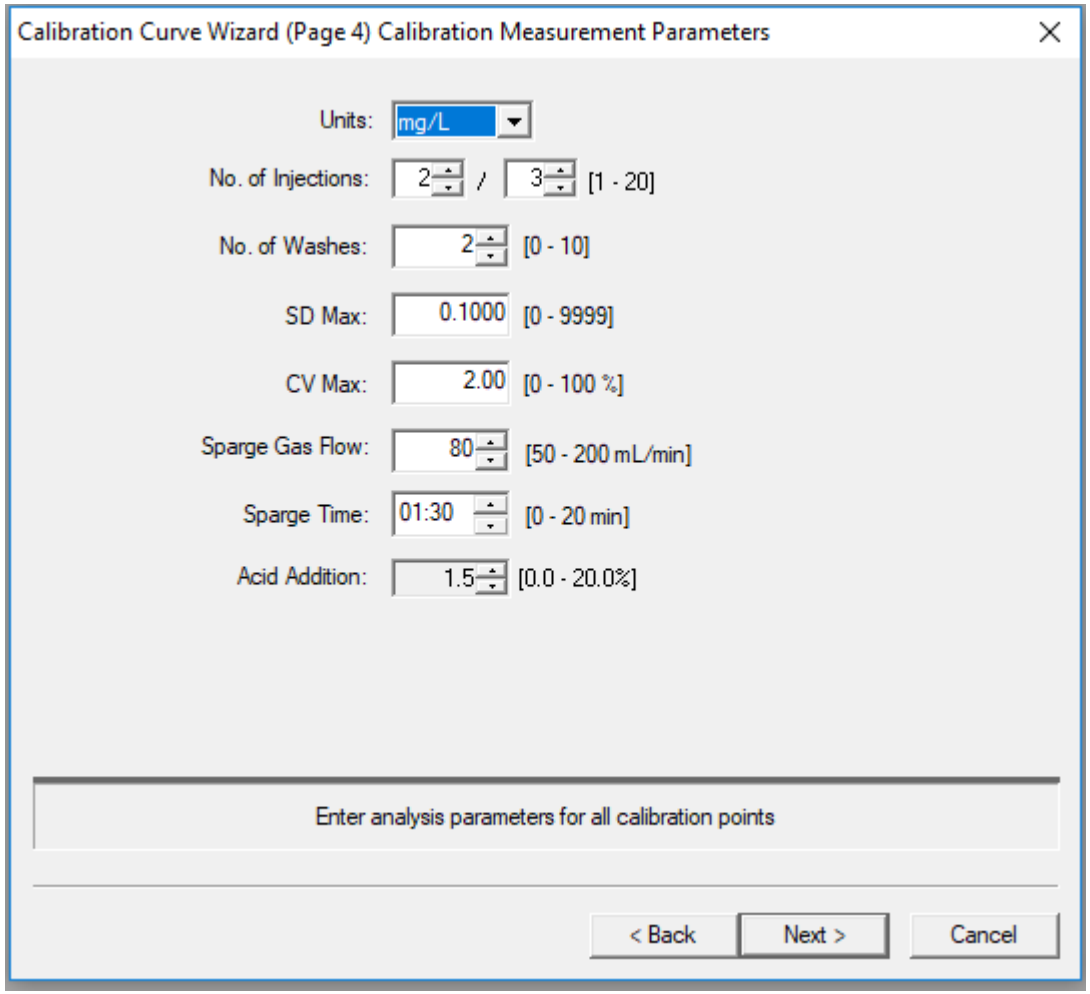
1. Select the type of analysis from the drop down tab
2. Select ‘Zero Shift’ to shift the curve down through the origin
3. Select ‘Multiple Injections’ to perform multiple sample injection from one syringe full of sample.
4. Give the curve a unique name



Creating a Calibration Curve

Select the parameters for the calibration curve.

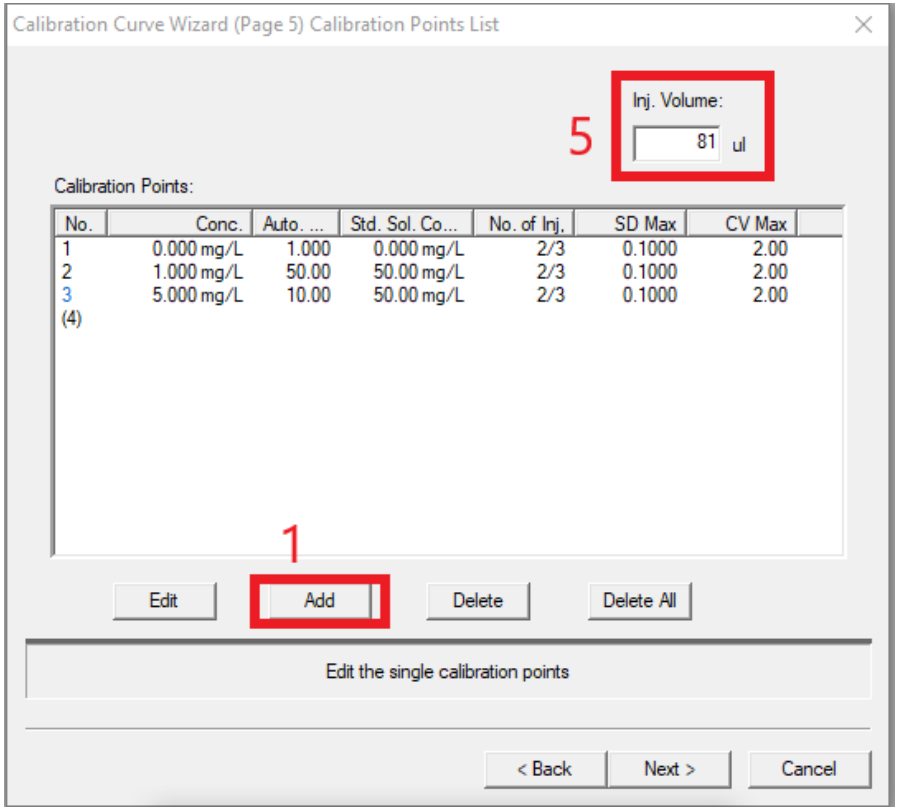
Please note that in this example the measurement type is NPOC so there is an option for selecting a sparge time and acid addition. When performing TC analysis there will be no option for selecting a sparge time.

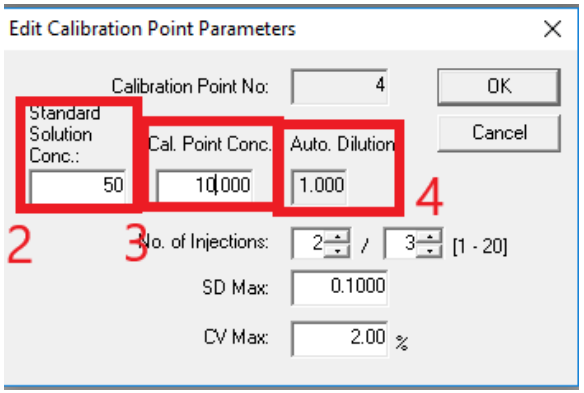


Creating a Calibration Curve

Creating a curve using the auto-dilution function

1. Select ‘Add’ to add a new point
2. Insert here the concentration of the high standard being used in the curve.
3. Insert here the actual concentration of the standard point.
4. The dilution rate will automatically populate here
5. The injection volume will be displayed here. The software will automatically select the most appropriate injection volume based upon the concentrations of points in the curve.





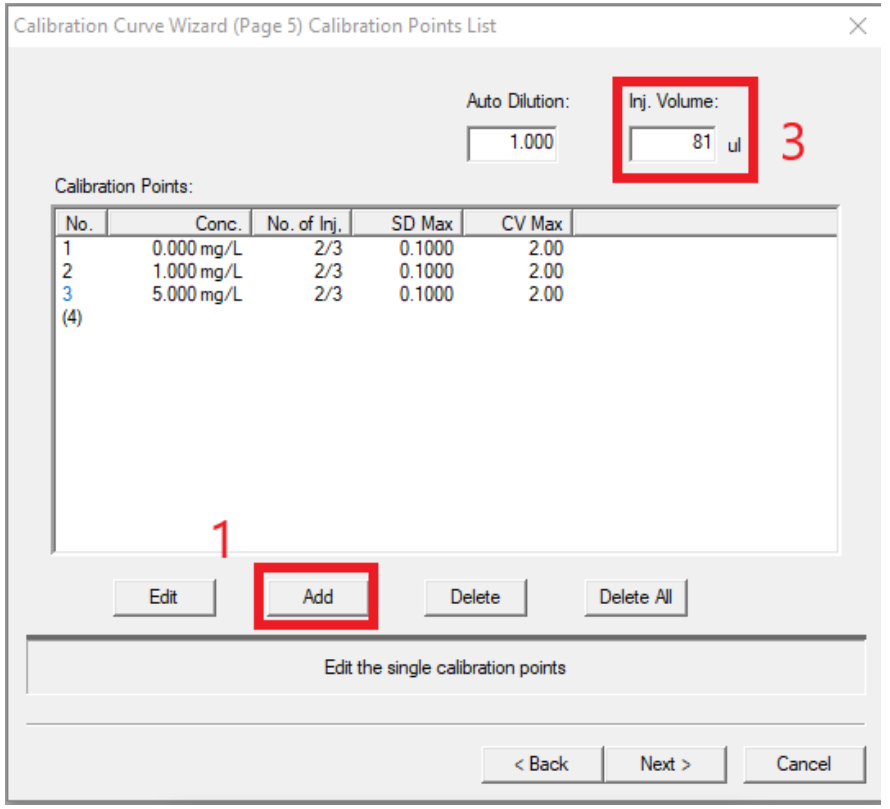
Creating a Calibration Curve

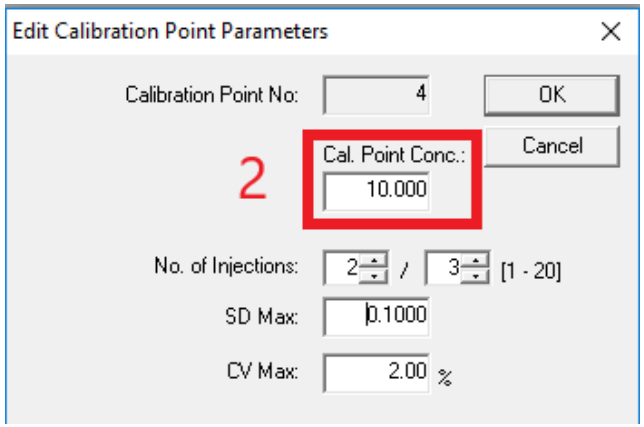
Creating a curve using individually prepped standards for each point

1. Select ‘Add’ to add a new point.

2. Insert here the concentration of the standard point.

3. The injection volume will be displayed here. The software will automatically select the most appropriate injection volume based upon the concentrations of points in the curve.



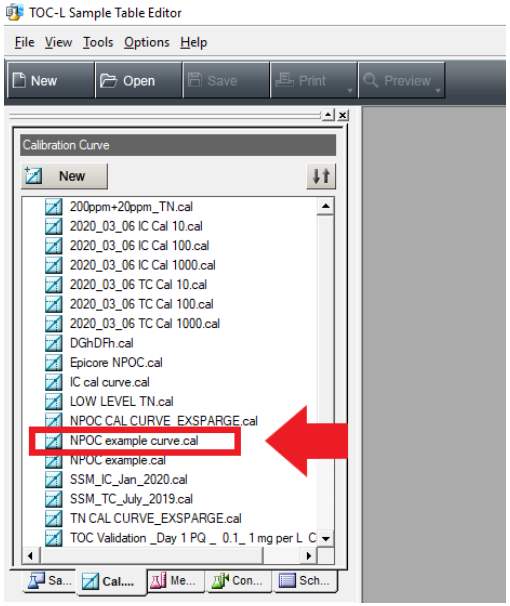


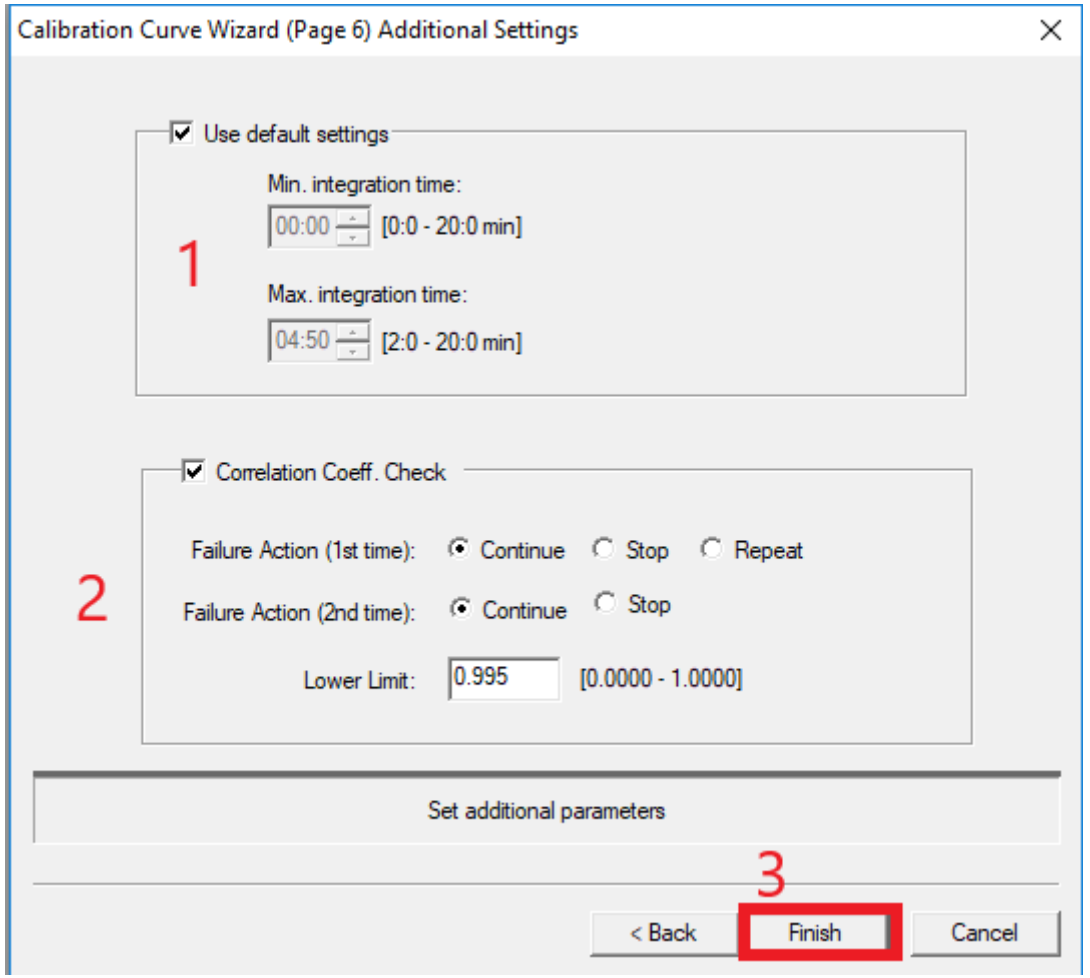
Creating a Calibration Curve

1. If integrations times need to be adjusted parameters can be changed here. Default settings a OK for 99% of analysis.

2. A pass/fail criteria can be set here to determine if a curve is OK based on a desired correlation coefficient.

3. Push ‘Finish’ to save the curve.





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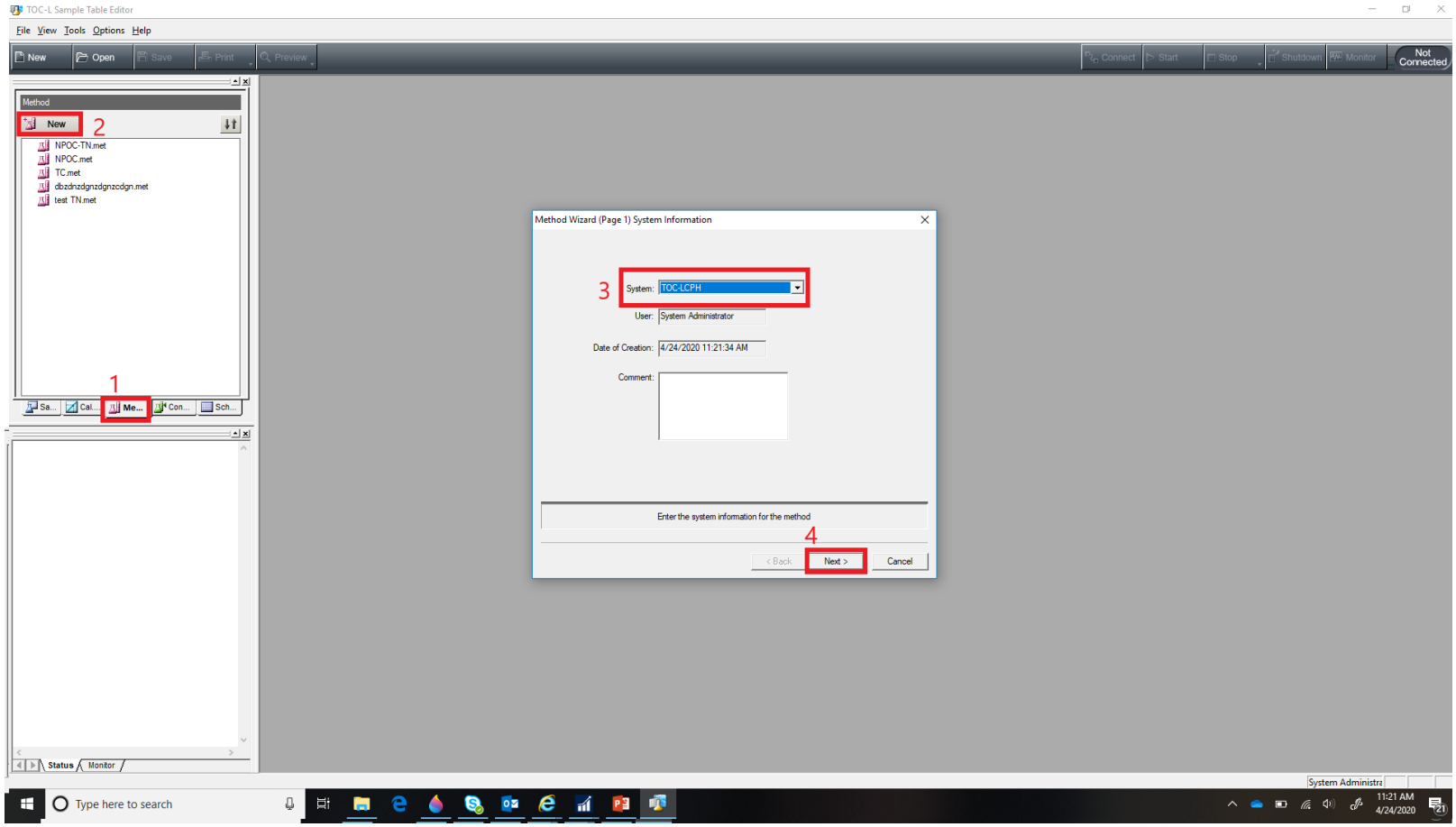
Section 7 / 7

Creating a Method

1. Push the ‘Method’ button to show the list of saved methods.
2. Push the ‘New’ button.
3. Select the correct hardware configuration.
4. Select ‘Next’



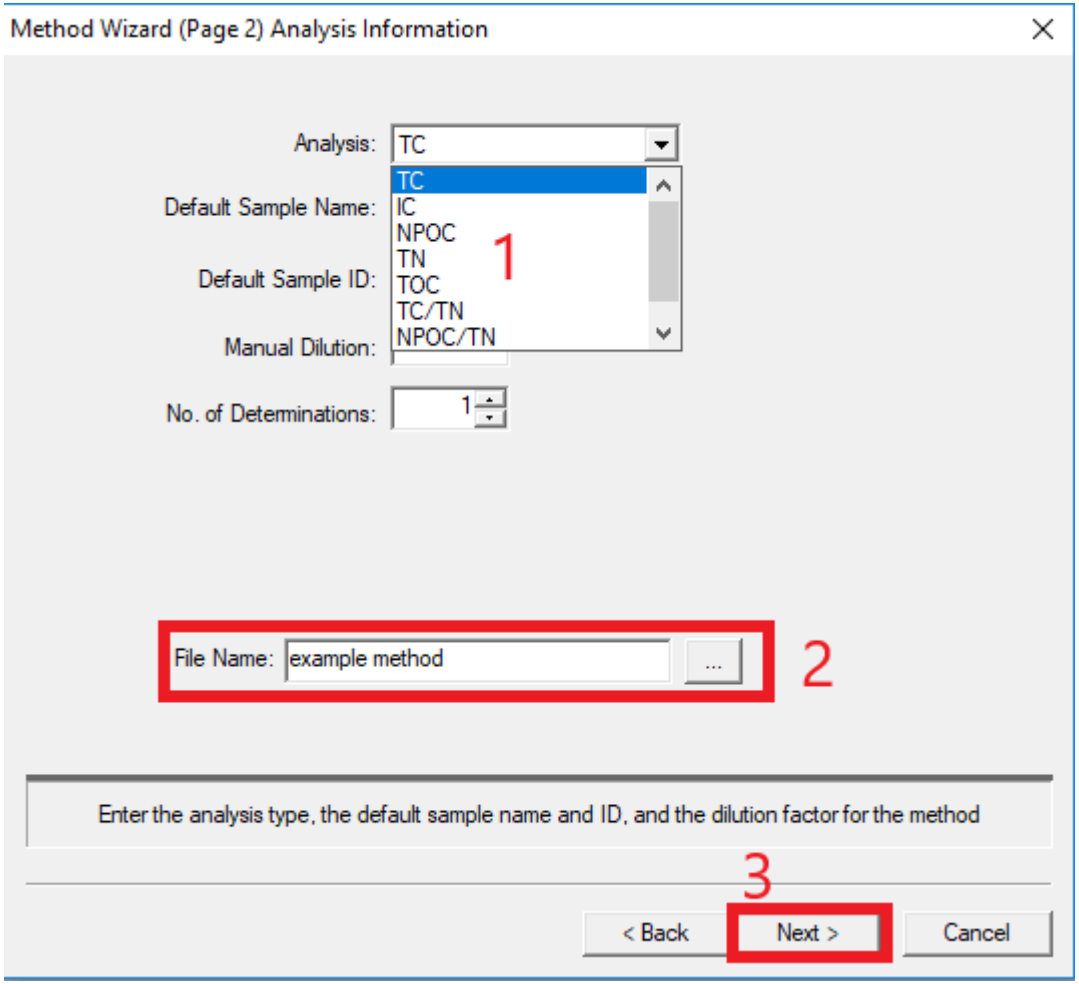
Section 1 / 9

Creating a Method

1. Select the type of measurement to be performed. Note that multiple measurements can be selected in a method. In this example we will select TOC which will prompt the instrument to perform both TC and IC measurement and then calculate TOC by the difference in TC and IC measurements. TOC = TC – IC.

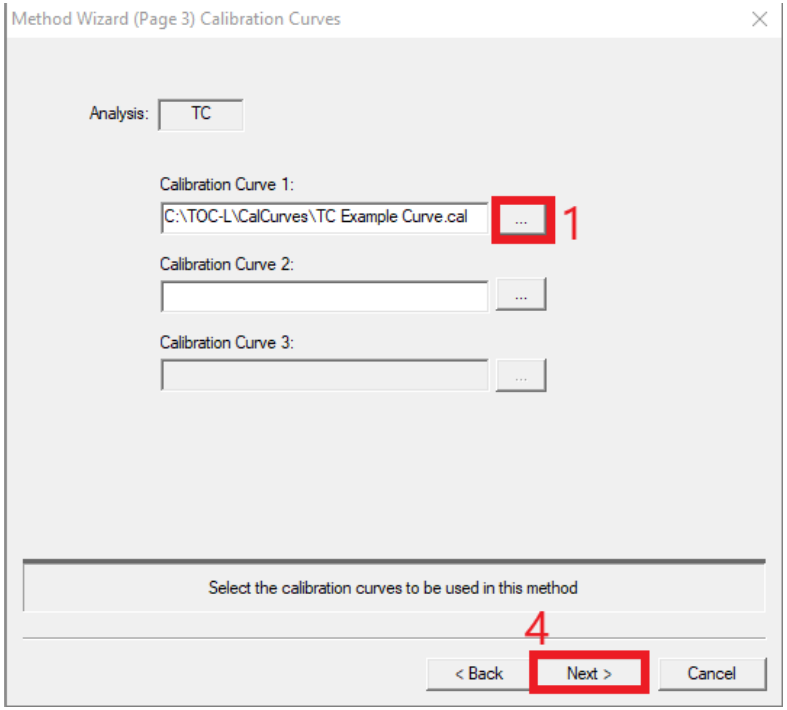
2. Give the method a unique name.

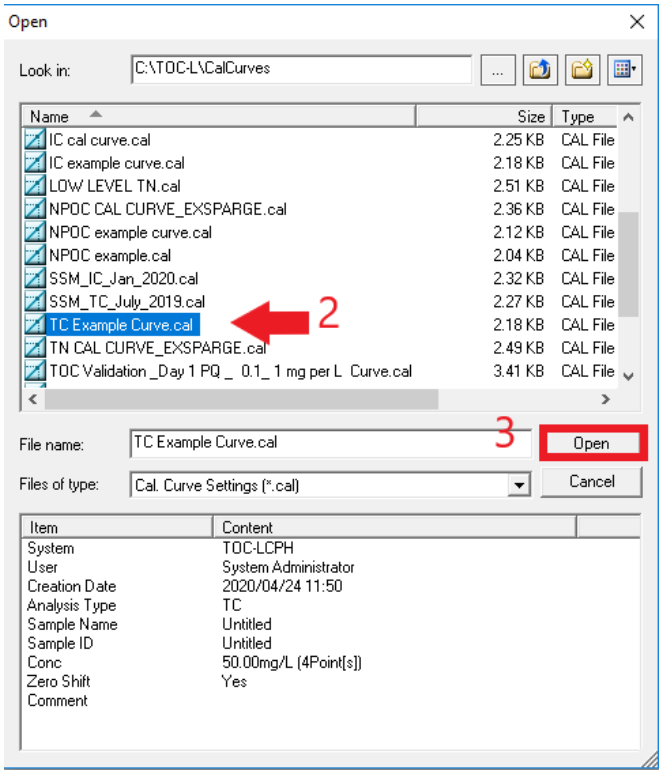
3. Select ‘Next’.



Creating a Method

1. Push the '…' button to select the curve for TC analysis.
2. Select the TC curve to be used for analysis.
3. Push 'Open' to select the curve and insert in 'Calibration Curve 1'
4. Push 'Next'

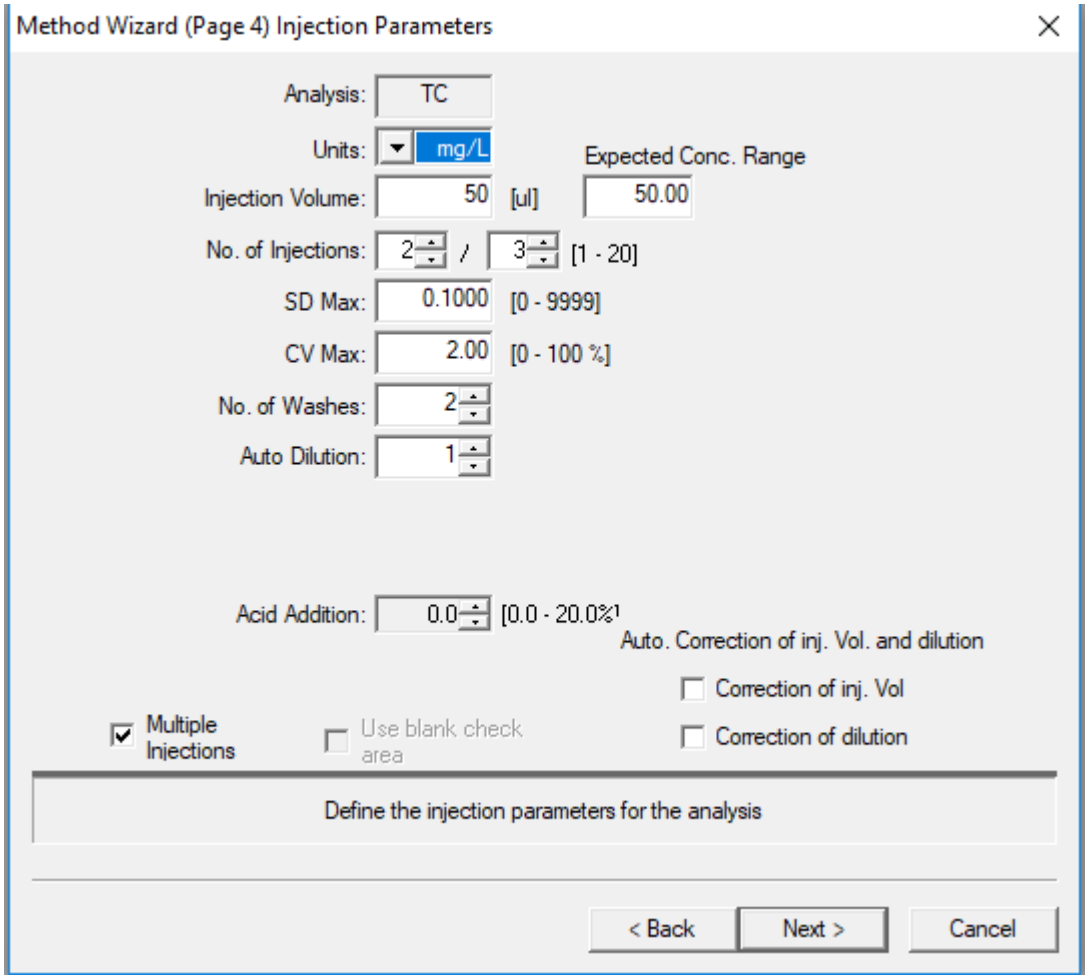




Creating a Method

Confirm the conditions for the TC analysis. Note that these conditions were pulled in from the calibration curve selected but can be changed if needed.

Check the box next to ‘Correction of inj. Vol’ or ‘Correction of dilution’ if you wish to have the instrument auto-dilute or lower injection volume for sample that are over the range of the calibration curve.

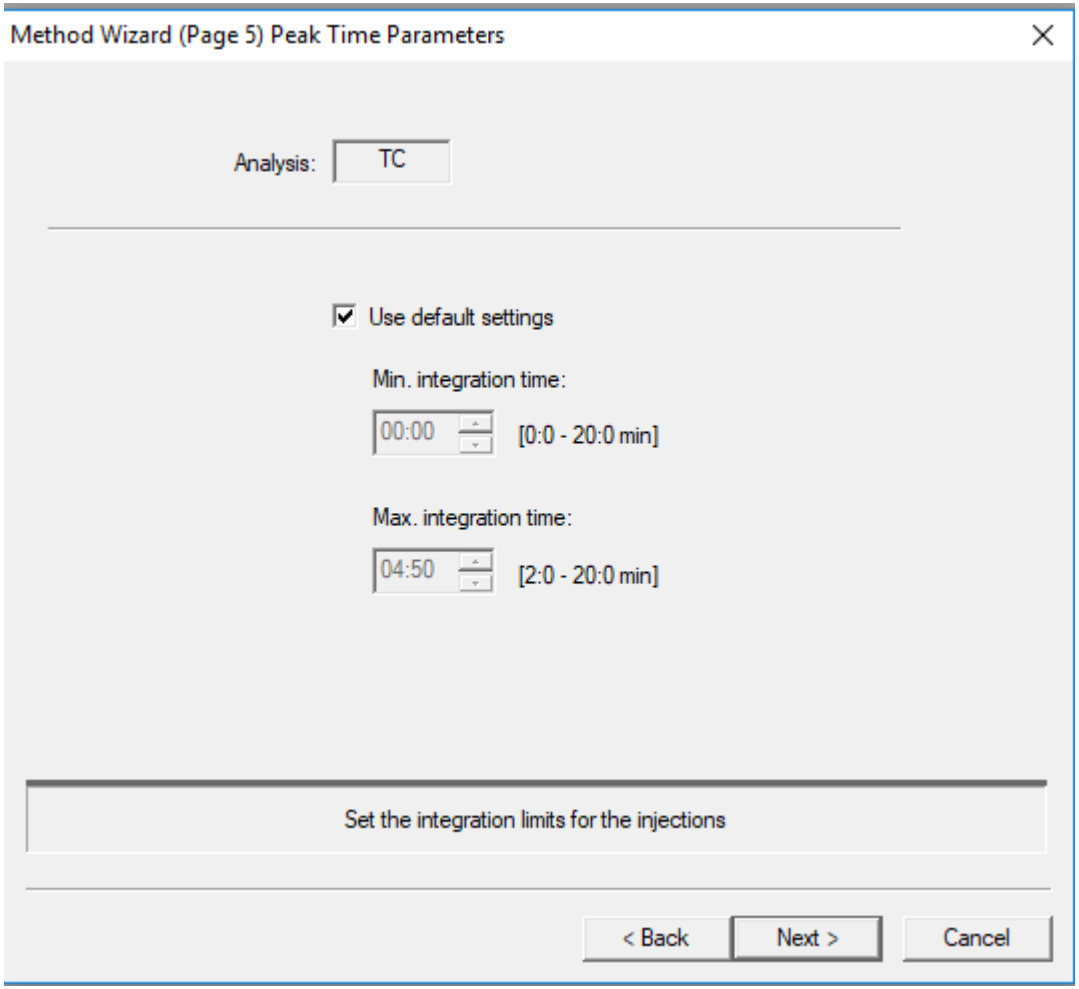


Creating a Method

Confirm the peak time parameters for the TC analysis.

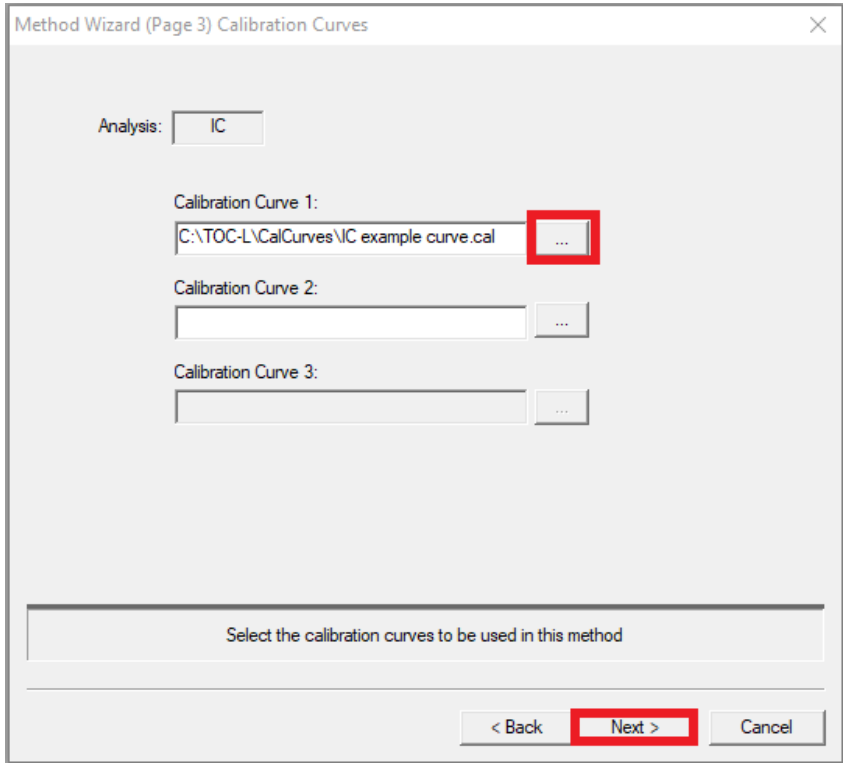
Note that defaults settings are OK for 99% of analysis.

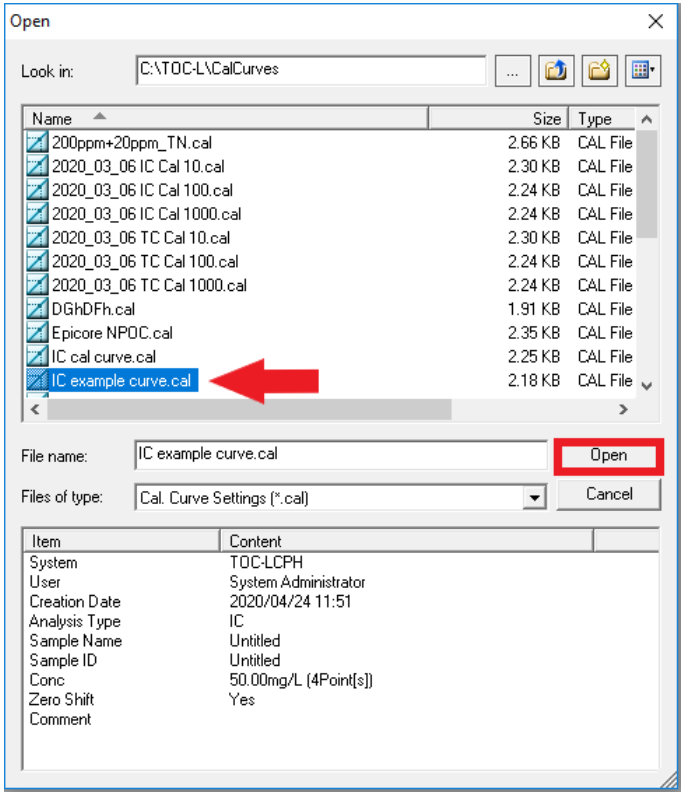
Select ‘Next’ to move on and set the parameters for the IC measurement.



Creating a Method

1. Push the '…' button to select the curve for IC analysis.
2. Select the IC curve to be used for analysis.
3. Push 'Open' to select the curve and insert in 'Calibration Curve 1'
4. Push 'Next'

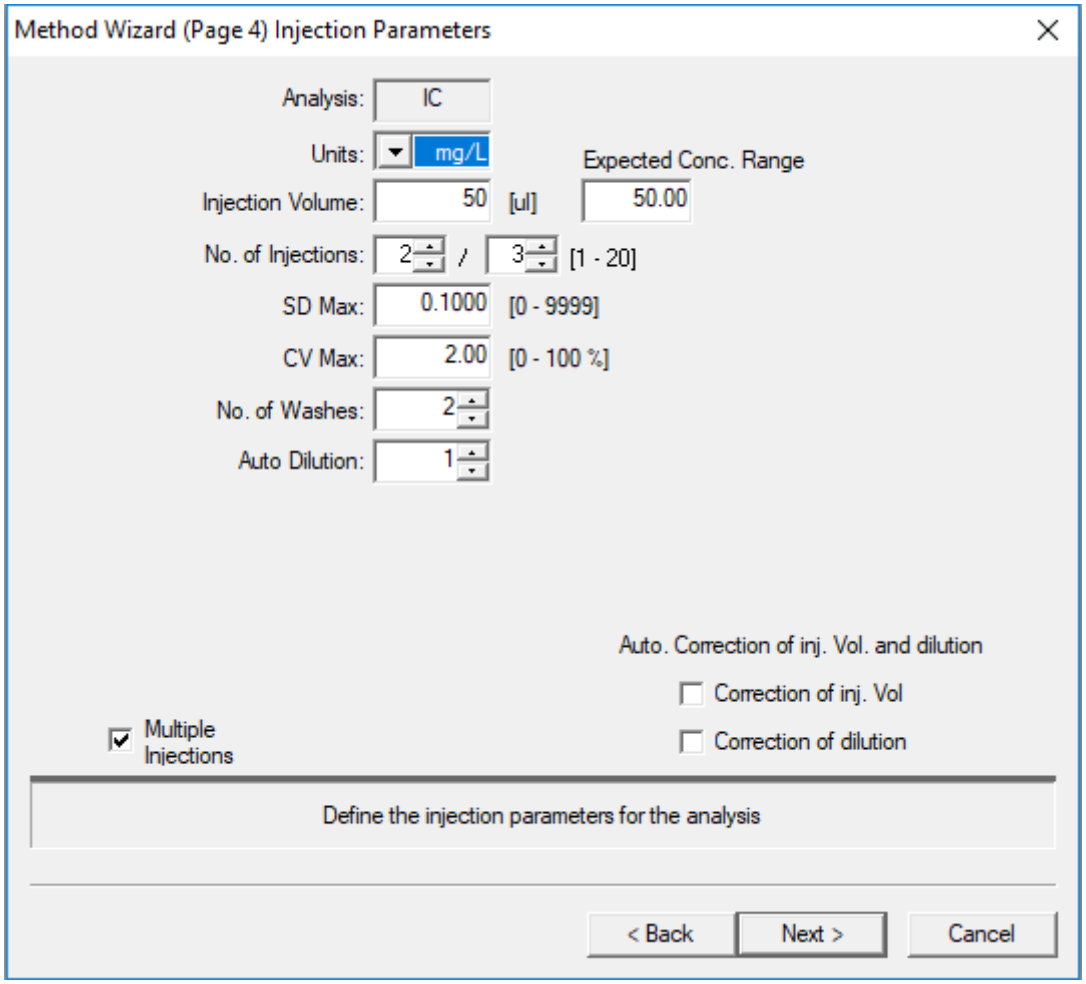




Creating a Method

Confirm the conditions for the IC analysis. Note that these conditions were pulled in from the calibration curve selected but can be changed if needed.

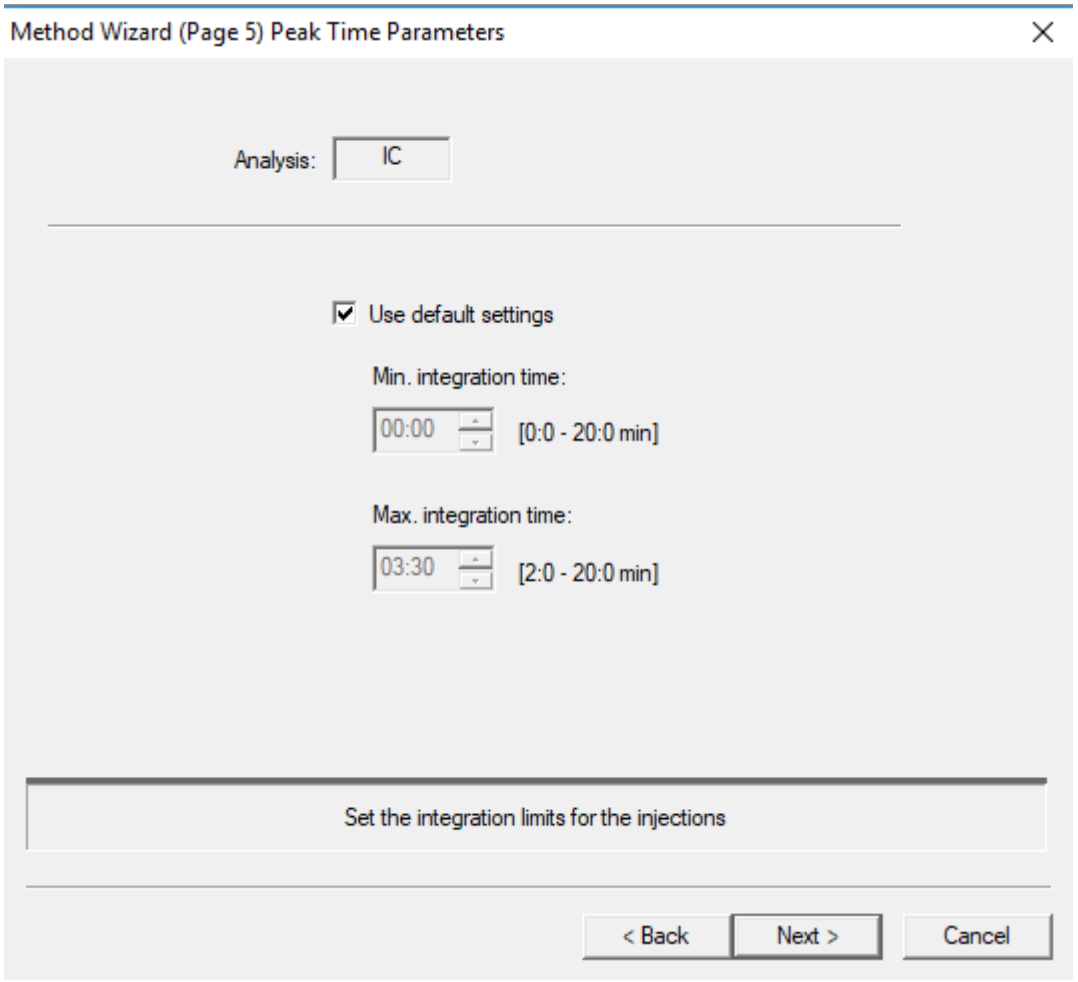
Check the box next to ‘Correction of inj. Vol’ or ‘Correction of dilution’ if you wish to have the instrument auto-dilute or lower injection volume for sample that are over the range of the calibration curve.



Creating a Method

Confirm the peak time parameters for the IC analysis.

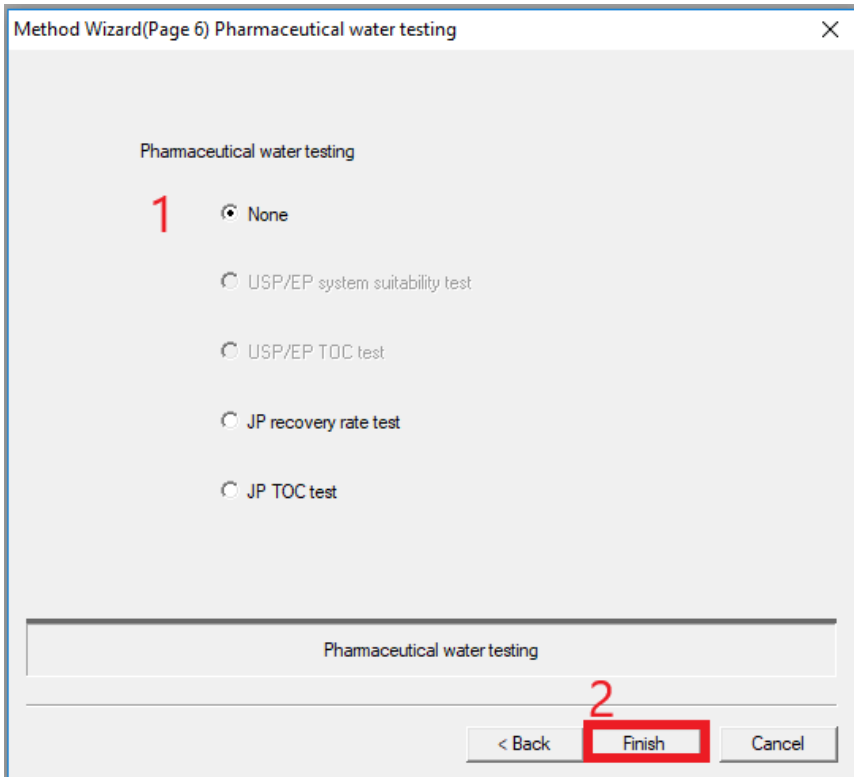
Note that the default parameters are OK for 99% of analysis.

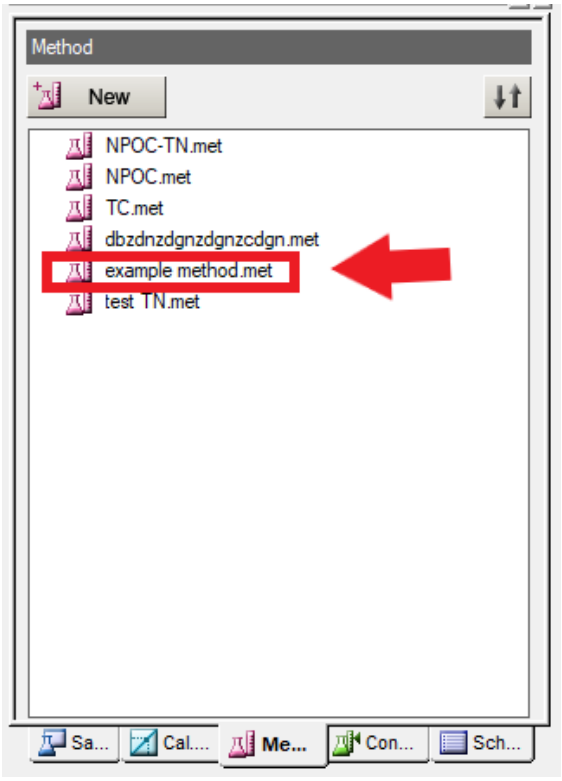


Creating a Method

1. If this method is pharmaceutical testing select the appropriate test otherwise select ‘None’

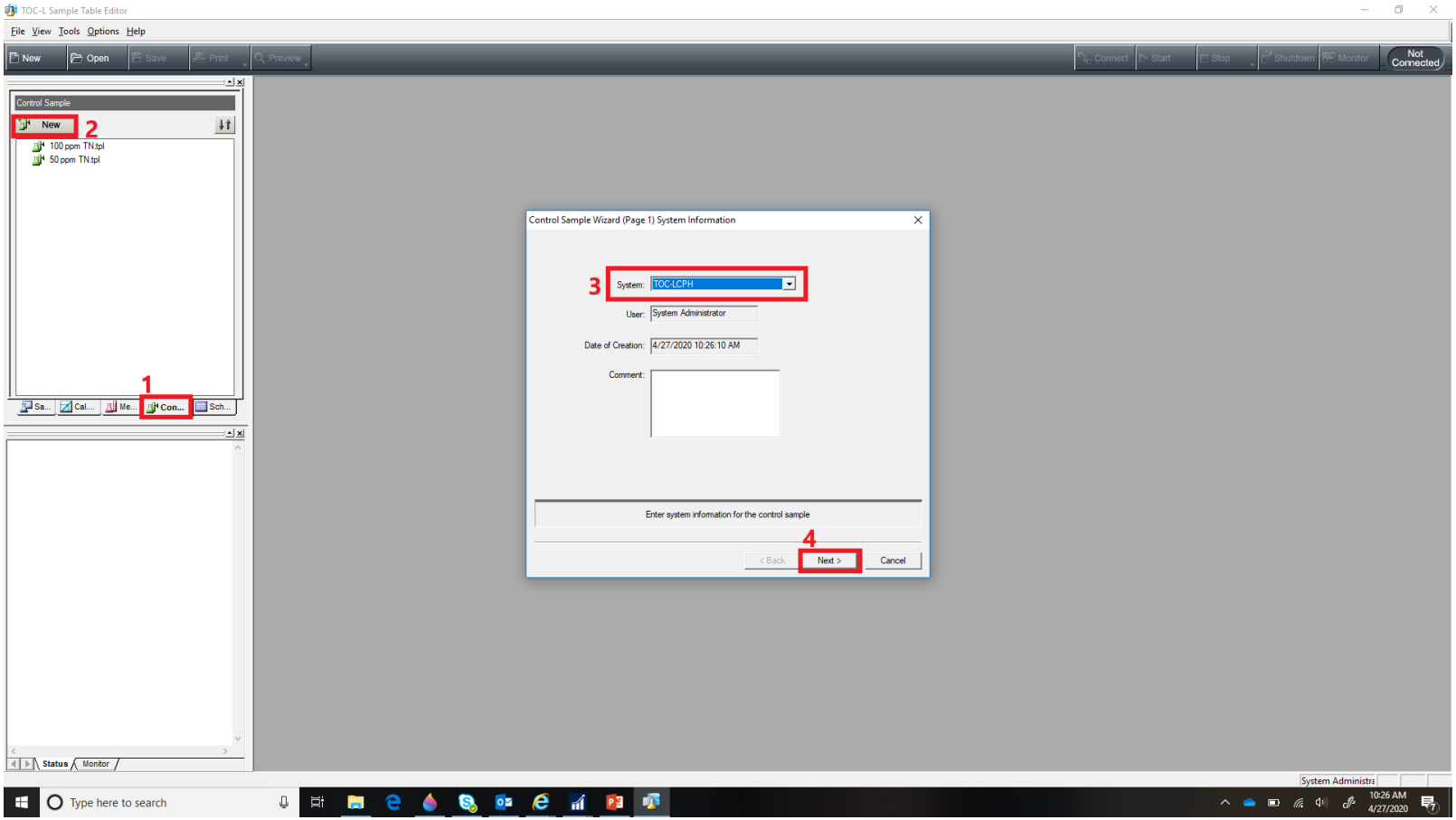
2. Push ‘Finish’ to save the method to the list of Methods.





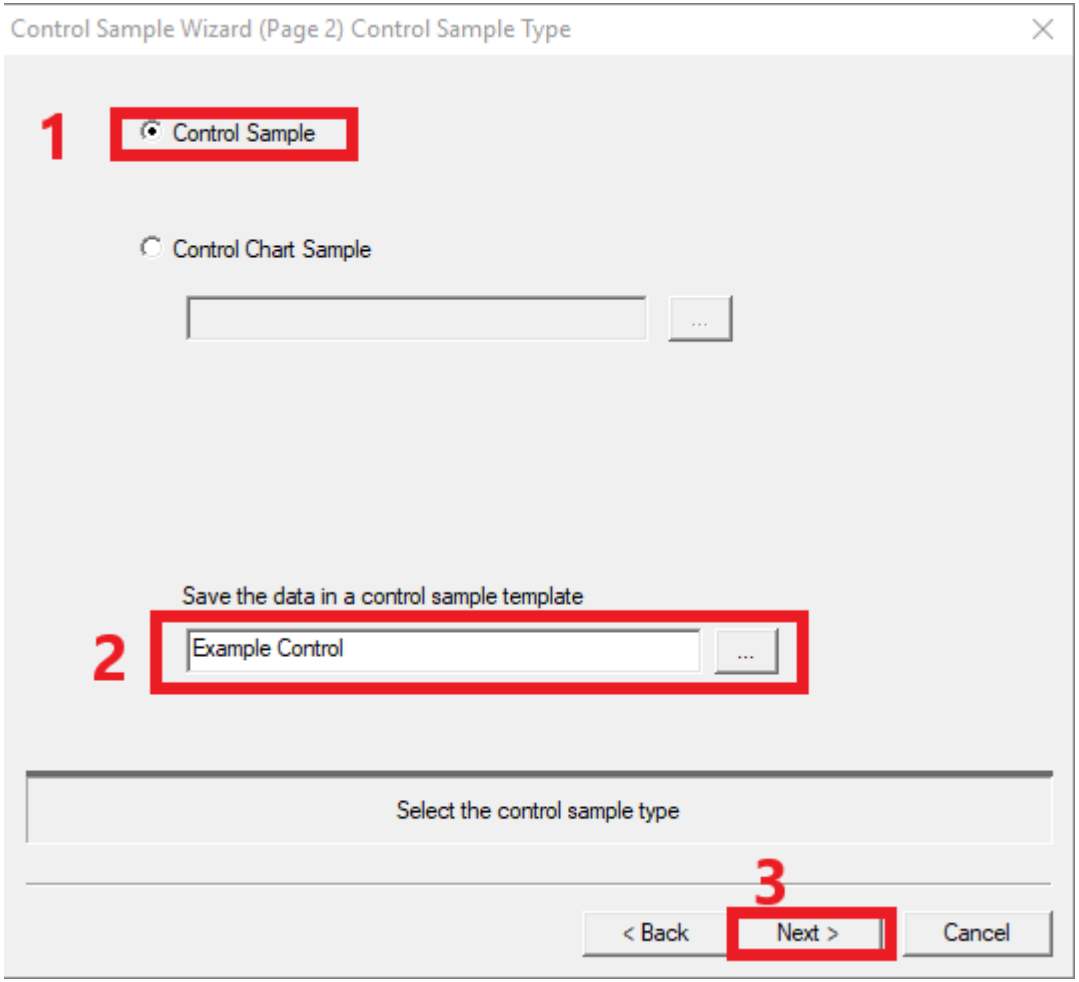
Creating Control Samples

1. Push the ‘Control Samples’ button to show the list of control samples
2. Push the ‘New’ button to create a new control sample
3. Select the correct hardware configuration from the drop down list
4. Press ‘Next’

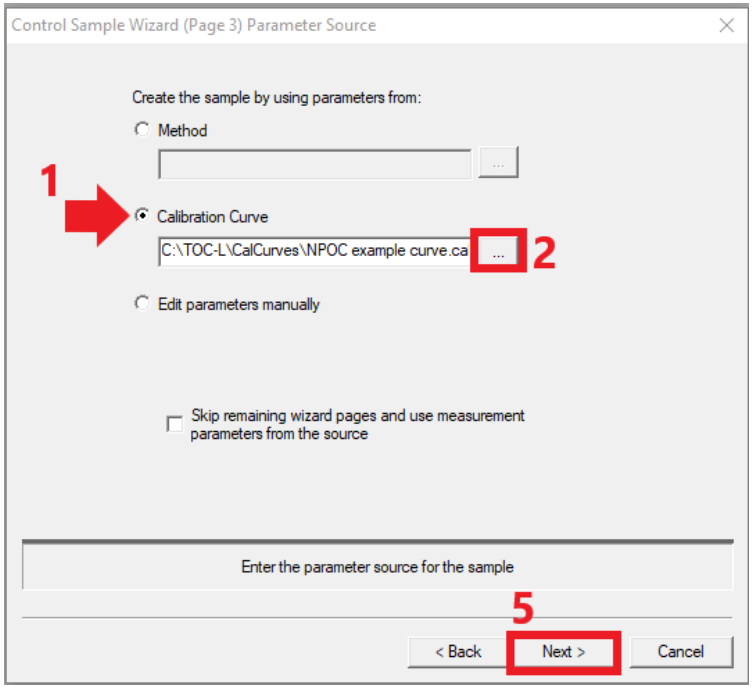


Creating Control Samples

1. Select ‘Control Sample’
2. Give the control sample a unique name
3. Press ‘Next’



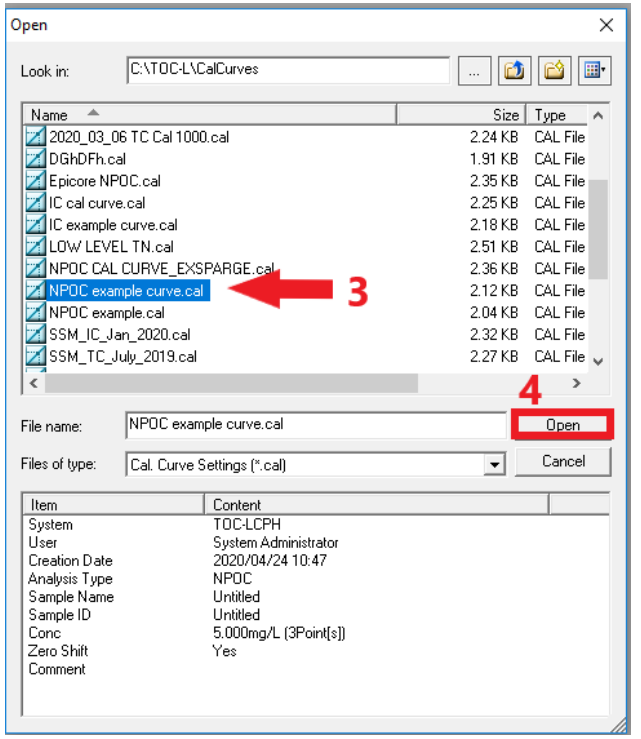
Creating Control Samples



1. A control sample can be analyzed using either a method file or calibration curve file. In This example a calibration curve file will be used
2. Push the '…' button to open the list of calibration curves
3. Select the calibration curve to be used

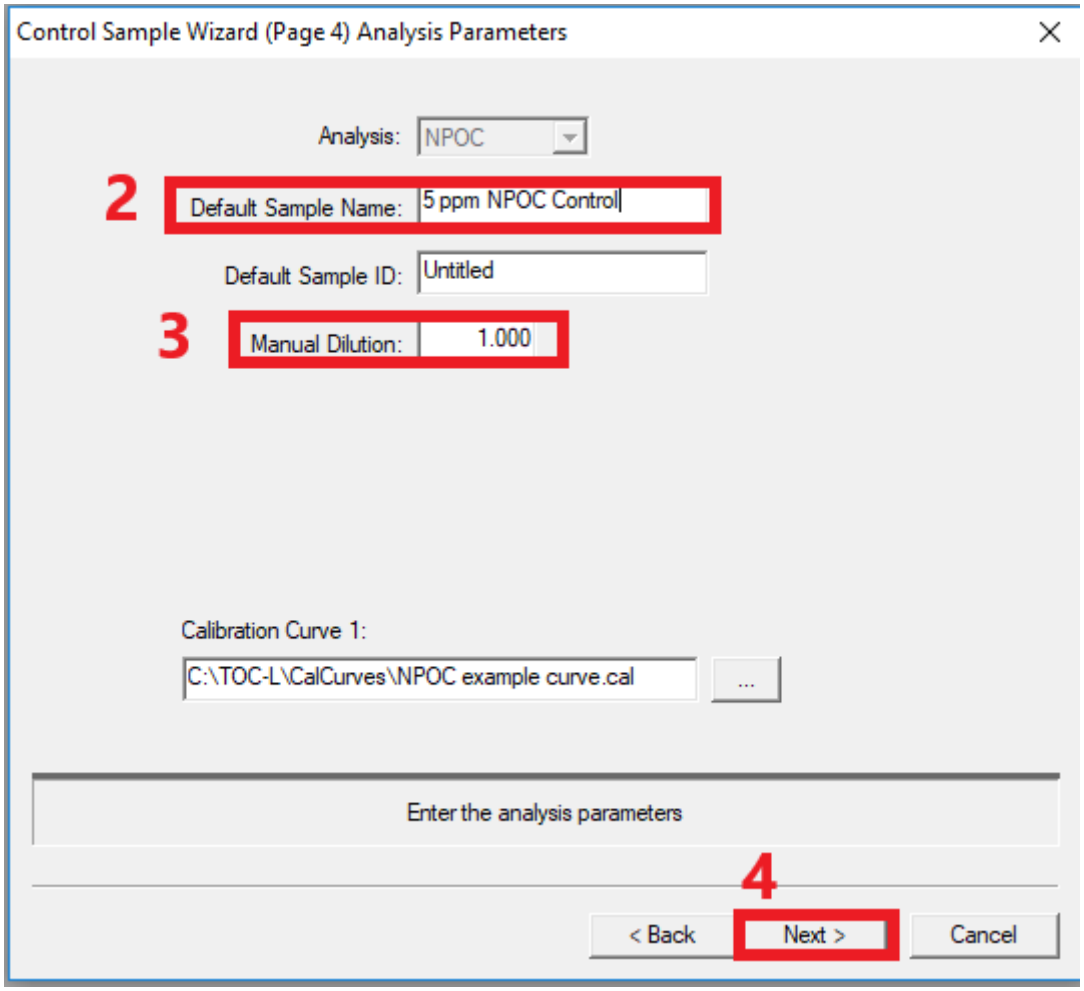
4. Push 'Open' to select the calibration curve

5. Push 'Next'



Creating Control Samples

1. Confirm that the conditions on this page are correct.
2. Give the control sample a unique default sample name which will appear when the control sample is inserted into a sample table.
3. If there is a manual dilution being performed on the samples select the appropriate dilution rate.
4. Press ‘Next’

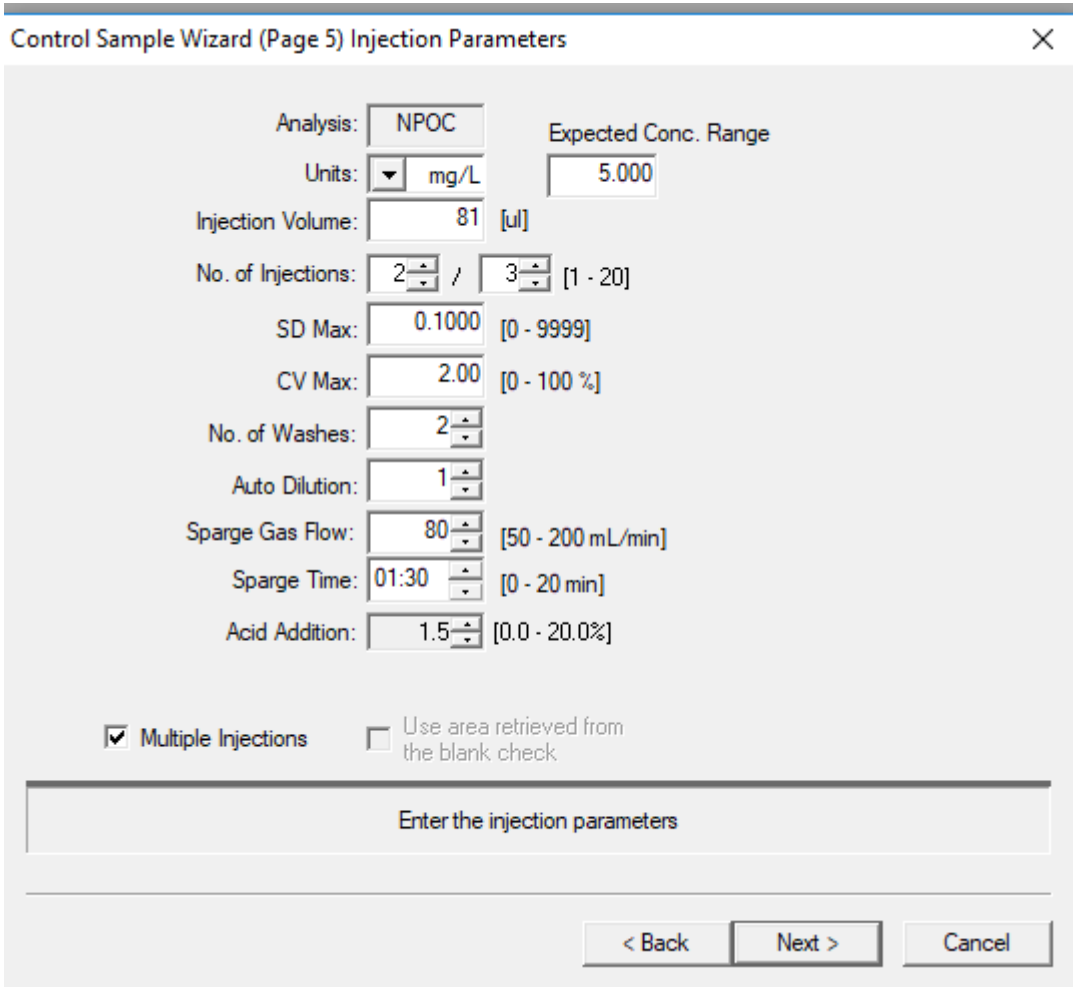


Creating Control Samples

Confirm the condition for analysis.

note: the parameters that appear on this page are the ones selected in the calibration curve or method file being used.

Press ‘Next’

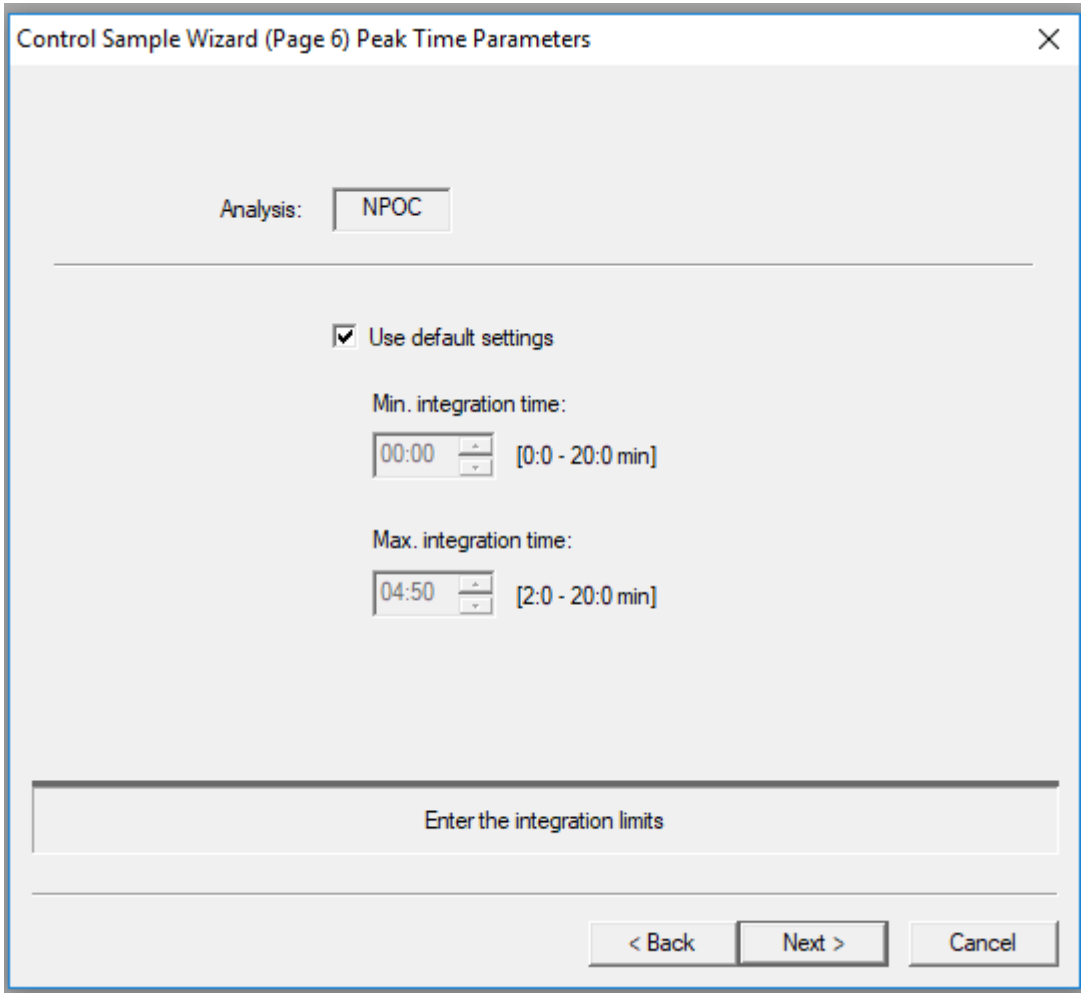


Creating Control Samples

Confirm the Peak Time Parameters

Note: the default parameters will be OK for 99% of analysis.

Press ‘Next’

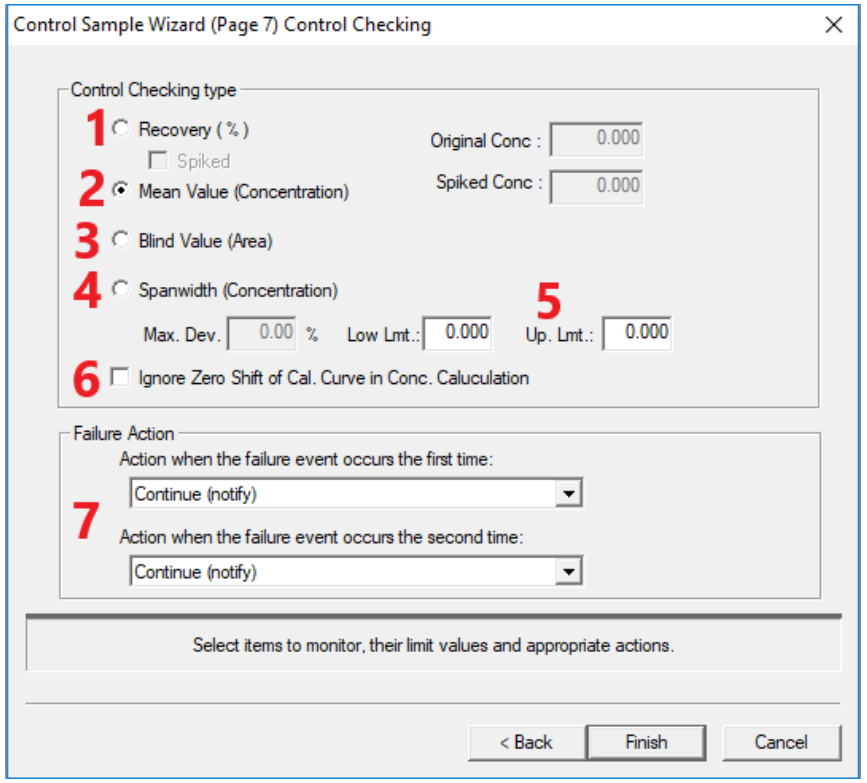


Creating Control Samples

Control samples can be set-up many different ways

1. Percent recovery of the control sample or percent recovery of a spiked sample
2. Mean value of the measured concentration of the control sample
3. Blind value area counts of the measured control sample
4. Spanwidth of the measured control sample concentration

5. This is where the accepted criteria is entered for each control checking type
6. This is where you have the option to ignore the zero shift of the calibration curve when calculating the controls. This should be selected if the control sample is prepped in the same water as the calibration curve standards. If the control sample is prepped in different water than the calibration curve standards then do not check this box
7. This is where you can select actions to be performed upon the failure of a control sample



Creating Control Samples

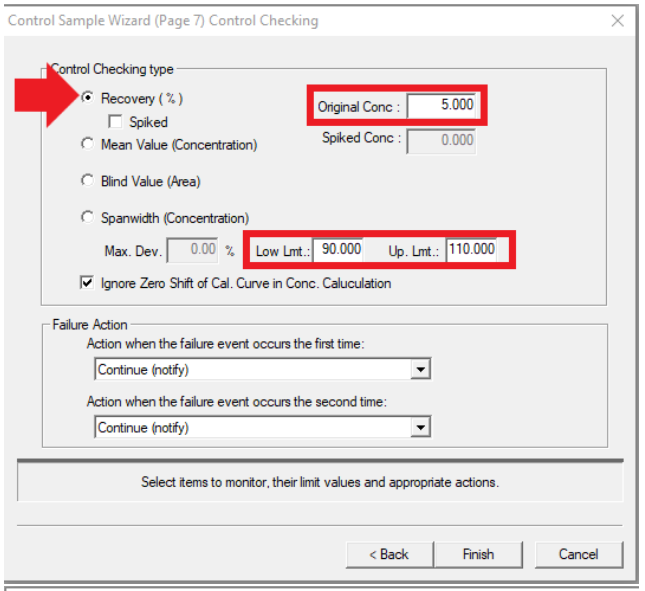
Recovery %

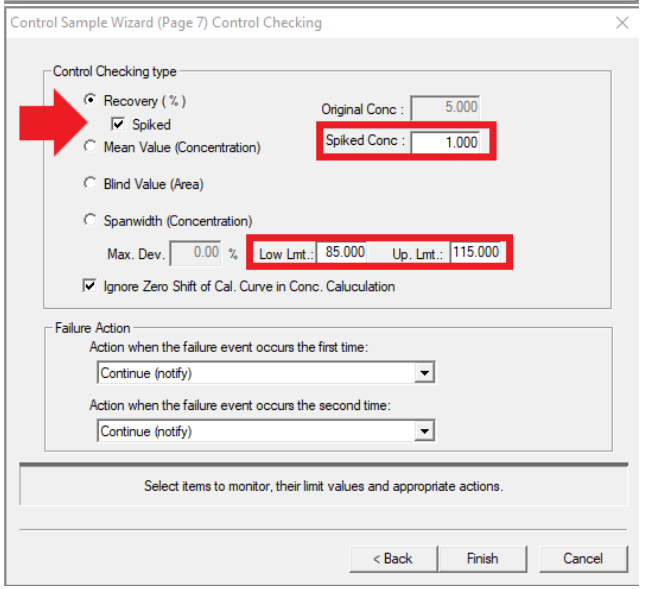
Select this control checking type to calclulate the % recovery a control sample or check ‘Spiked’ to calculate the % recovery of the amount spiked into sample.

Enter the correct concentration of the control sample or the amount spiked into the sample.

Enter the correct acceptance criteria for the analysis. In the top example, a control sample that is measured between 90%-110% of 5 ppm would pass. In the bottom example, a recovery of 85%-115% of the 1 ppm spiked into the sample would pass.

Note: when ‘Spiked’ is selected when you insert the control into the sample table, two lines in the table will be entered. The first line is for the un-spiked sample and the second line is for the spiked sample. The result of the sample spike is calculated as the difference between these two values.



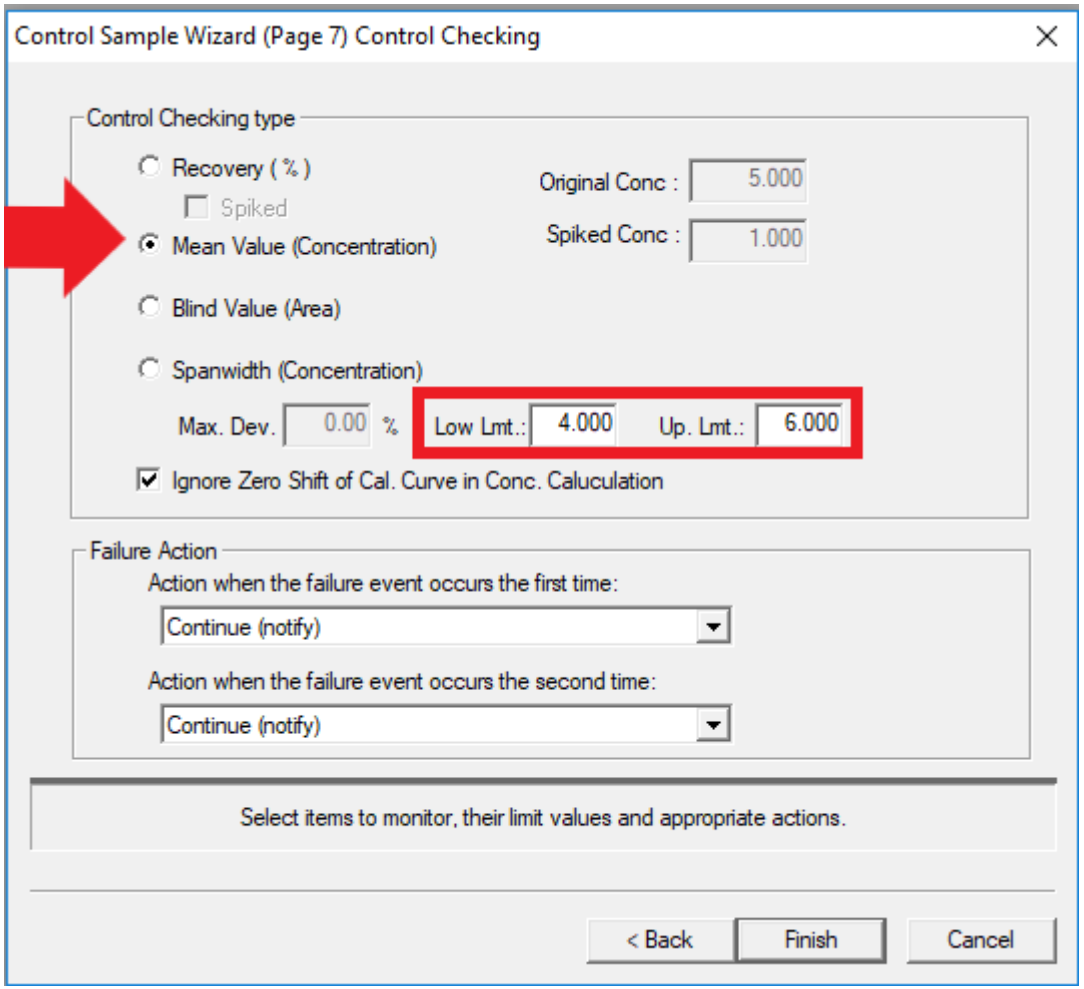


Creating Control Samples

Mean Value Concentration

Select this control checking type to determine a passing control sample using a measured concentration.

In this example, a 5 ppm control is used and the acceptance criteria is a measured concentration between 4 an 6 ppm.

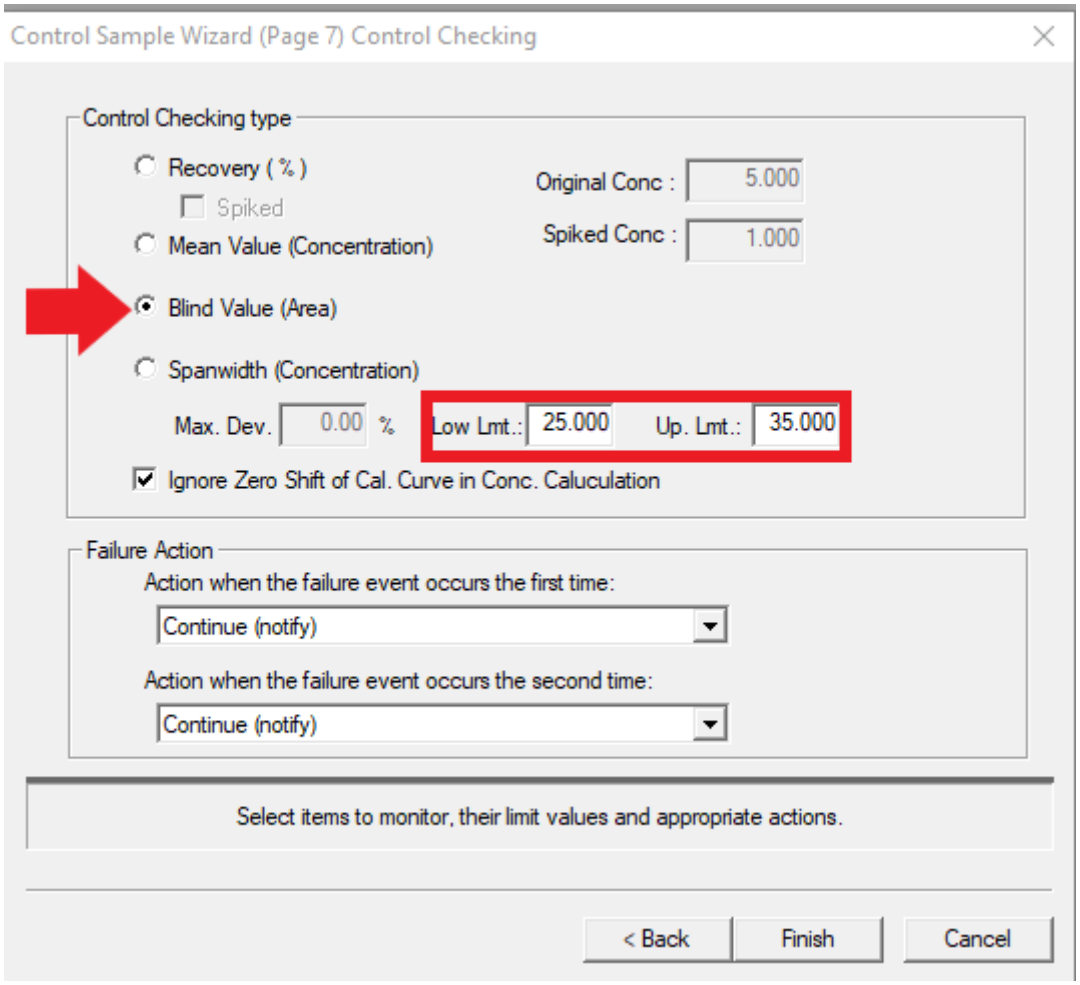


Creating Control Samples

Blind Value of Area

Select this control checking type to calculate the control sample based on area count response of the sample.

In this example the acceptance criteria for the control sample is an area count response between 25 and 35 area counts.

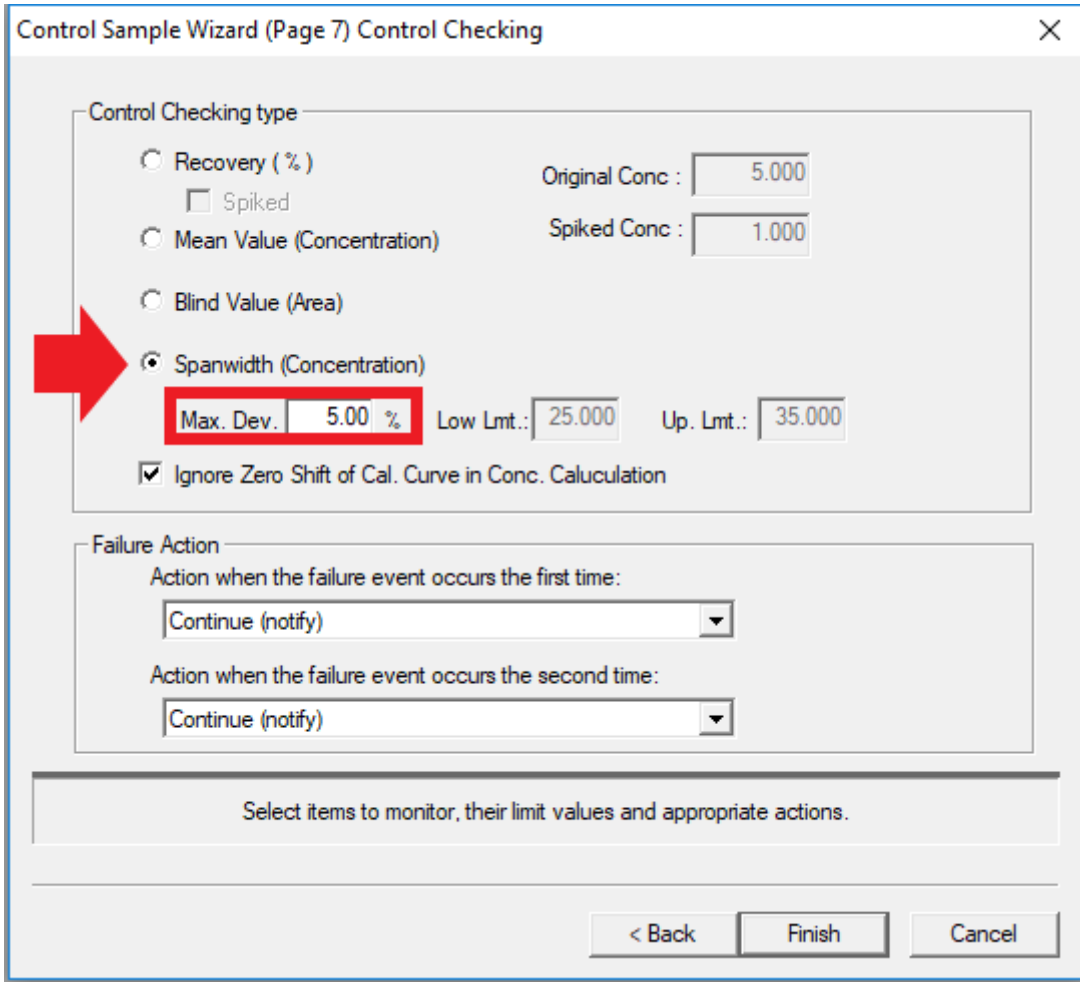


Creating Control Samples

Spanwidth of Control Concentration

Use this control checking type to calculate the control sample based on the maximum deviation of replicate injections.

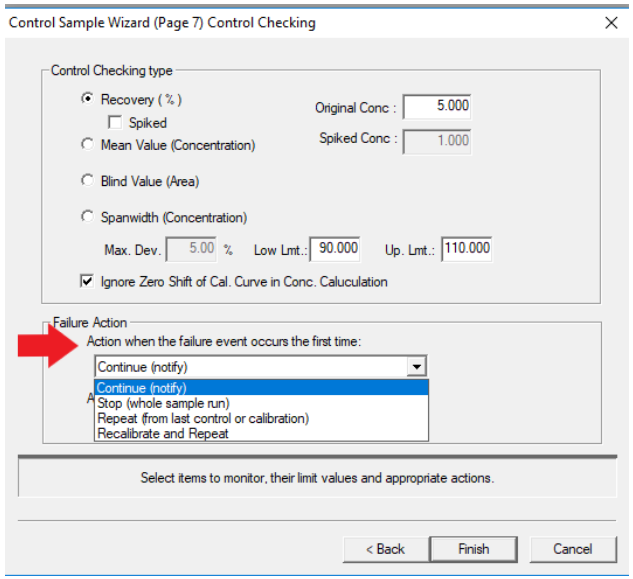
In this example the acceptance criteria is replicate control sample injections that do not deviate between 5%.



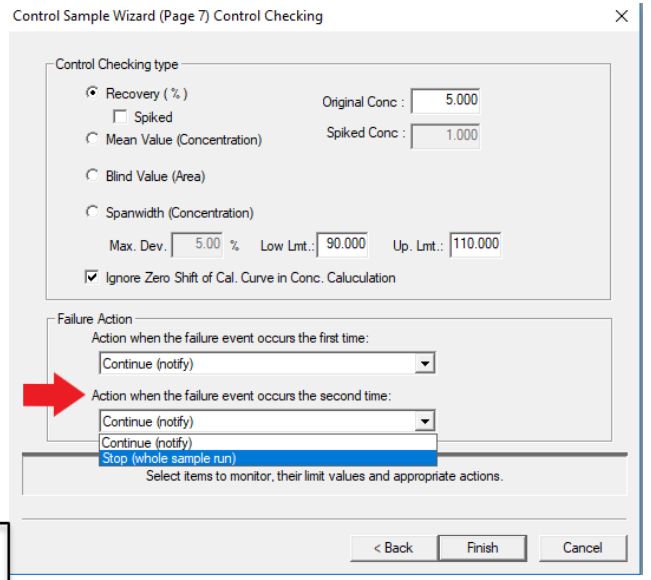
Creating Control Samples

Selecting actions for control failures

Select the desired actions for first time failure of a control sample.

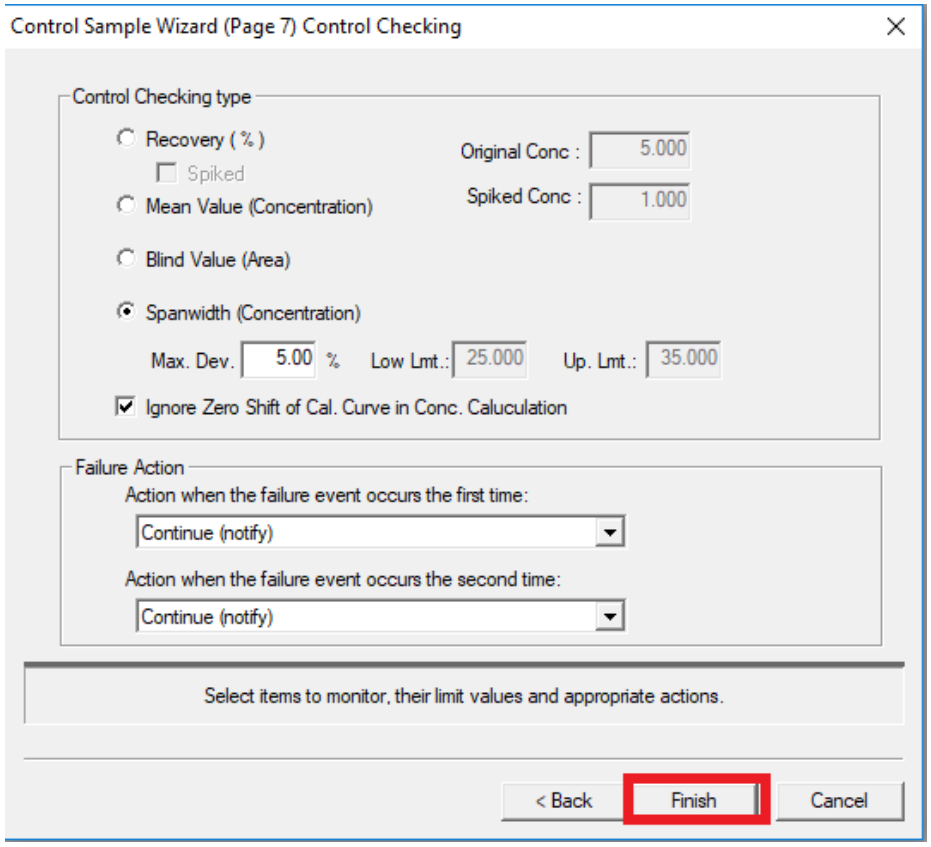


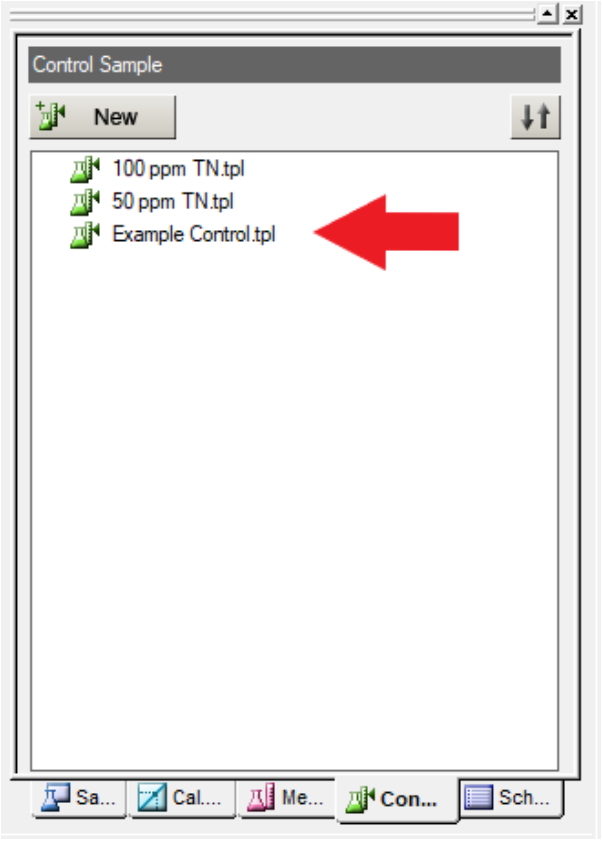
If applicable based on the action selected for the first failed control sample, select the actions for a second failure of the control sample.



Creating Control Samples

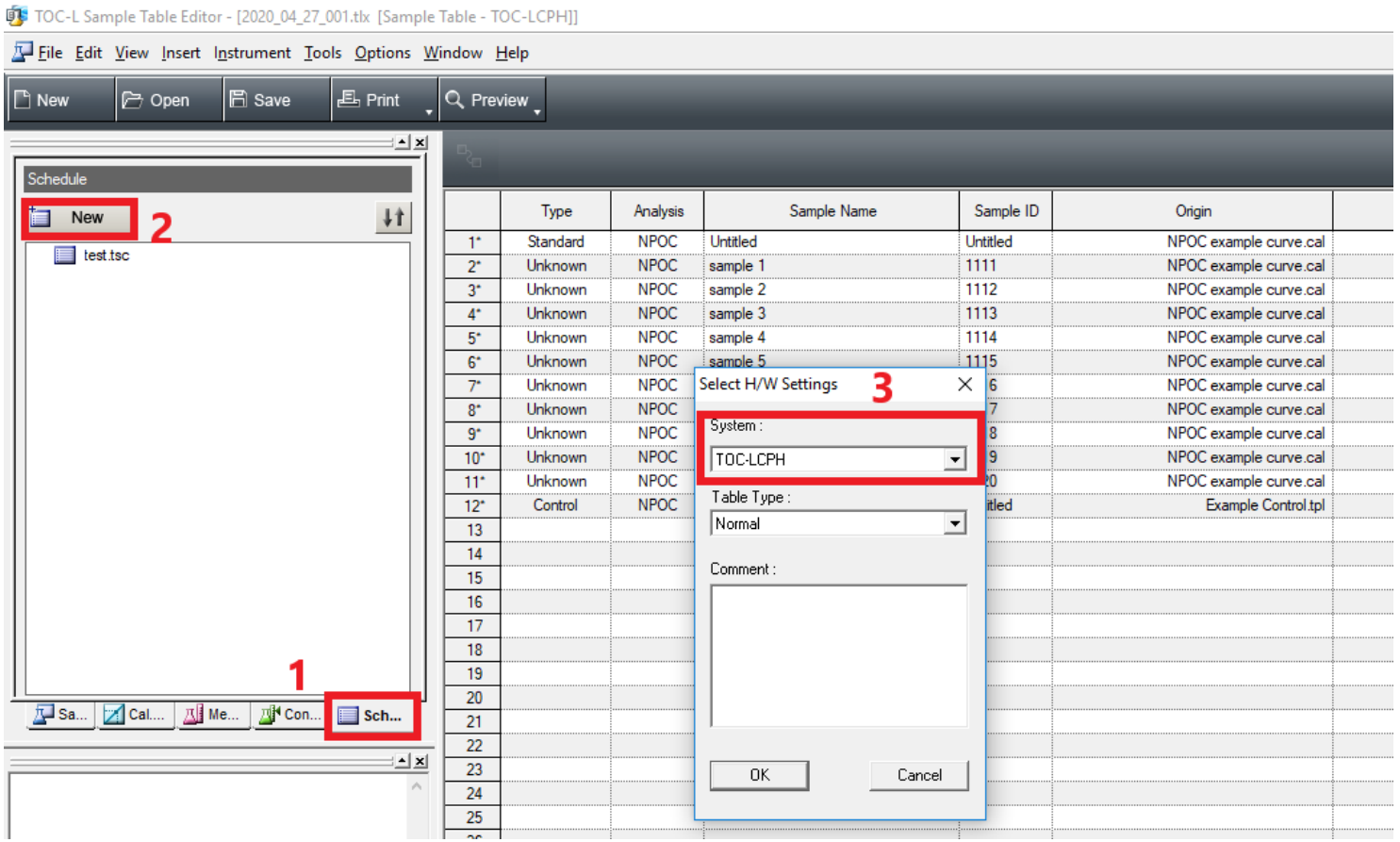
Select ‘Finish’ to save the control sample template to the list of control samples.





Creating a Schedule

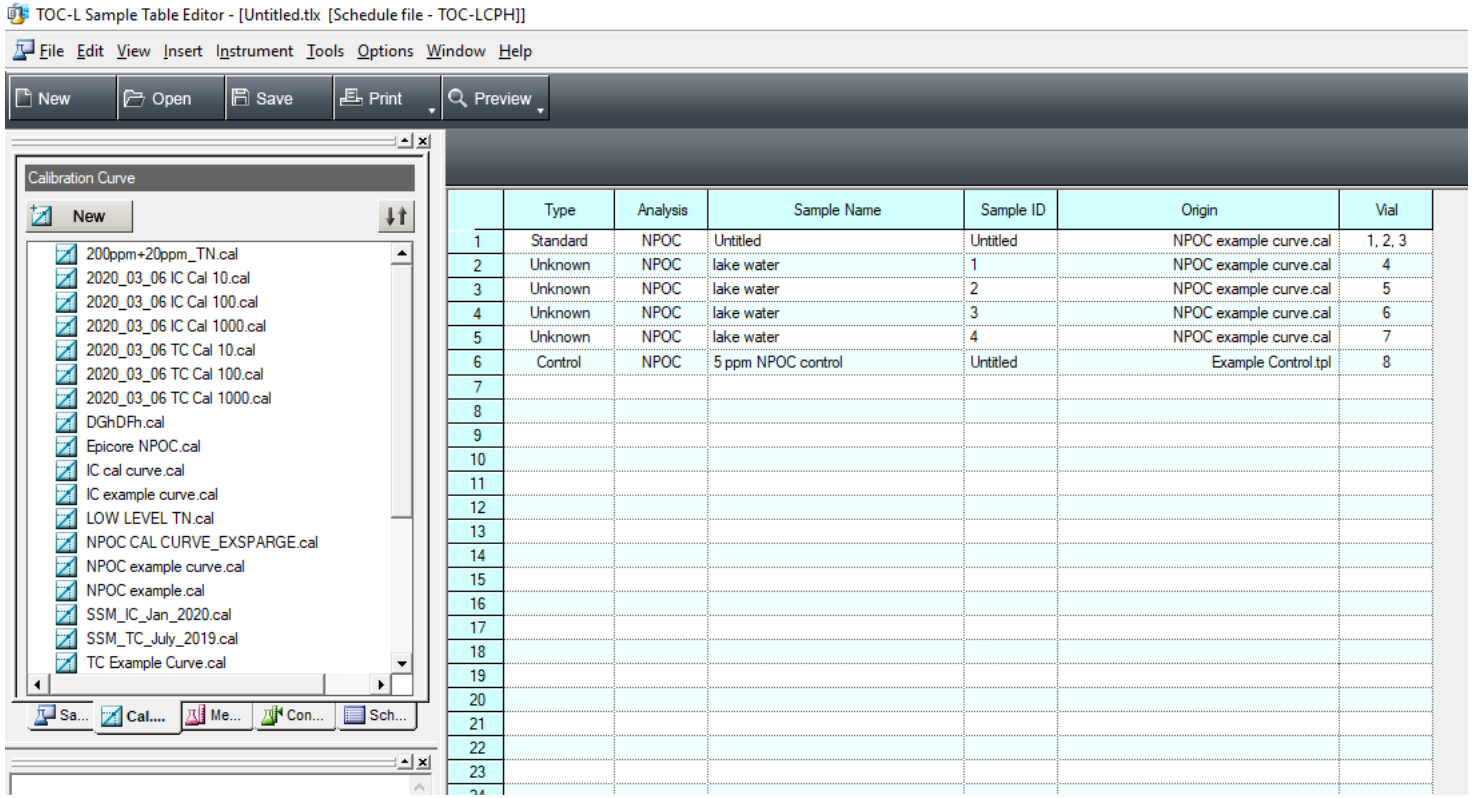
1. Push the ‘Schedule’ button to show the list of saved schedules.
2. Push the ‘New’ button to create a new schedule.
3. Use the drop down list to select the correct hardware configuration



Creating a Schedule

A schedule is a stored file which contains measurement parameters for multiple samples, including the specified sequence of analyses. The saved content of the file can be called up as desired, and loaded into a sample table.

Note: Analysis based on the information in a schedule is conducted by loading the information into a sample table. Analysis cannot be conducted using the schedule file alone.



	Type	Analysis	Sample Name	Sample ID	Origin	Vial
1	Standard	NPOC	Untitled	Untitled	NPOC example curve.cal	1, 2, 3
2	Unknown	NPOC	lake water	1	NPOC example curve.cal	4
3	Unknown	NPOC	lake water	2	NPOC example curve.cal	5
4	Unknown	NPOC	lake water	3	NPOC example curve.cal	6
5	Unknown	NPOC	lake water	4	NPOC example curve.cal	7
6	Control	NPOC	5 ppm NPOC control	Untitled	Example Control.tpl	8
7						
8						
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10						
11						
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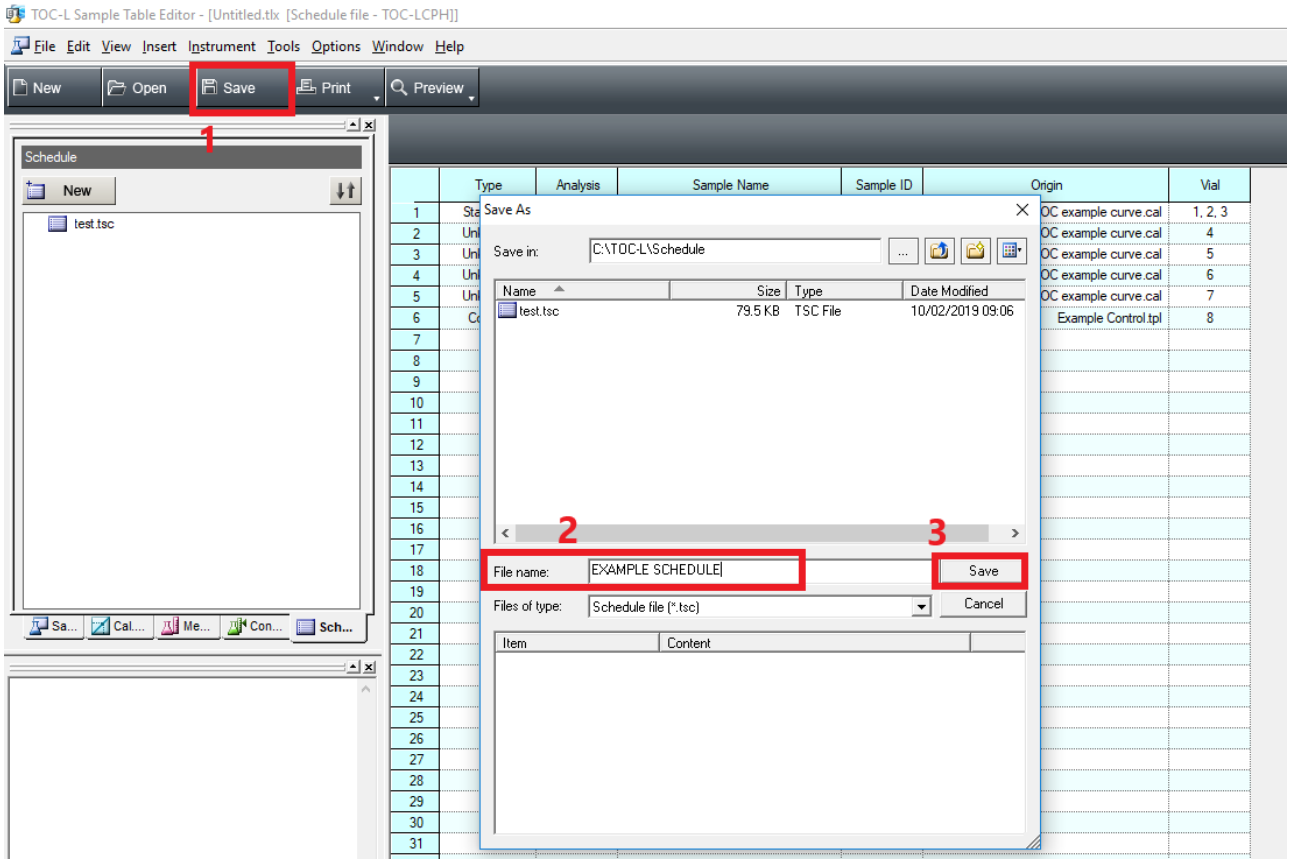
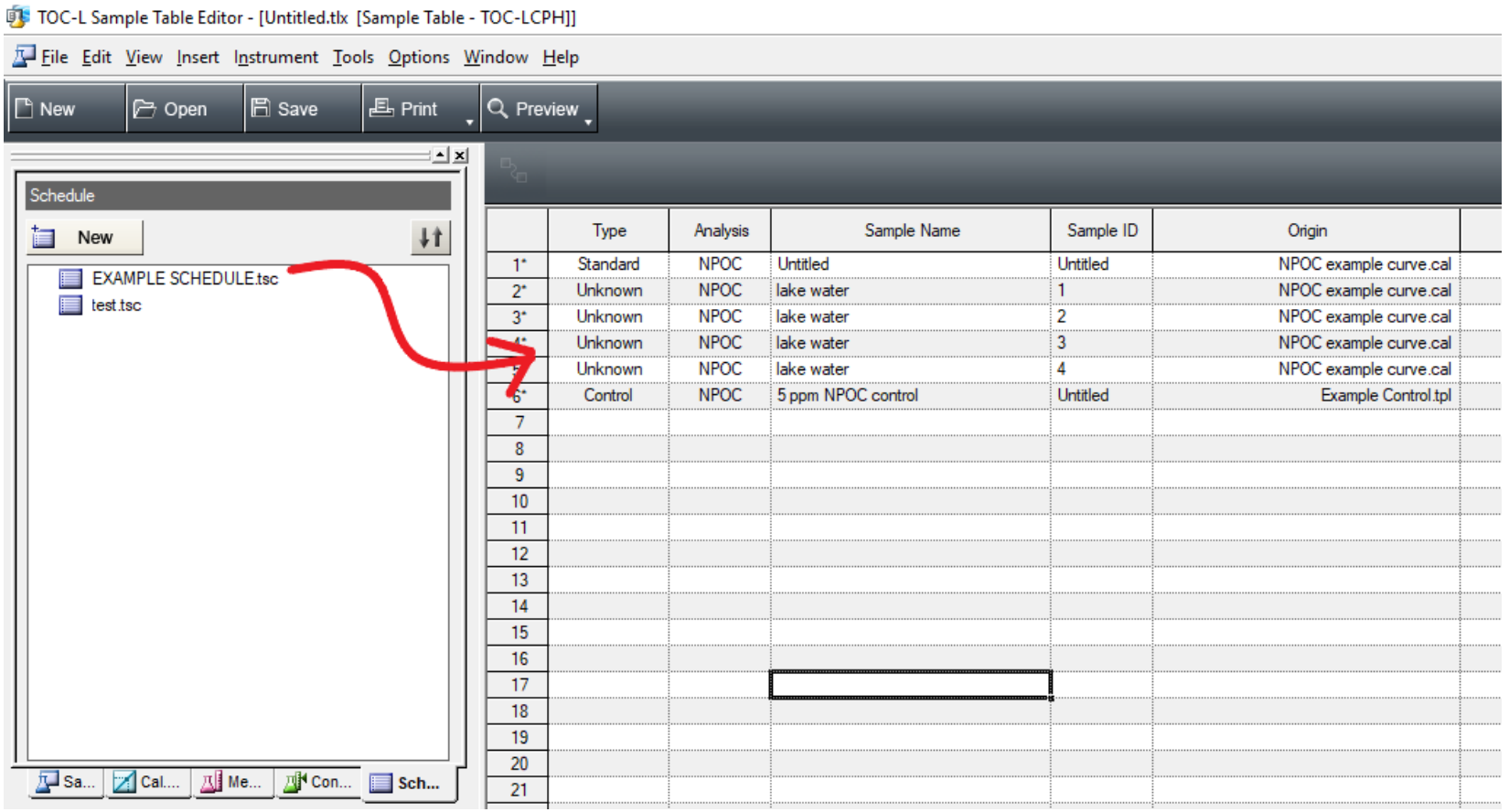
Creating a Schedule

1. To save the schedule, push ‘Save’.
2. Give the file a unique name.
3. Push ‘Save’



Creating a Schedule

A schedule file can then be inserted into a sample table. Schedule files are useful for labs where the same samples are run often.

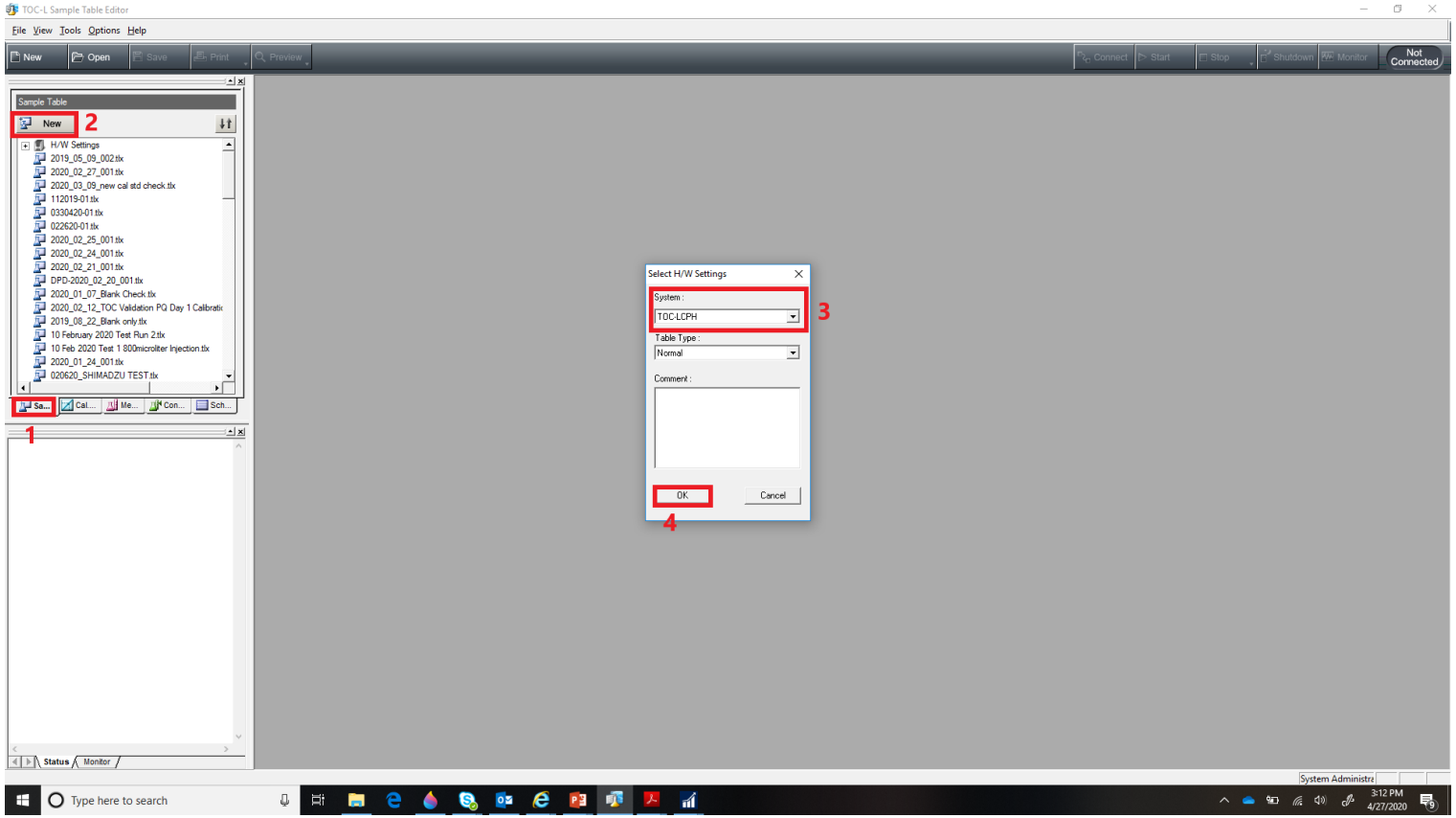


Return to Table of Contents

Section 4 / 4

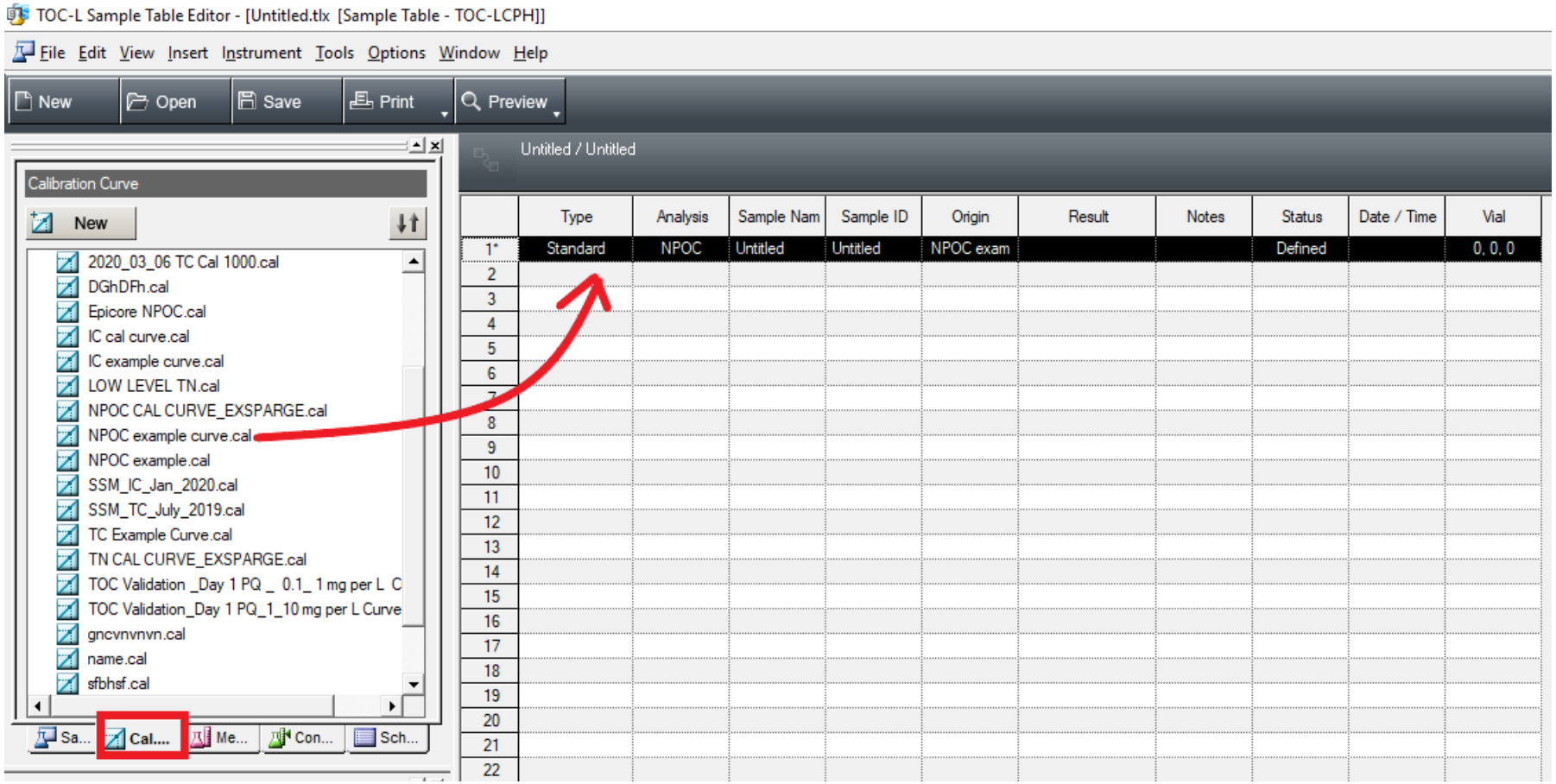
Creating a Sample Table

1. Push the ‘Sample Table’ button to show the list of saved sample tables.
2. Push the ‘New’ button to open a new table
3. Select the correct hardware configuration from the drop down list
4. Press ‘OK’



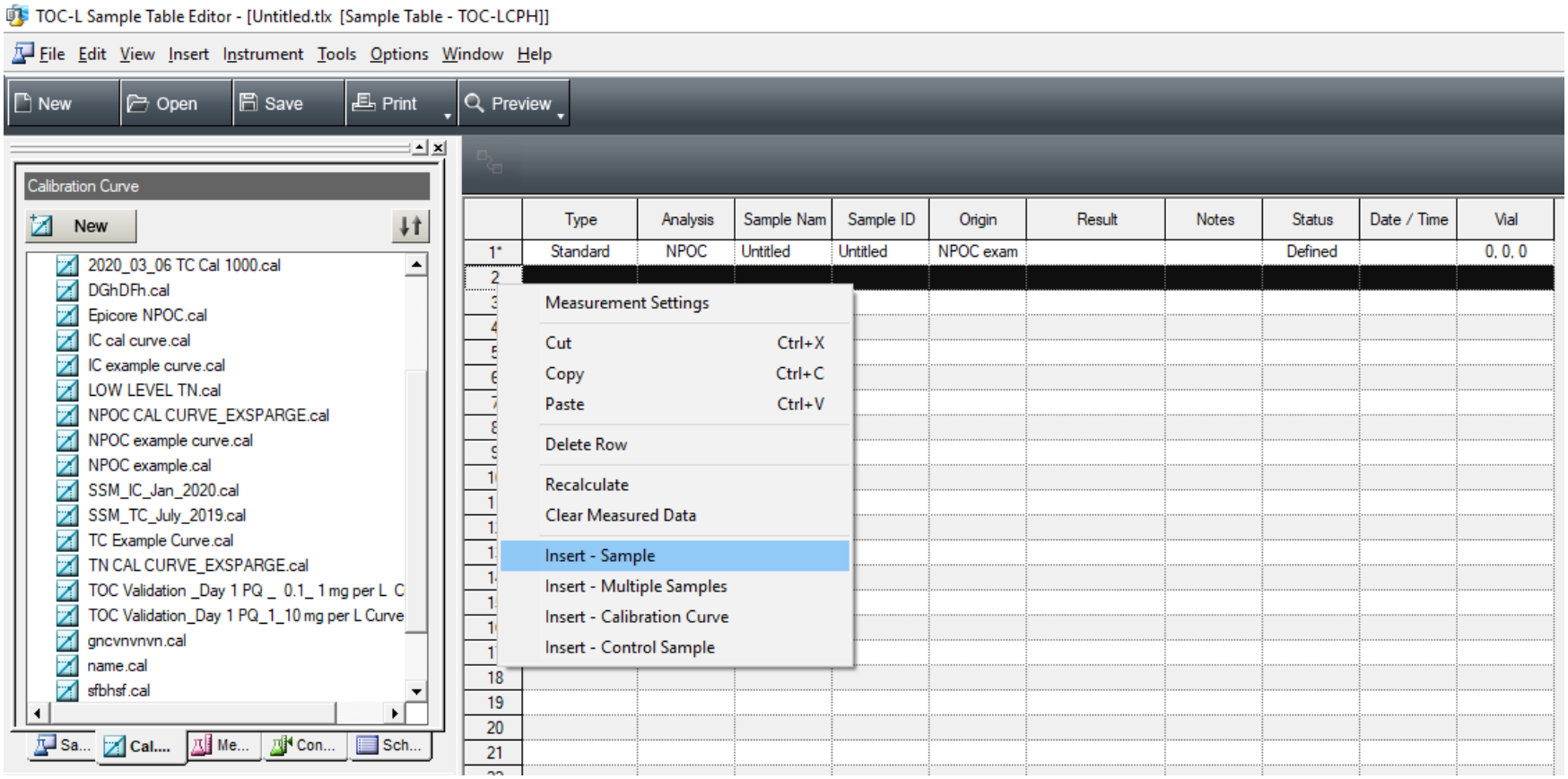
Creating a Sample Table

Push the ‘Calibration Curve’ button to display the saved calibration curves and drag and drop the desired Calibration curve into the sample table

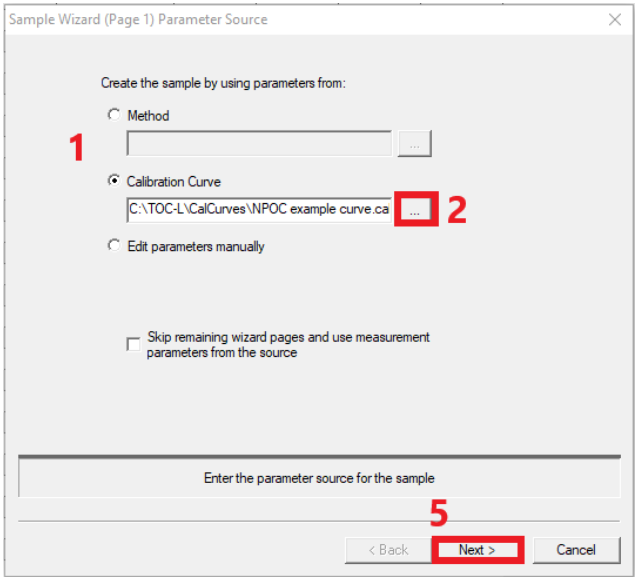


Creating a Sample Table

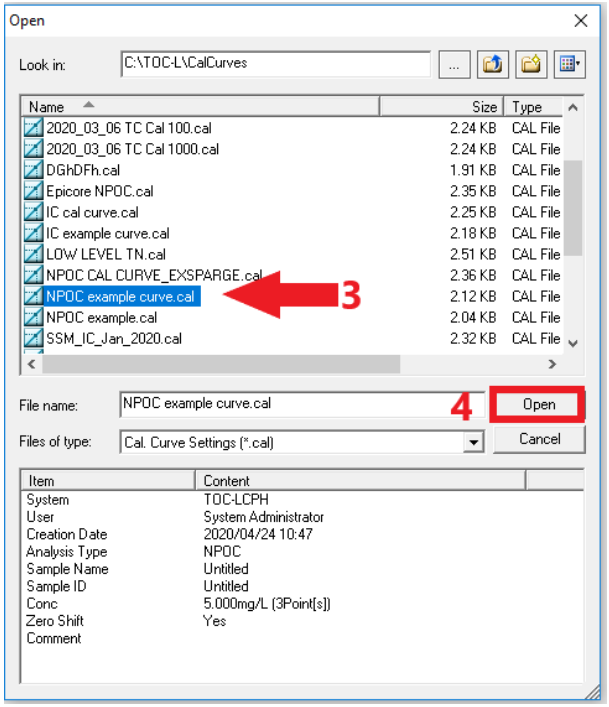
Highlight the next row in the table and right click on the mouse. From the drop down list select ‘Insert – Sample’



Creating a Sample Table

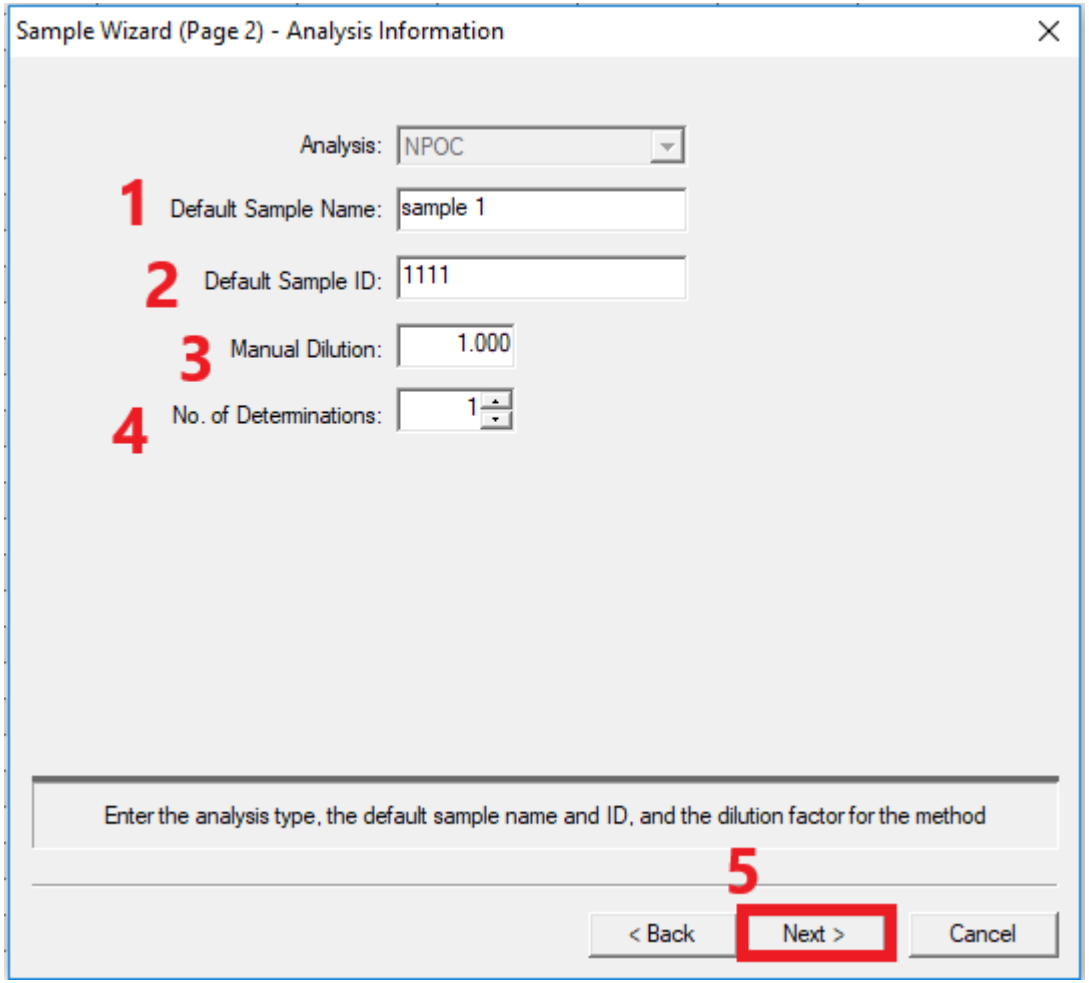


1. Select to either run your sample using a method or a calibration curve.
2. Push the '…' button to open the list of methods or calibration curves.
3. Select the desired method or calibration curve.
4. Push 'Open'
5. Push 'Next'



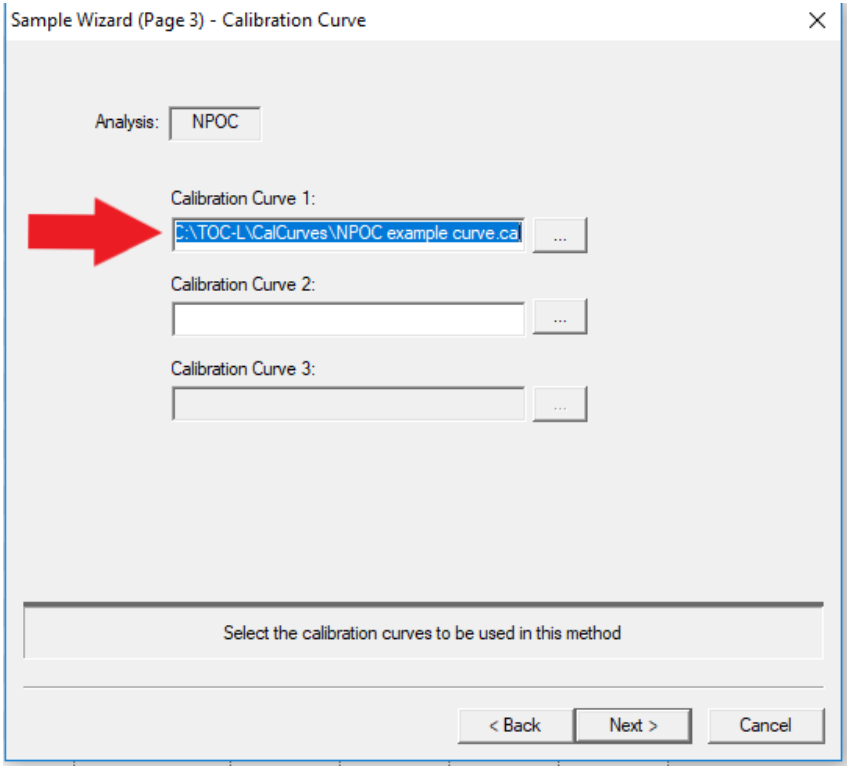
Creating a Sample Table

1. Give the sample a name
2. Give the sample an ID
3. If there was a manual dilution performed to the sample prior to running it on the TOC enter that factor here.
4. If you wish to perform the sample measurement more than once and then average those results together, enter that number here.
5. Press ‘Next’.



Creating a Sample Table

make sure the correct calibration curve is listed in ‘Calibration Curve 1’. Additional calibration curves can be added to positions 2 or 3 if desired.



The automatic selection process occurs on a measurement-by-measurement basis. Measurement values are always calculated first using Calibration Curve 1. The TOC-Control L software then evaluates the result, applies the optimum calibration curve, and recalculates the value if necessary. The calibration curve with a concentration range that is greater than and closest to the measured value is used. Examples are shown below:

(1) If the measured value is less than the concentration of Calibration Curve 1 and greater than the concentration of Calibration Curve 2 and Calibration Curve 3, Calibration Curve 1 is used.

Conc. of Calibration Curve 1 > Measured Value > Conc. of Calibration Curves 2 and 3 →

Calibration Curve 1 is used

(2) If the measured value is less than the concentrations of Calibration Curve 1 and Calibration Curve 2, and if the concentration of Calibration Curve 2 is less than that of Calibration Curve 1, Calibration Curve 2 is used.

Conc. of Calibration Curve 1 > Conc. of Calibration Curve 2 > Measured Value →

Calibration Curve 2 is used

(3) If three calibration curves are specified, the calibration curve that is greater than and closest to the measured value is used.

Conc. of Calibration Curve 1 > Conc. of Calibration Curve 3 > Measured Value > Conc. of Calibration Curve 2 →

Calibration Curve 3 is used

(4) If the measured value is greater than the concentration of all curves and the concentration of Calibration Curve 1 is greater than the concentration of Calibration Curves 2 and 3, Calibration Curve 1 is used.

Measured Value > Conc. of Calibration Curve 1 > Conc. of Calibration Curve 2 > Conc. of Calibration Curve 3 →

Calibration Curve 1 is used

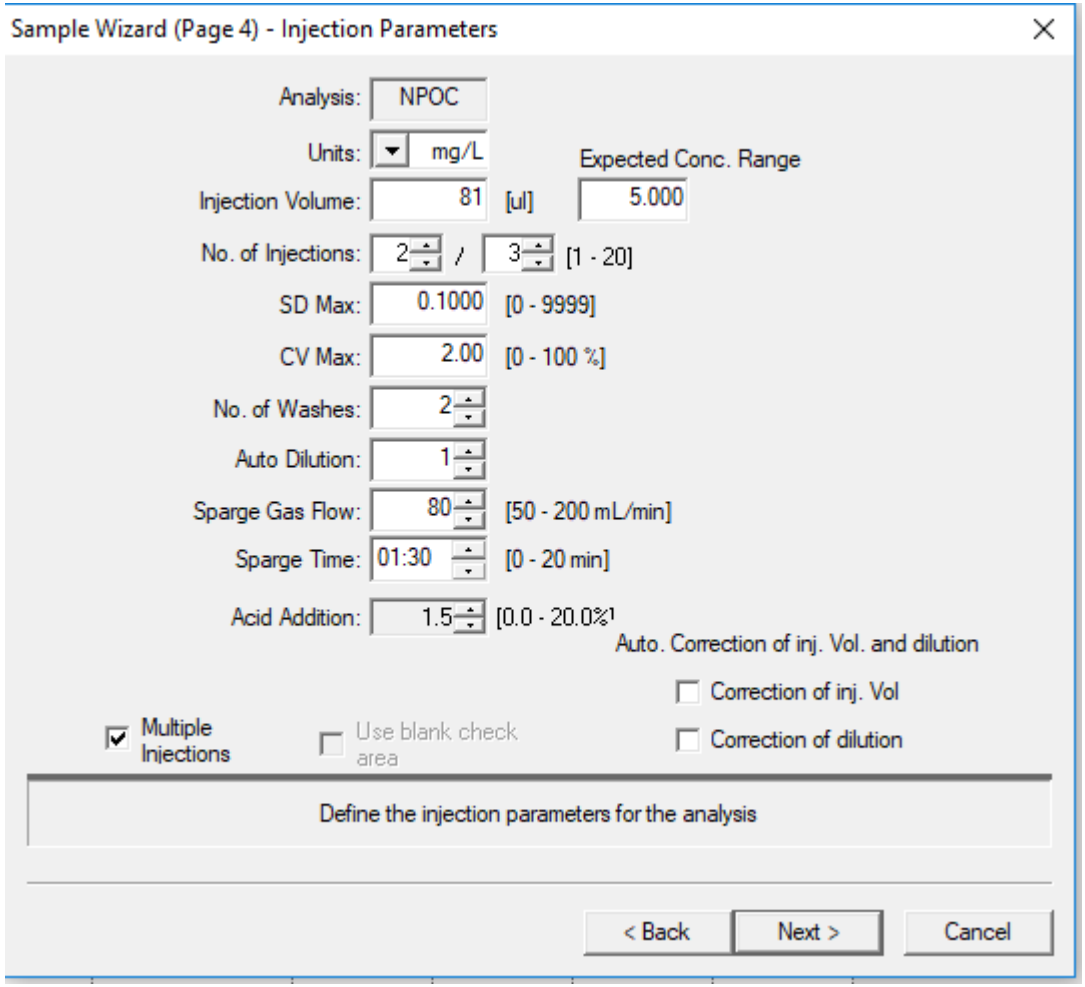
Note: By setting the concentration ratio between calibration curves to a factor of approximately 10, the automatic selection function can be effectively utilized for analyses covering a wide range of concentrations. For example, use a concentration of 10 mg/L for Calibration Curve 1, 100 mg/L for Calibration Curve 2, and 1000 mg/L for Calibration Curve 3.

Creating a Sample Table

Confirm that conditions in this window are correct.

Parameters in this window are pulled in from either the calibration curve or method file selected.

It is possible to have the instrument correct for samples over the range of the calibration curve. Select ‘Correction of inj. Vol’ to have the instrument lower the injection volume for over range samples or ‘Correction of dilution’ to have the instrument auto-dilute over range samples.

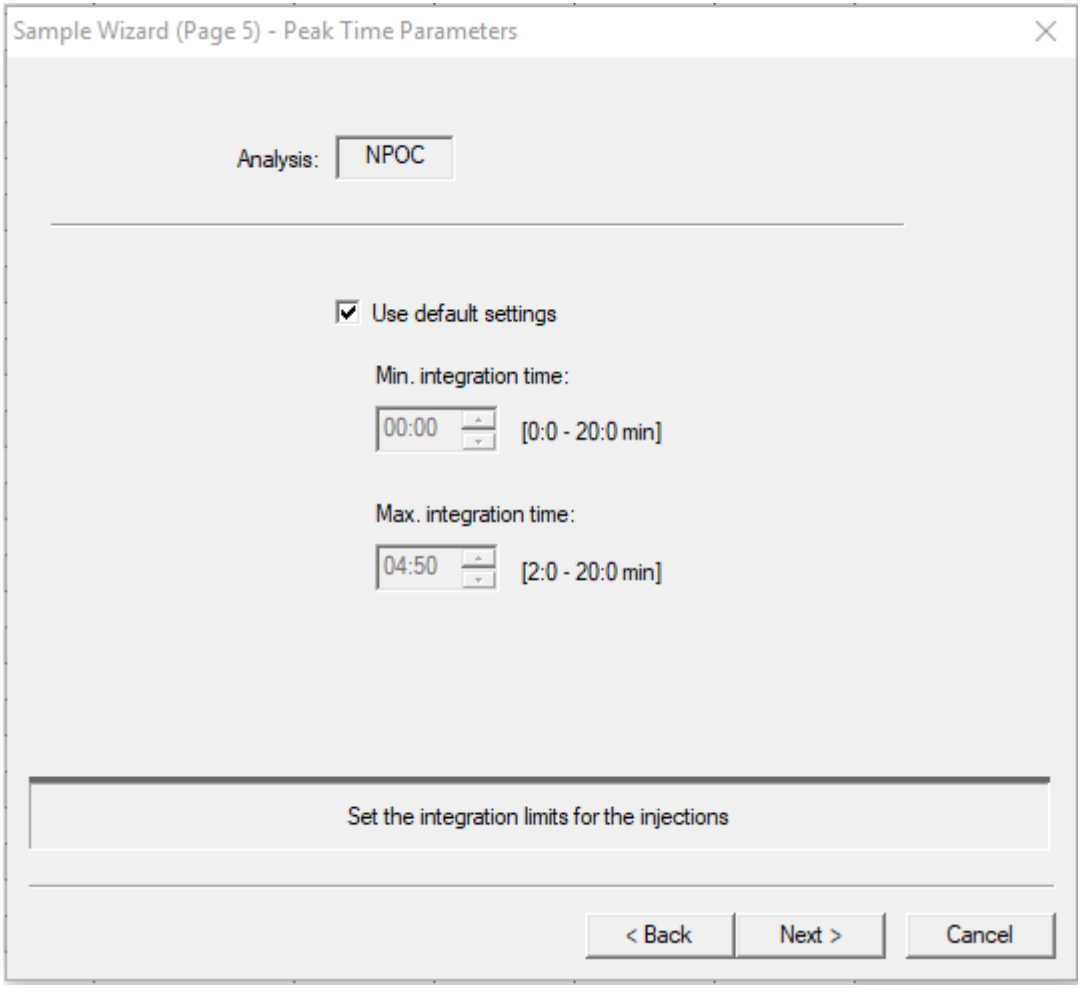


Creating a Sample Table

Confirm the Peak Time Parameters

Note: the default parameters will be OK for 99% of analysis.

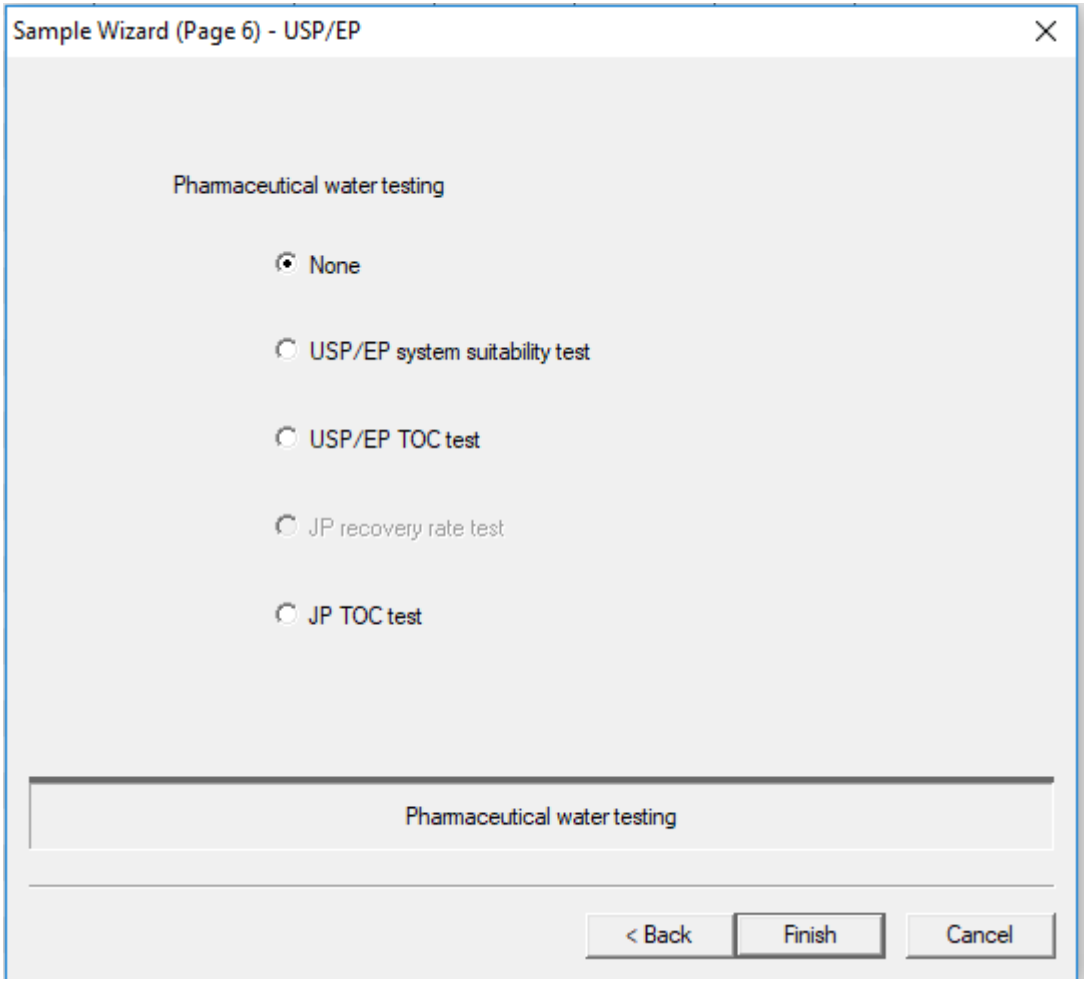
Press ‘Next’



Creating a Sample Table

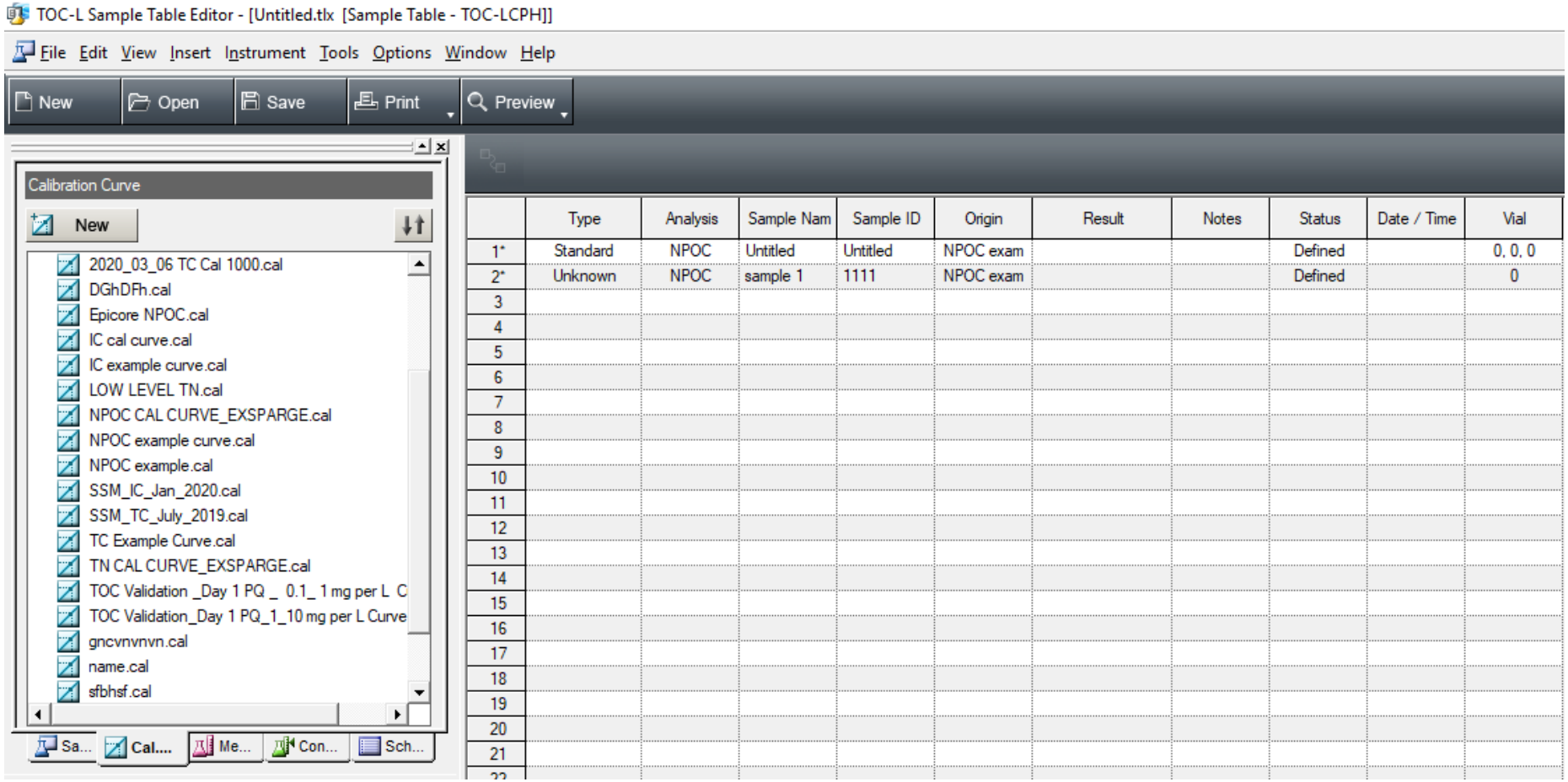
If the sample entered is following pharmaceutical water testing guidelines select the appropriate protocol to follow. For all other analysis check ‘None’

Press ‘Finish’ to save the sample to the sample table.



Creating a Sample Table

The sample should now appear in the table.



	Type	Analysis	Sample Nam	Sample ID	Origin	Result	Notes	Status	Date / Time	Vial
1*	Standard	NPOC	Untitled	Untitled	NPOC exam			Defined		0, 0, 0
2*	Unknown	NPOC	sample 1	1111	NPOC exam			Defined		0
3										
4										
5										
6										
7										
8										
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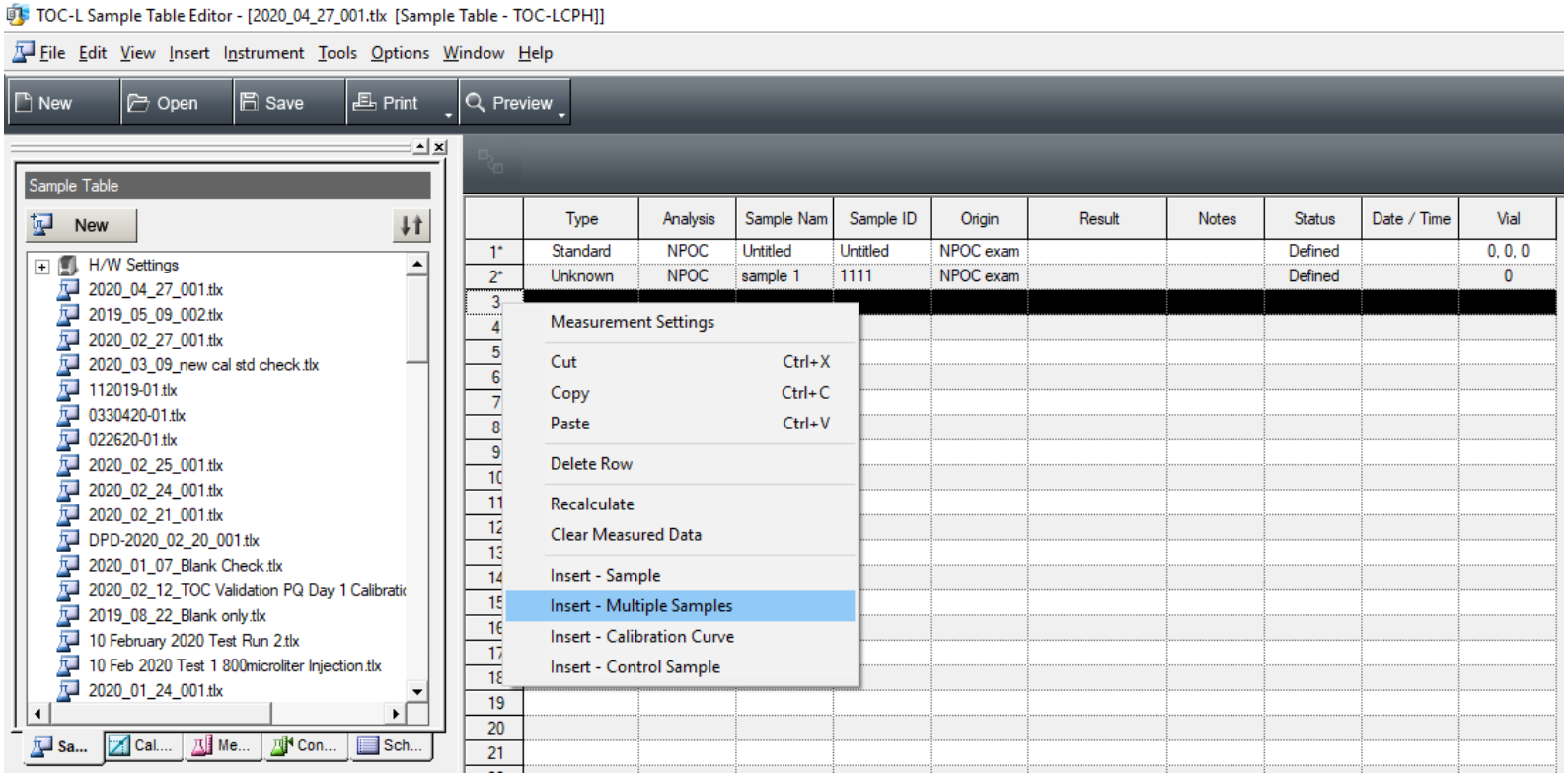
Creating a Sample Table

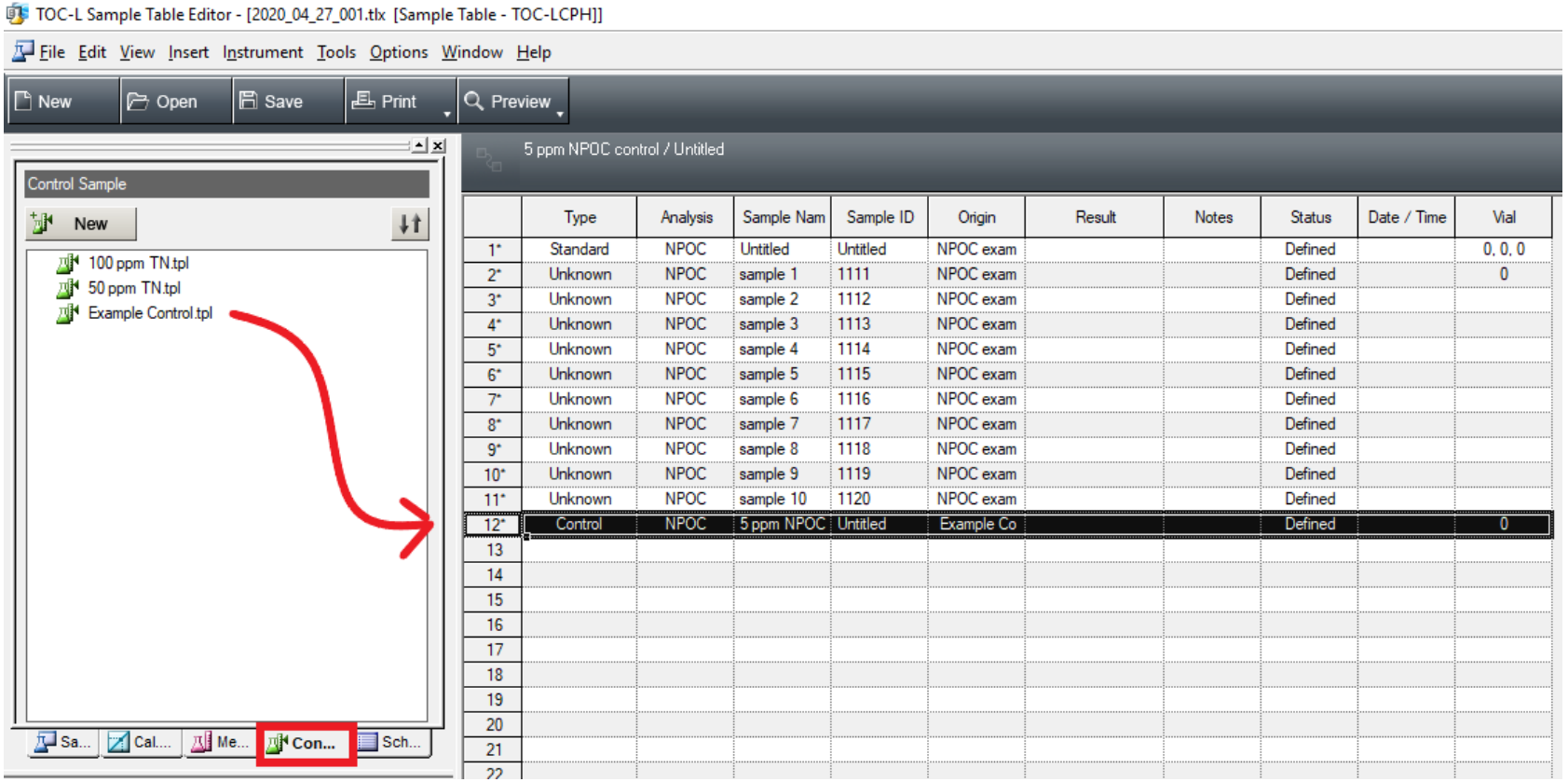
Add as many sample to the table as needed. Note that multiple samples can be added to the table at once by selecting ‘Insert-Multiple Samples’



Creating a Sample Table

Push the ‘Control Sample’ button to show the list of saved control samples.

Drag and drop the appropriate control sample into the sample table.



	Type	Analysis	Sample Nam	Sample ID	Origin	Result	Notes	Status	Date / Time	Vial
1*	Standard	NPOC	Untitled	Untitled	NPOC exam			Defined		0, 0, 0
2*	Unknown	NPOC	sample 1	1111	NPOC exam			Defined		0
3*	Unknown	NPOC	sample 2	1112	NPOC exam			Defined		
4*	Unknown	NPOC	sample 3	1113	NPOC exam			Defined		
5*	Unknown	NPOC	sample 4	1114	NPOC exam			Defined		
6*	Unknown	NPOC	sample 5	1115	NPOC exam			Defined		
7*	Unknown	NPOC	sample 6	1116	NPOC exam			Defined		
8*	Unknown	NPOC	sample 7	1117	NPOC exam			Defined		
9*	Unknown	NPOC	sample 8	1118	NPOC exam			Defined		
10*	Unknown	NPOC	sample 9	1119	NPOC exam			Defined		
11*	Unknown	NPOC	sample 10	1120	NPOC exam			Defined		
12*	Control	NPOC	5 ppm NPOC	Untitled	Example Co			Defined		0
13										
14										
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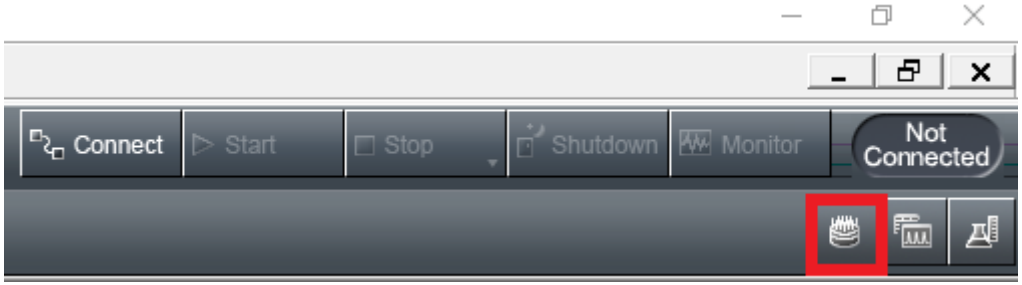
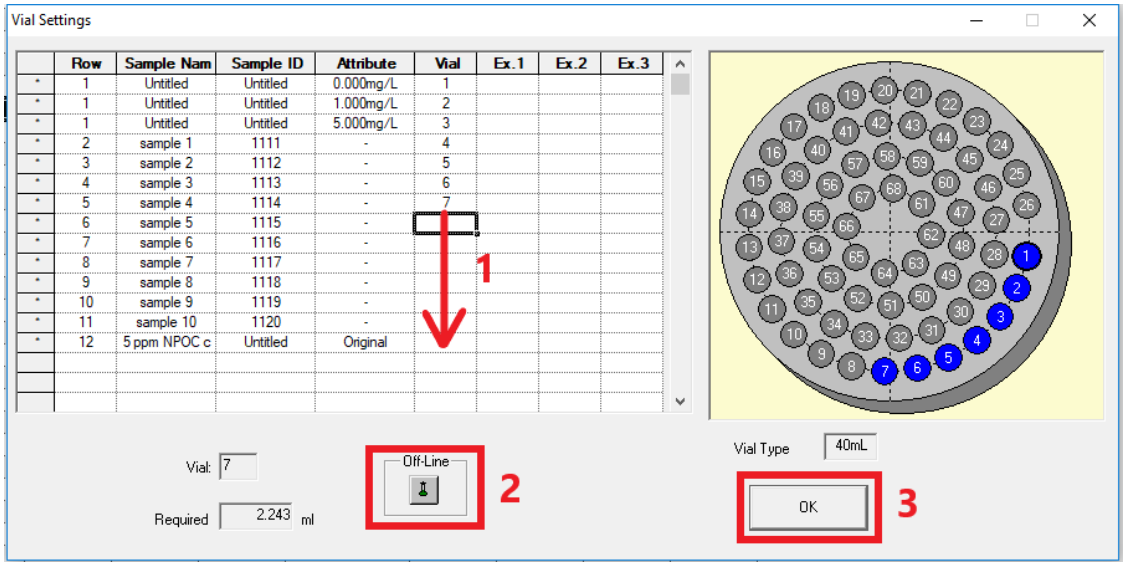
Creating a Sample Table

When finished entering all curves, samples, and control standards are into the sample table, push the “Birthday Cake” button.





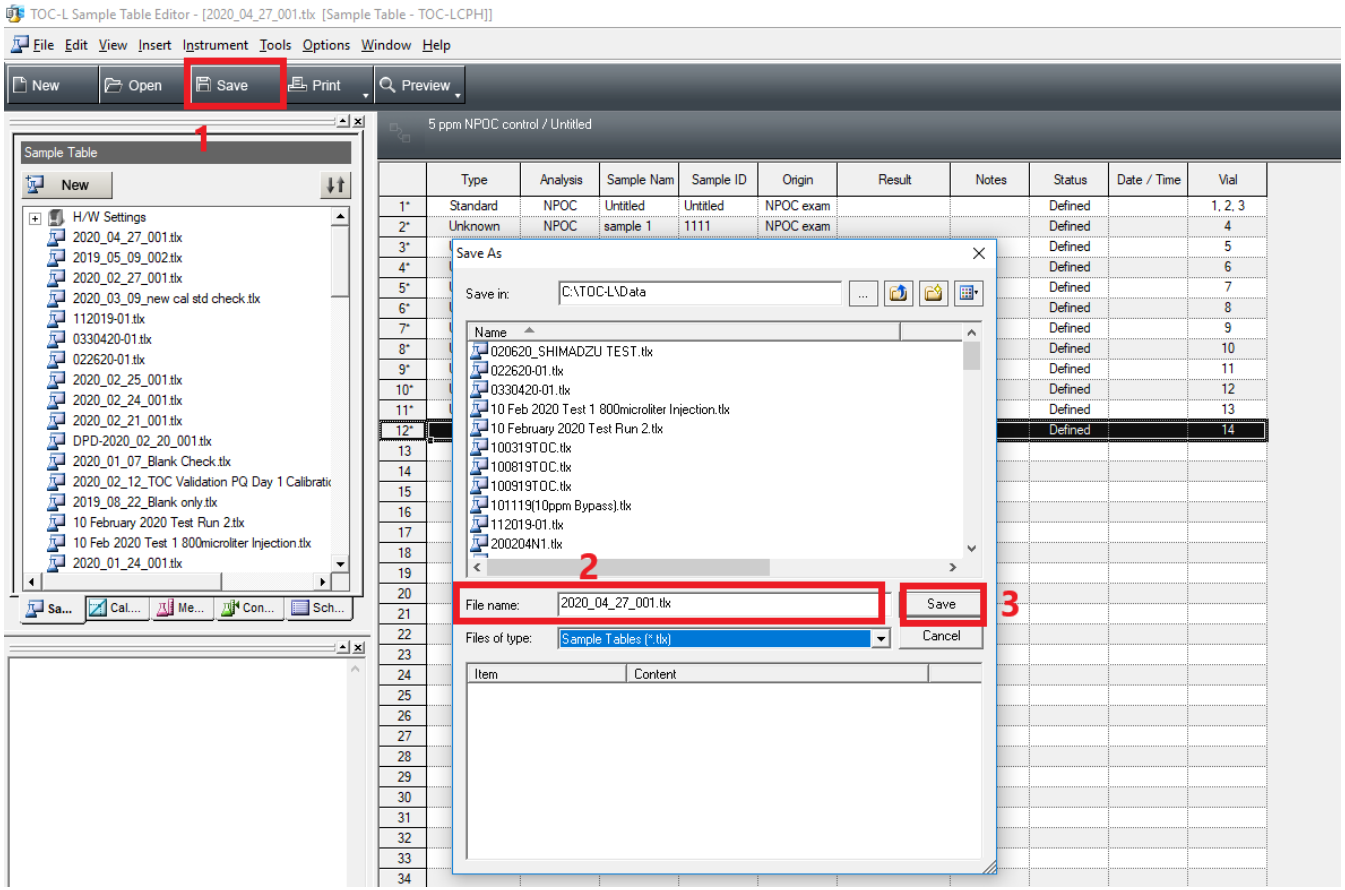
1. Enter the correct the vial position on the auto-sampler tray for each sample. You can manually enter each number or double click on the position in the diagram.
2. Use this to measure a sample ‘Off-Line’. This refers to using the port 1 sampling line for measurement instead of the auto-sampler which is connected to port 2 of the valve. Selecting ‘Off-Line’ will enter the vial position as ‘0’. This can be useful for samples like controls that may be run multiple times throughout a run.
3. Press ‘OK’

Creating a Sample Table

1. Press the ‘Save’ button to save the sample table.

2. By default the file name will be entered with date and time stamp but it can be changed to another unique name.

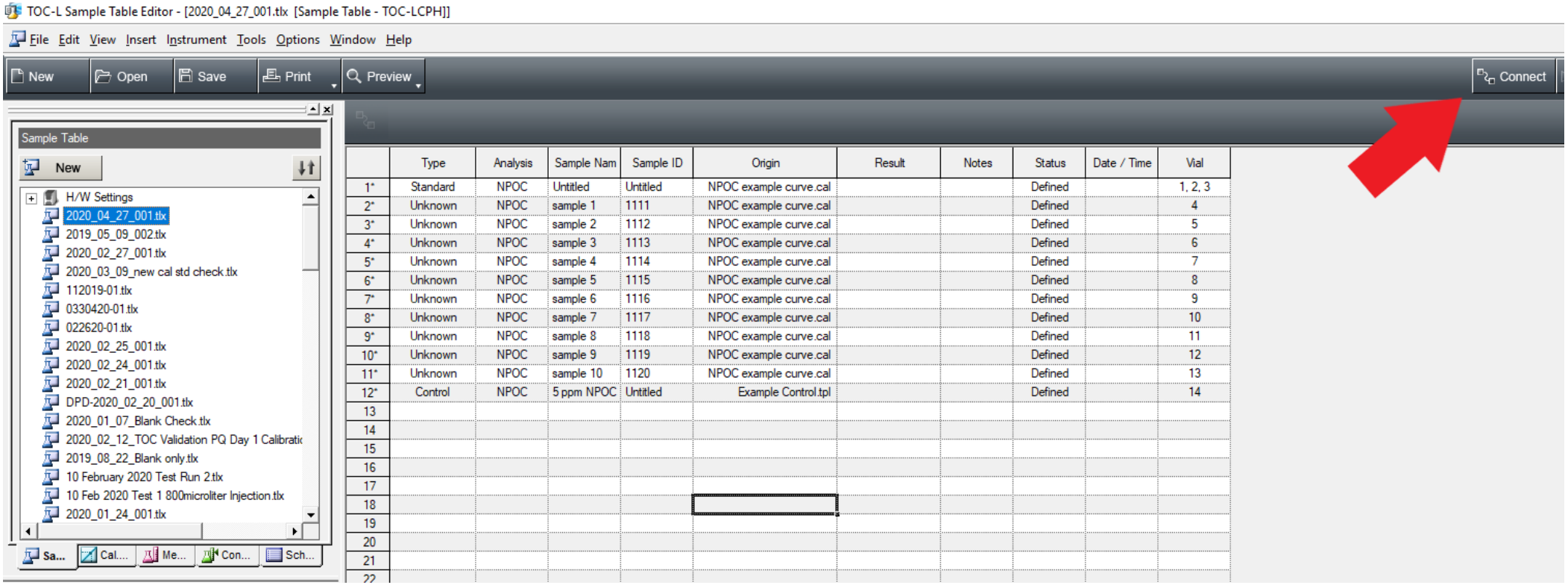
3. Press ‘Save’ to save it to the list of sample tables.



Starting a Sample Run

Open an existing sample table or create a new sample table.

Then press ‘Connect’ to establish a connection with the instrument.



	Type	Analysis	Sample Nam	Sample ID	Origin	Result	Notes	Status	Date / Time	Vial
1*	Standard	NPOC	Untitled	Untitled	NPOC example curve.cal			Defined		1, 2, 3
2*	Unknown	NPOC	sample 1	1111	NPOC example curve.cal			Defined		4
3*	Unknown	NPOC	sample 2	1112	NPOC example curve.cal			Defined		5
4*	Unknown	NPOC	sample 3	1113	NPOC example curve.cal			Defined		6
5*	Unknown	NPOC	sample 4	1114	NPOC example curve.cal			Defined		7
6*	Unknown	NPOC	sample 5	1115	NPOC example curve.cal			Defined		8
7*	Unknown	NPOC	sample 6	1116	NPOC example curve.cal			Defined		9
8*	Unknown	NPOC	sample 7	1117	NPOC example curve.cal			Defined		10
9*	Unknown	NPOC	sample 8	1118	NPOC example curve.cal			Defined		11
10*	Unknown	NPOC	sample 9	1119	NPOC example curve.cal			Defined		12
11*	Unknown	NPOC	sample 10	1120	NPOC example curve.cal			Defined		13
12*	Control	NPOC	5 ppm NPOC	Untitled	Example Control.tpl			Defined		14
13										
14										
15										
16										
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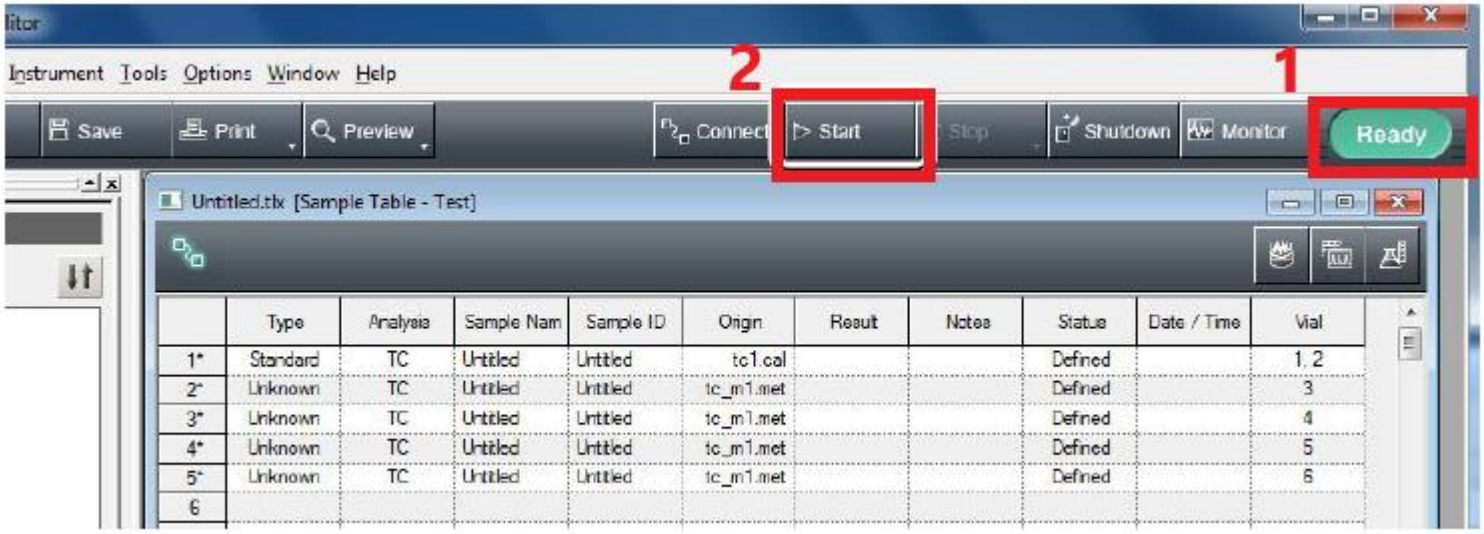
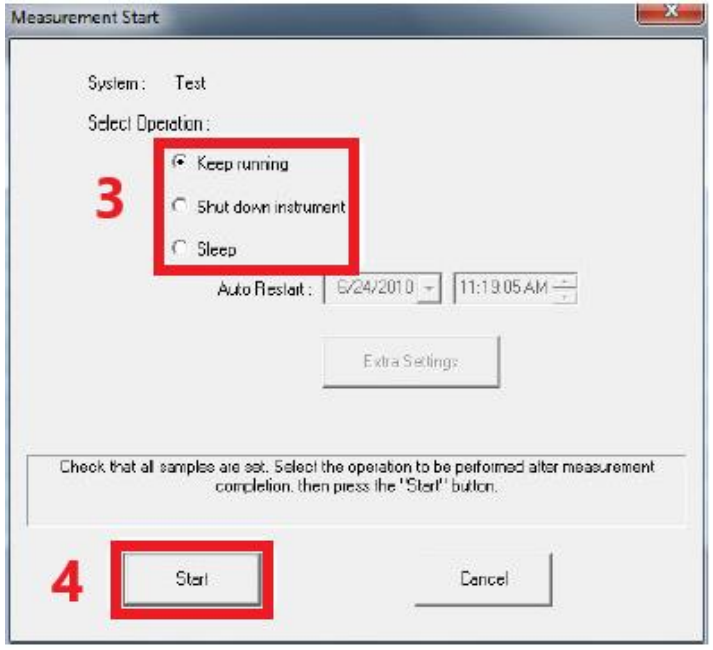
Section 1 / 3

Starting a Sample Run

1. Wait until the green ‘Ready’ status is displayed in the top right corner.

2. Press ‘Start’. If you are using the ASI auto-sampler the window below will appear.





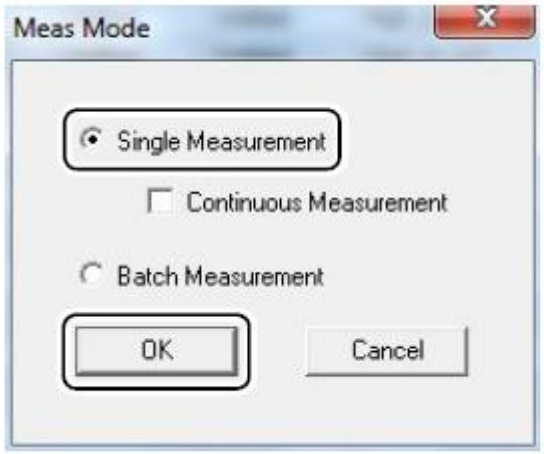
3. Select the action to be performed upon completion of the sample table. ‘Sleep’ function allows the user to put the instrument into a sleep mode. A date and time can then be entered to wake the instrument.

4. Push ‘Start’

Section 2 / 3

Starting a Sample Run

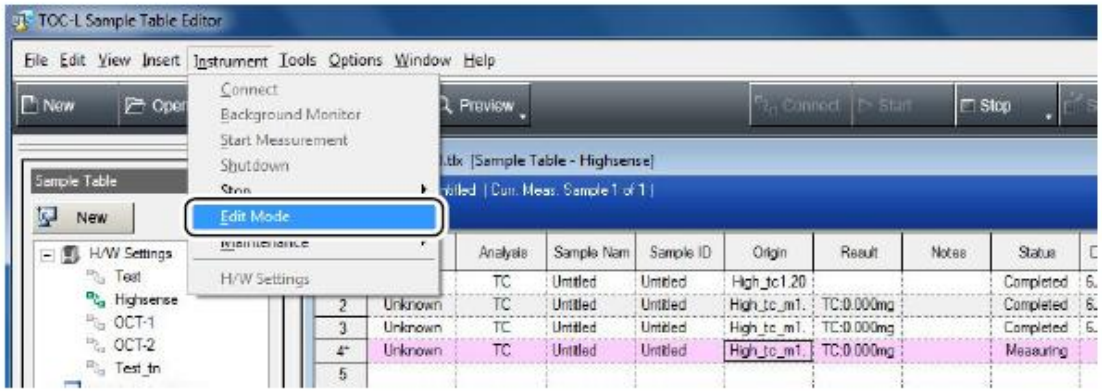
When NOT using the ASI Auto-sampler the window below will appear when ‘Start’ is pushed.



Item	Description
Single Measurement	Measurement stops after a single sample measurement and the measurement results are displayed. If the analysis type is a compound type, such as TOC (which is a combination of TC and IC), measurement stops after each type of analysis and the measurement results are displayed. If the Continuous Measurement checkbox is selected, measurement continues regardless of analysis type when processing compound types, such as TOC (which is a combination of TC and IC).
Batch Measurement	All samples in the sample table are measured without stopping.

Editing a Sample Table

1. During analysis, select Instrument – Edit Mode.



The sample table is placed in the Edit Mode.
In the Edit Mode, the indicator at the right end of the tool bar changes to read "Edit."
This mode allows the following operations to be conducted in the sample table in the same way as when analysis is not being conducted.

- Adding and deleting samples
- Changing analysis parameters and vial numbers
- Printing reports for samples that are already analyzed

2. Click 

to cancel the Edit Mode and restart analysis.
The restart analysis confirmation window is displayed.

The Edit Mode can also be cancelled by selecting Instrument – Edit Mode.

3. Click OK.
The Edit Mode is cancelled, and analysis continues.

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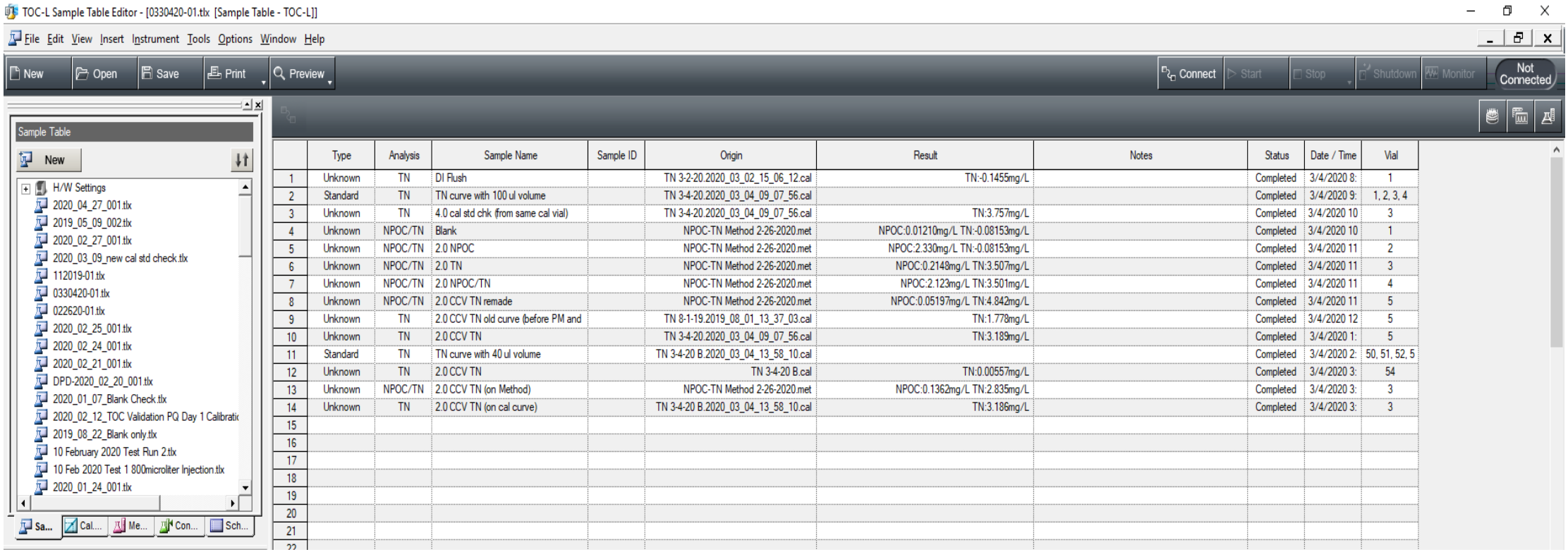
Viewing Sample Results



	Type	Analysis	Sample Name	Sample ID	Origin	Result	Notes	Status	Date / Time	Vial
1	Unknown	TN	DI Flush		TN 3-2-20.2020_03_02_15_06_12.cal	TN:-0.1455mg/L		Completed	3/4/2020 8:	1
2	Standard	TN	TN curve with 100 ul volume		TN 3-4-20.2020_03_04_09_07_56.cal			Completed	3/4/2020 9:	1, 2, 3, 4
3	Unknown	TN	4.0 cal std chk (from same cal vial)		TN 3-4-20.2020_03_04_09_07_56.cal	TN:3.757mg/L		Completed	3/4/2020 10	3
4	Unknown	NPOC/TN	Blank		NPOC-TN Method 2-26-2020.met	NPOC:0.01210mg/L TN:-0.08153mg/L		Completed	3/4/2020 10	1
5	Unknown	NPOC/TN	2.0 NPOC		NPOC-TN Method 2-26-2020.met	NPOC:2.330mg/L TN:-0.08153mg/L		Completed	3/4/2020 11	2
6	Unknown	NPOC/TN	2.0 TN		NPOC-TN Method 2-26-2020.met	NPOC:0.2148mg/L TN:3.507mg/L		Completed	3/4/2020 11	3
7	Unknown	NPOC/TN	2.0 NPOC/TN		NPOC-TN Method 2-26-2020.met	NPOC:2.123mg/L TN:3.501mg/L		Completed	3/4/2020 11	4
8	Unknown	NPOC/TN	2.0 CCV TN remade		NPOC-TN Method 2-26-2020.met	NPOC:0.05197mg/L TN:4.842mg/L		Completed	3/4/2020 11	5
9	Unknown	TN	2.0 CCV TN old curve (before PM and		TN 8-1-19.2019_08_01_13_37_03.cal	TN:1.778mg/L		Completed	3/4/2020 12	5
10	Unknown	TN	2.0 CCV TN		TN 3-4-20.2020_03_04_09_07_56.cal	TN:3.189mg/L		Completed	3/4/2020 1:	5
11	Standard	TN	TN curve with 40 ul volume		TN 3-4-20 B.2020_03_04_13_58_10.cal			Completed	3/4/2020 2:	50, 51, 52, 5
12	Unknown	TN	2.0 CCV TN		TN 3-4-20 B.cal	TN:0.00557mg/L		Completed	3/4/2020 3:	54
13	Unknown	NPOC/TN	2.0 CCV TN (on Method)		NPOC-TN Method 2-26-2020.met	NPOC:0.1362mg/L TN:2.835mg/L		Completed	3/4/2020 3:	3
14	Unknown	TN	2.0 CCV TN (on cal curve)		TN 3-4-20 B.2020_03_04_13_58_10.cal	TN:3.186mg/L		Completed	3/4/2020 3:	3
15										
16										
17										
18										
19										
20										
21										

Sample results can be found in the ‘Result’ column of the sample table. Note that samples that are using a Method with multiple analysis types show results for all measurements in the ‘Result’ column.

To view more detailed information for each sample push 

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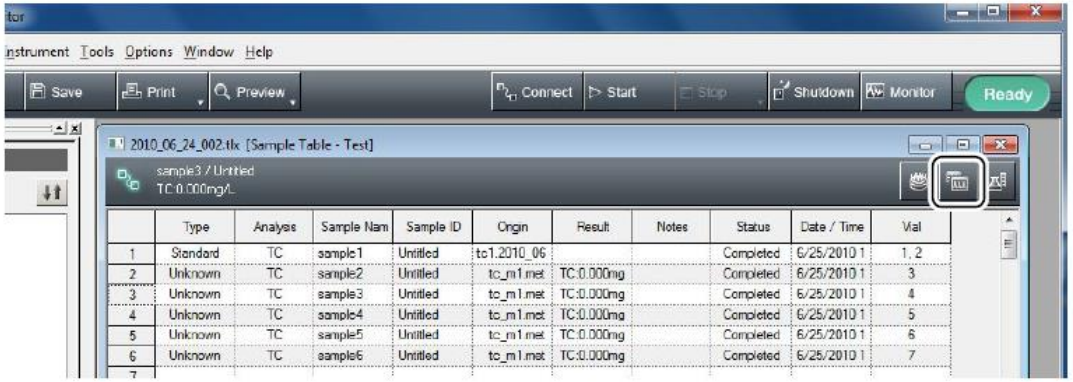
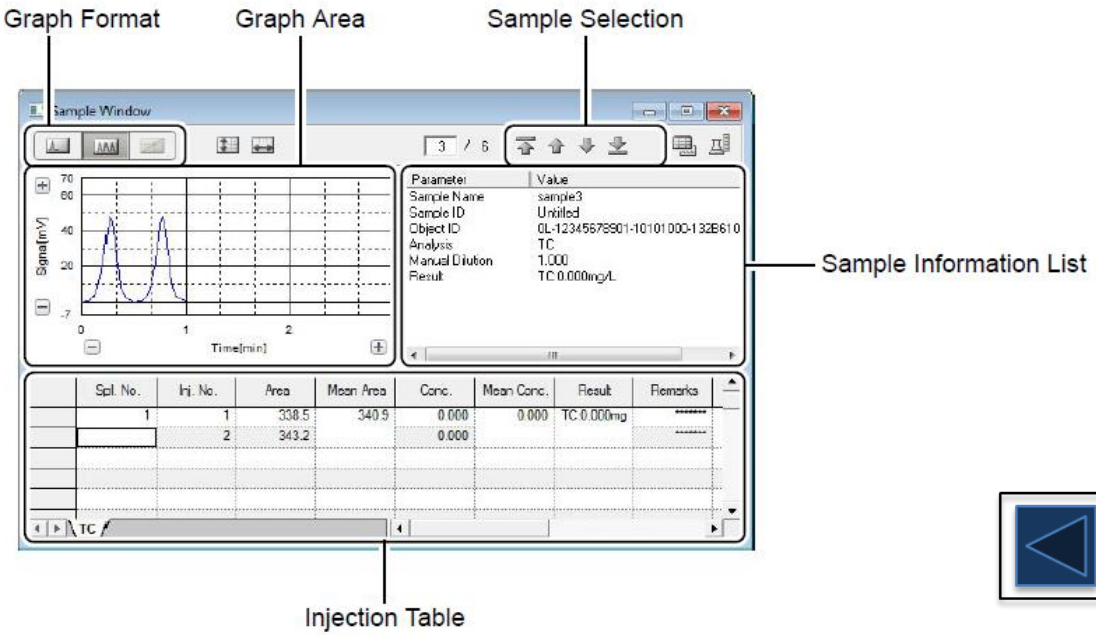
Viewing Sample Results

Click 

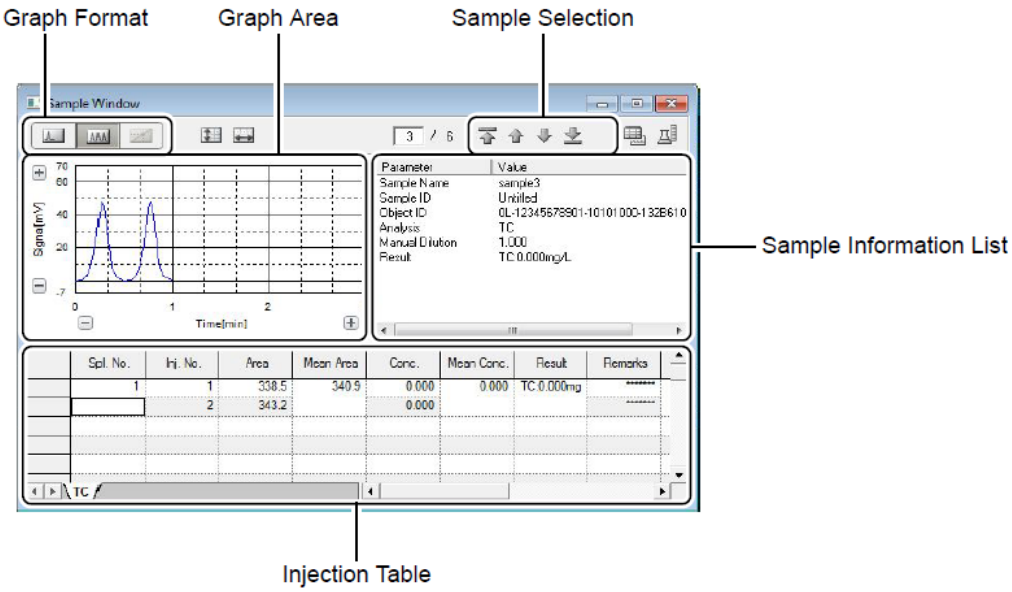


The Sample Window is displayed.

The Sample Window can also be displayed by selecting View – Sample Window.



Viewing Sample Results



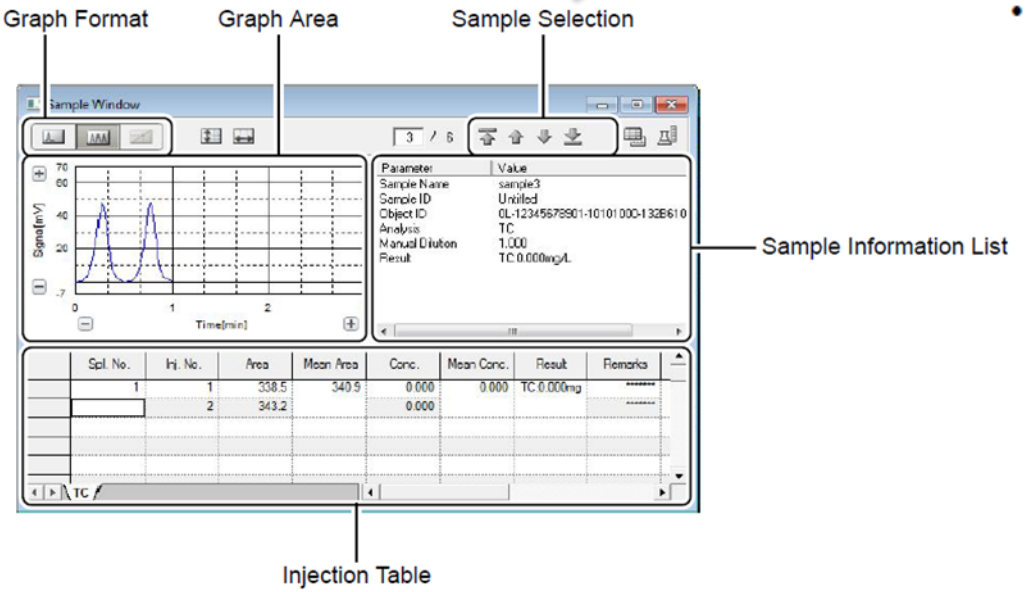
Set the graph format to Current Peak, All Peaks, or Cal. Curve.

- 
  Displays the peak profile of the selected injection in the table.
- 
  Displays all the injection peaks in the table.
- 
  Displays the calibration curve peak. This is not enabled during analysis.

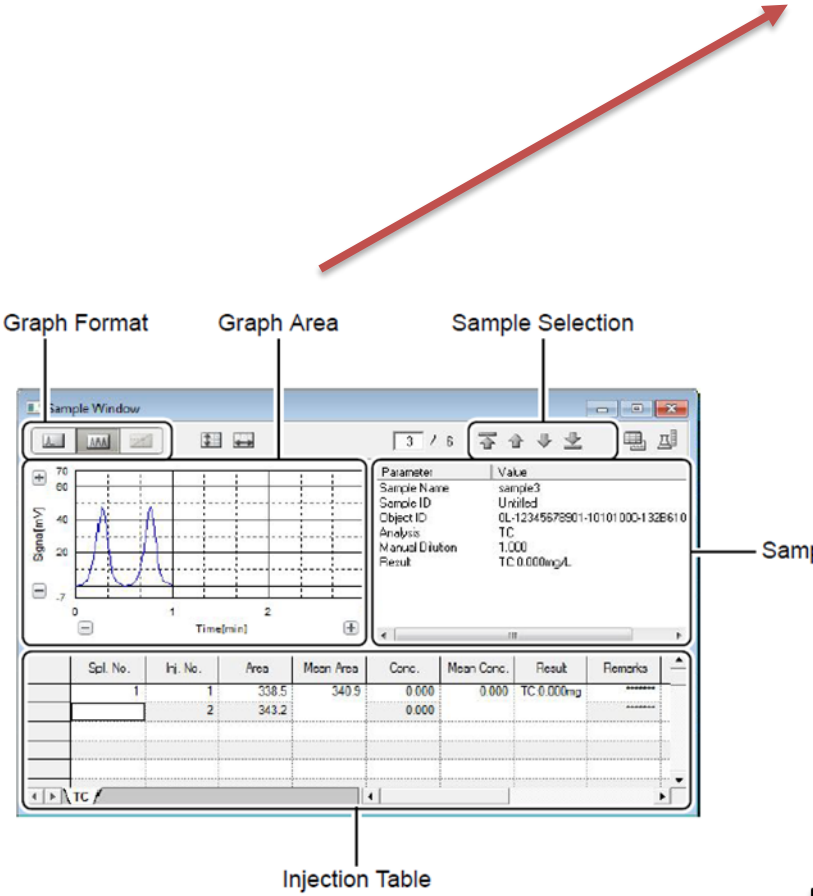
Viewing Sample Results

Select the sample (row) for display from the sample table.

- Sample number/Row number: Select a sample to display.
- : Display the first sample.
- : Display the sample above the current one.
- : Display the sample below the current one.
- : Display the last sample.



Viewing Sample Results



The graph display can be manipulated using the following procedures.

Enlarging or Reducing the Graph Display

The size of the graph display can be enlarged or reduced.

Operating Procedure

Using the ⊕/⊖ buttons:

Click the ⊕ and ⊖ buttons on the vertical or horizontal axes on the graph.

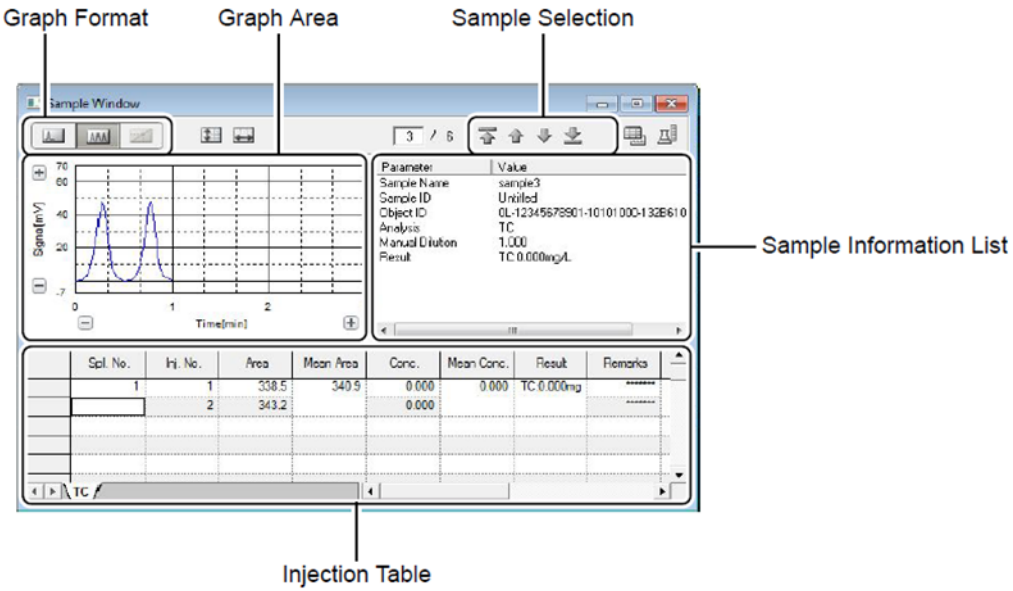
Each click either enlarges or reduces the size of the corresponding axis display.

Using the ↕/↔ buttons:

Click ↕ or ↔ and then select and click the full scale for modification.

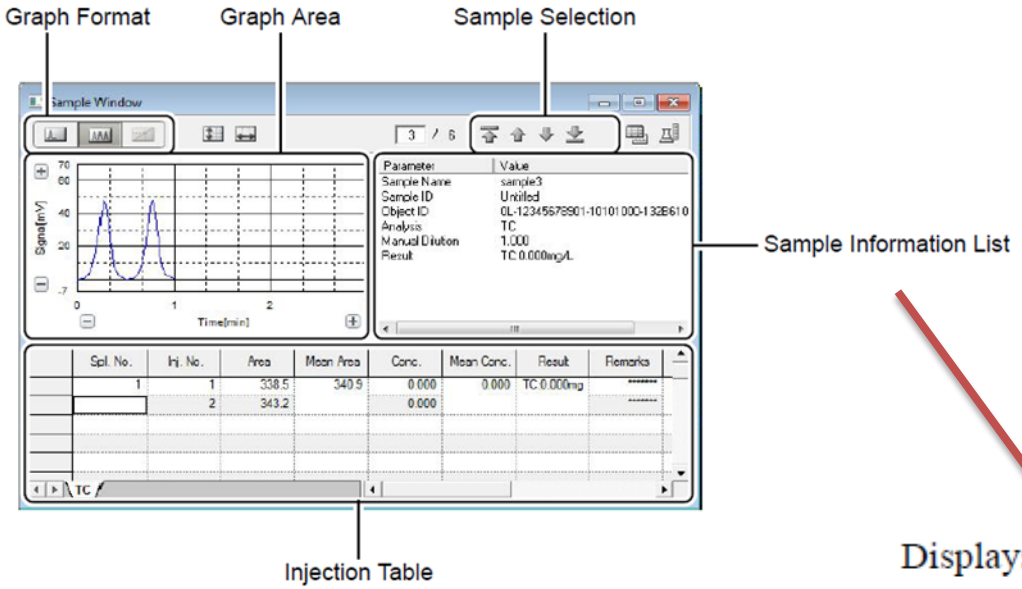
The full scale of the corresponding axis becomes the selected value.

Viewing Sample Results



If the sample was analyzed by more than one analysis method, several tabs are displayed at the bottom of the Injection Table window. Click a tab to display the respective injection data. Example: For analysis type TOC, both the TC and IC tabs are displayed at the bottom of the injection table.

Viewing Sample Results



Displays the injection information. Click on a cell to display the peak profile for that sample in the graph area.

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Viewing Calibration Curves

The TOC-L software will create two different types of calibration curve files.

1. Calibration curve template files. (calibration curve.cal)
   - These files contain the calibration curve parameters
   - They DO NOT contain any data
   - Are NOT saved with a date and time stamp.

2. Calibration curve data files. (calibration curve.cal.date.time)
   - These files contain the calibration curve parameters
   - They DO contain data and resultant curves
   - When a calibration curve template file is inserted into a sample table and ran, the software will save a calibration curve file with a date and time stamp from when the curve was created.

It is important to understand this difference as it is designed as a simple way to ensure that the most current calibration curve data file is being used to calculate results. The calibration curve template file can be used to run samples or can be entered into a method file. When the instrument runs the samples, the software will look for a calibration curve data file of the same name and use the most current one to calculate results.

Viewing Calibration Curves

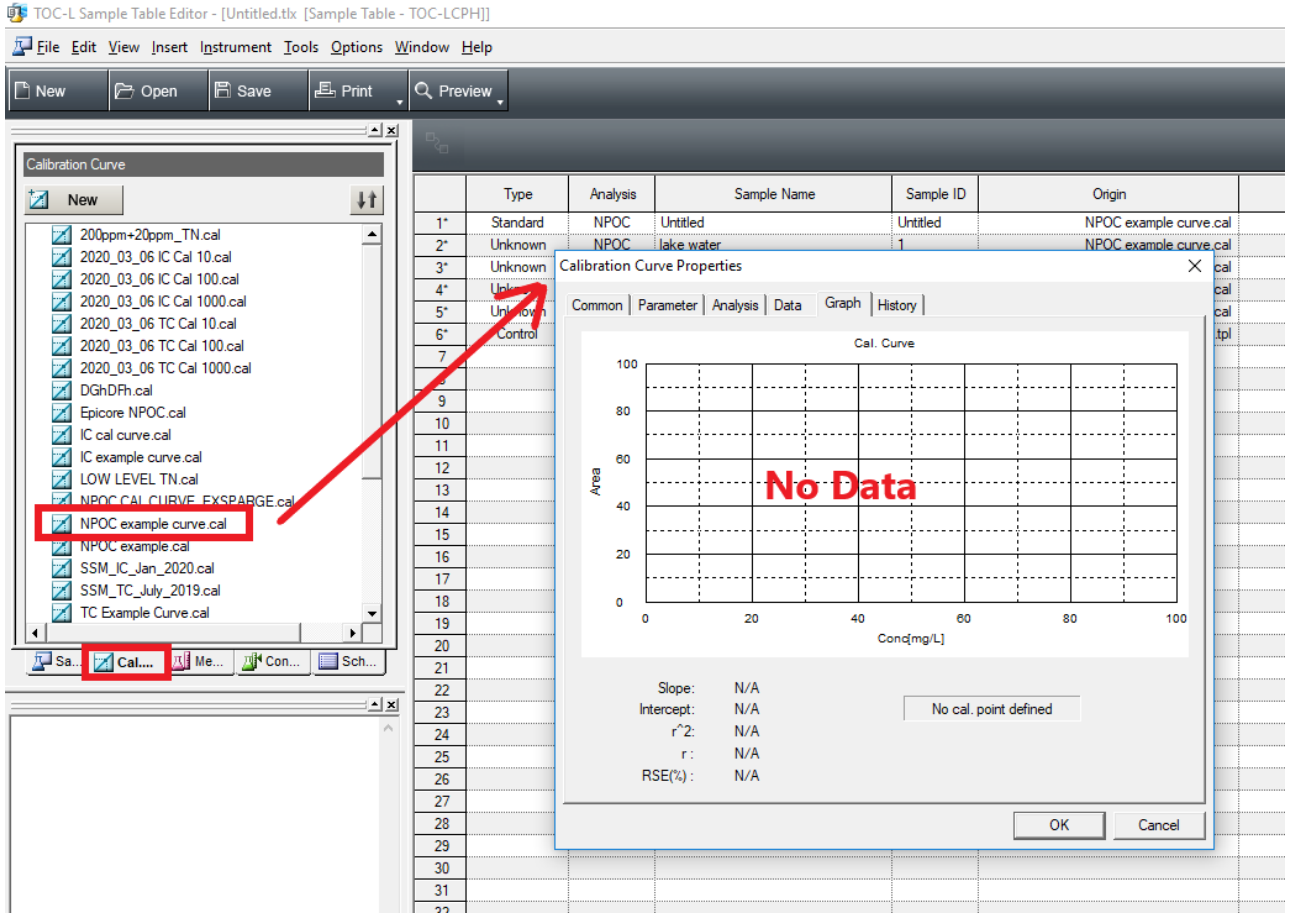
To view a calibration curve template file:

1. Press the ‘Cal….’ button to show the list of saved curves.

2. Double click the mouse on the desired curve to open the ‘Calibration Curve Properties Window’

3. Use the tabs across the top of the window to view the calibration curve parameters.

NOTE: there is no data saved in this file.



Viewing Calibration Curves

To open a calibration curve data file:

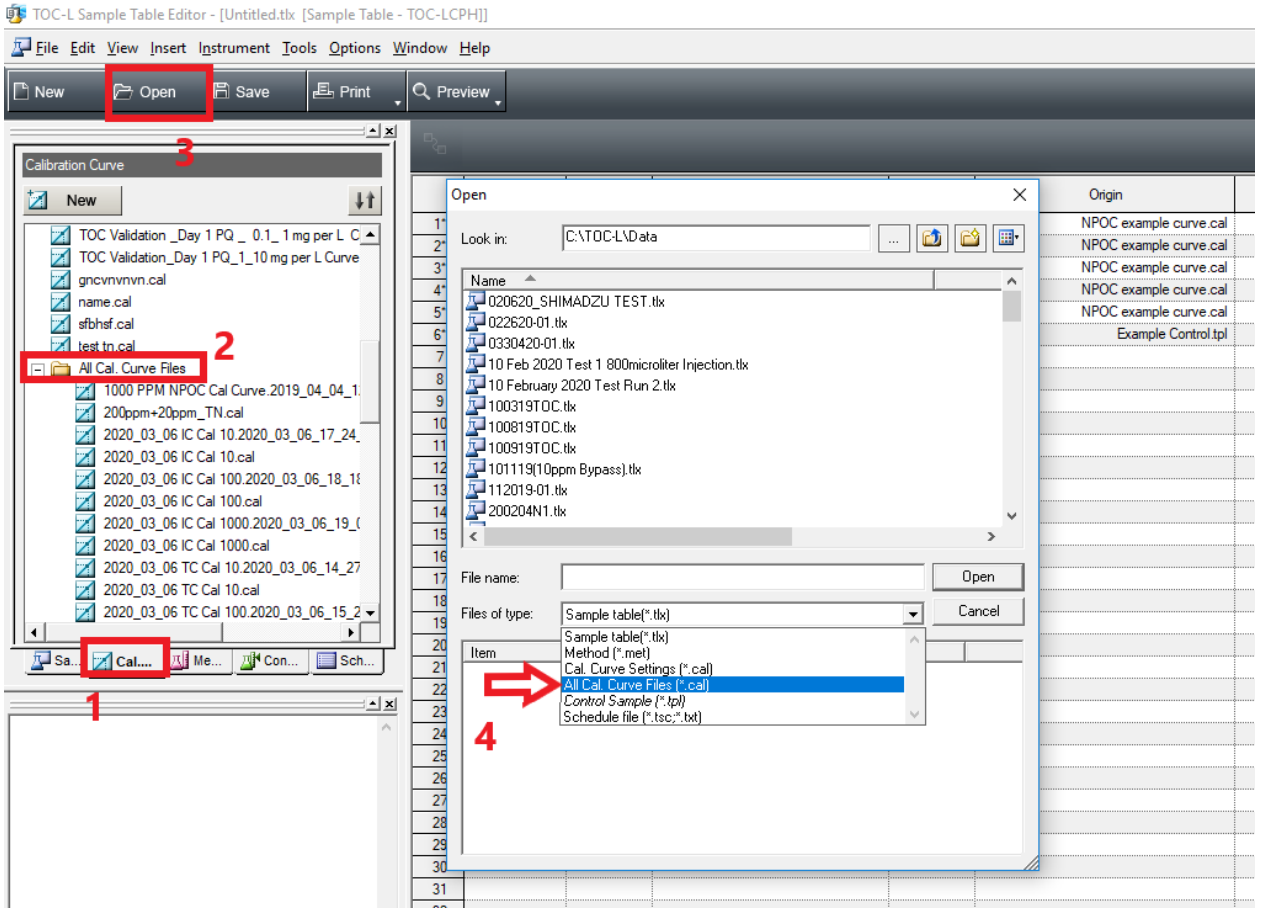
1. Press the ‘Cal…’ button to show the list of saved calibration curves.

2. Scroll to the bottom of the list and use the ‘+’ button to expand the list to see all files.

OR

3. Push ‘Open’

4. Use the drop down tab for ‘Files of Type’ to select ‘All Cal. Curve Files’

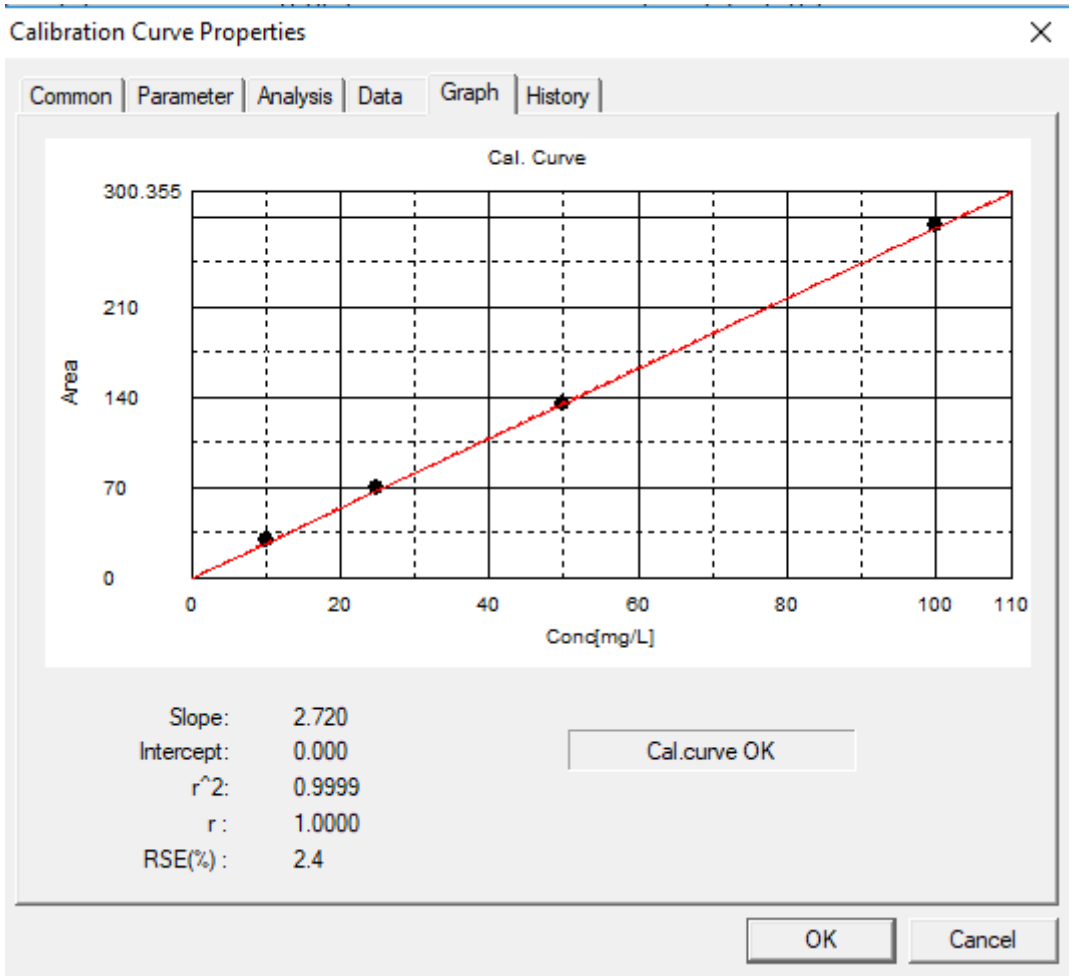


Viewing Calibration Curves

The calibration curve properties will now be opened.

Use the tabs across the top to view the curve parameters.

NOTE: there will be data in these files that can be viewed.



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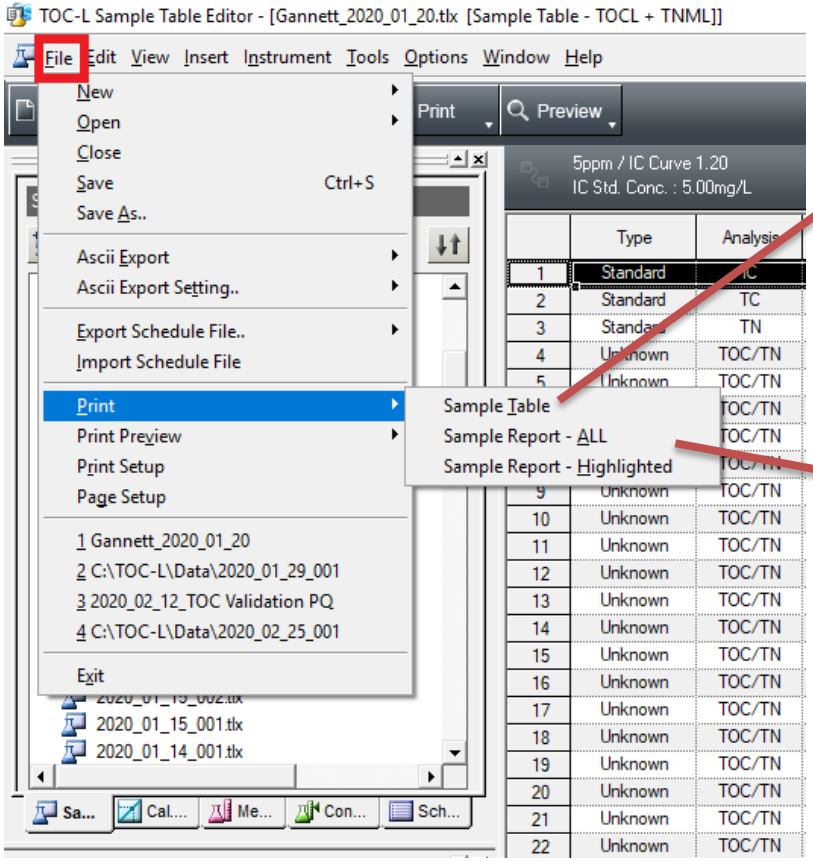
Section 4 / 4

Creating a Sample Report

There are two different ways to print sample reports.

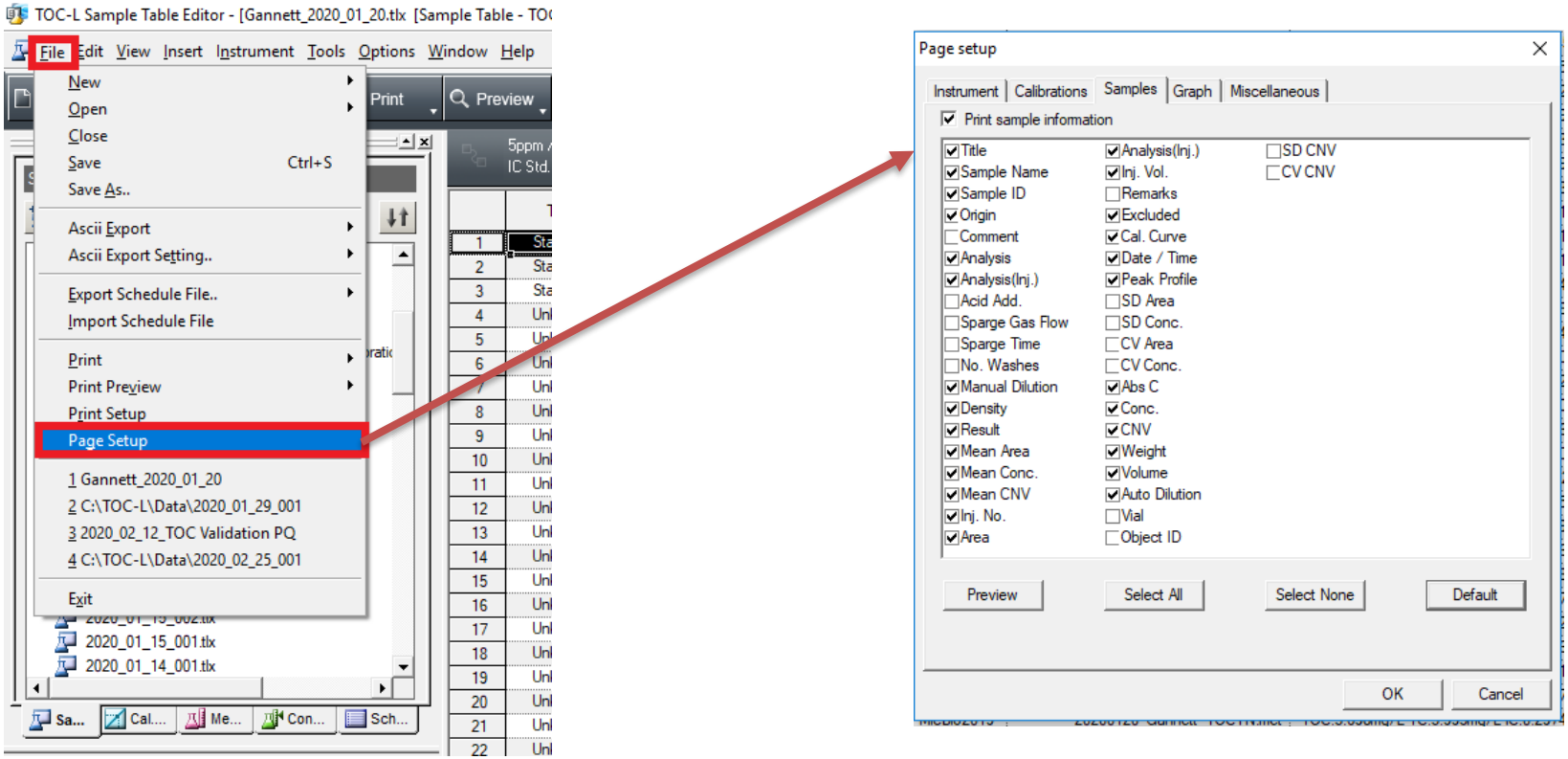


The sample table can be printed. It will appear in the report exactly as it does on the screen in the software.

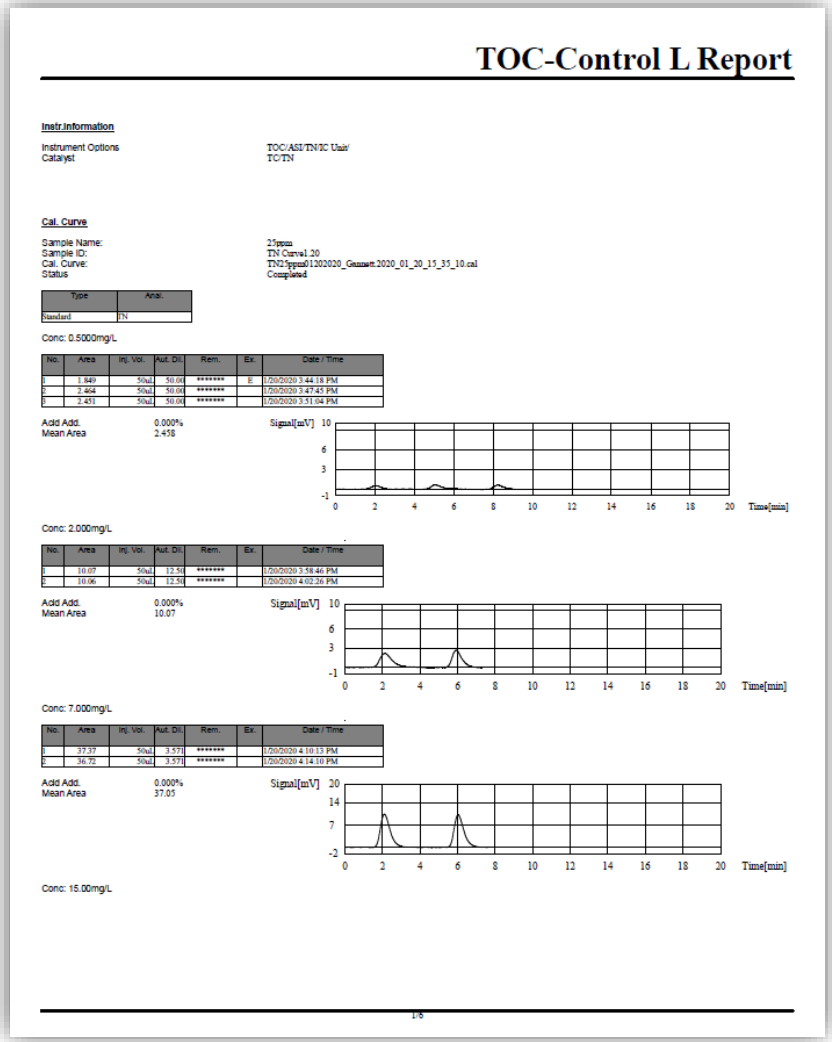
A customized report can be created and printed. The entire report can be printed or certain samples in the table can be highlighted to only print those samples.

Creating a Sample Report

To customize a report select ‘File’ then ‘Page Setup’. In the page setup window select the items to be displayed in the report.



Creating a Sample Report



TOC-Control L Report

Instr.information

Instrument Options: TOC/ASI/TN/IC Unit/
Catalyst: TC/TN

Cal. Curve

Sample Name: 2?ppm
Sample ID: TN Curve1.20
Cal. Curve: TN2?ppm01202020_Gunnett.2020_01_20_15_35_10.cal
Status: Completed

Type	Anal.
Standard	TN

Conc: 0.5000mg/L

No.	Area	Inj. Vol.	Aut. Dil.	Rem.	Ex.	Date / Time
1	1.849	50uL	50.00	*******	E	1/20/2020 3:44:18 PM
2	2.464	50uL	50.00	*******		1/20/2020 3:47:45 PM
3	2.451	50uL	50.00	*******		1/20/2020 3:51:04 PM

Add Add. 0.000%
Mean Area 2.458

Conc: 2.000mg/L

No.	Area	Inj. Vol.	Aut. Dil.	Rem.	Ex.	Date / Time
1	10.07	50uL	12.50	*******		1/20/2020 3:58:46 PM
2	10.06	50uL	12.50	*******		1/20/2020 4:02:26 PM

Add Add. 0.000%
Mean Area 10.07

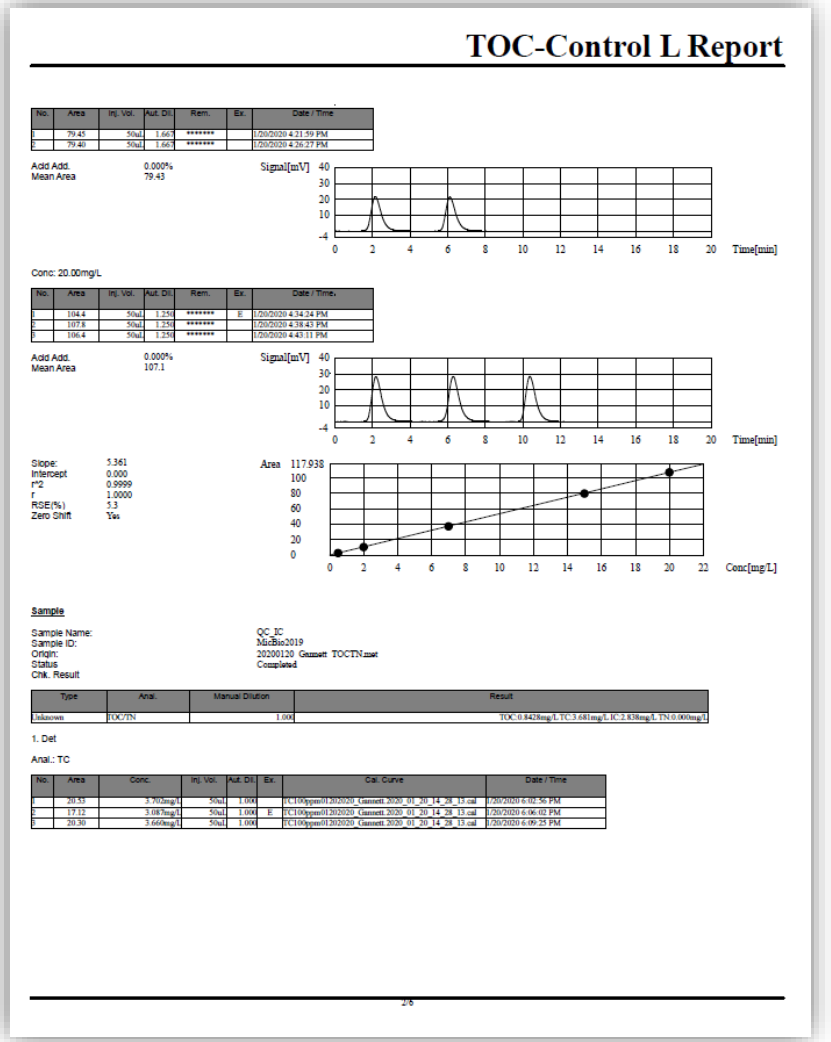
Conc: 7.000mg/L

No.	Area	Inj. Vol.	Aut. Dil.	Rem.	Ex.	Date / Time
1	37.37	50uL	3.571	*******		1/20/2020 4:10:13 PM
2	36.72	50uL	3.571	*******		1/20/2020 4:14:10 PM

Add Add. 0.000%
Mean Area 37.05

Conc: 15.00mg/L

1/6



TOC-Control L Report

No.	Area	Inj. Vol.	Aut. Dil.	Rem.	Ex.	Date / Time
1	79.45	50uL	1.667	*******		1/20/2020 4:21:59 PM
2	79.40	50uL	1.667	*******		1/20/2020 4:26:27 PM

Add Add. 0.000%
Mean Area 79.43

Conc: 20.00mg/L

No.	Area	Inj. Vol.	Aut. Dil.	Rem.	Ex.	Date / Time
1	104.4	50uL	1.250	*******	E	1/20/2020 4:34:24 PM
2	107.8	50uL	1.250	*******		1/20/2020 4:38:43 PM
3	106.4	50uL	1.250	*******		1/20/2020 4:43:11 PM

Add Add. 0.000%
Mean Area 107.1

Slope: 5.361
Intercept 0.000
r^2 0.9999
r 1.0000
RSE(%) 5.3
Zero Shift Yes

Sample

Sample Name: QC_IC
Sample ID: MicBio2019
Origin: 20200120_Gunnett_TOCTN.mat
Status: Completed
Chk. Result:

Type	Anal.	Manual Dilution	Result
Unknown	TOC/TN	1.000	TOC:0.8428mg/L TC:3.681mg/L IC:2.838mg/L TN:0.000mg/L

1. Det

Anal.: TC

No.	Area	Conc.	Inj. Vol.	Aut. Dil.	Ex.	Cal. Curve	Date / Time
1	20.53	3.702mg/L	50uL	1.000		TC100ppm01202020_Gunnett.2020_01_20_14_28_13.cal	1/20/2020 6:02:56 PM
2	17.12	3.087mg/L	50uL	1.000	E	TC100ppm01202020_Gunnett.2020_01_20_14_28_13.cal	1/20/2020 6:06:02 PM
3	20.30	3.660mg/L	50uL	1.000		TC100ppm01202020_Gunnett.2020_01_20_14_28_13.cal	1/20/2020 6:09:25 PM

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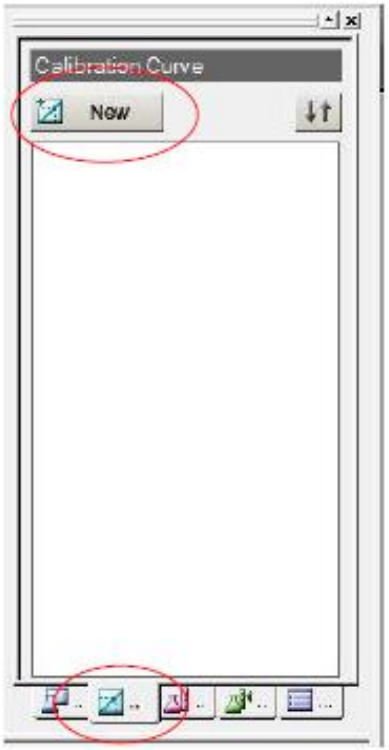
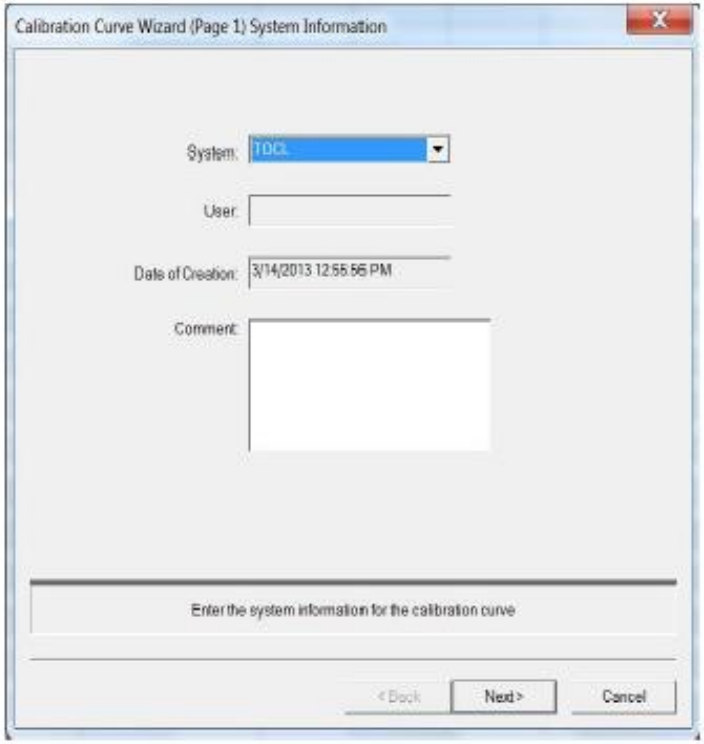
Section 3 / 3

Solid Sample Module Analysis

The SSM-5000A is an accessory module used to measure samples that are not well suited for the TOC such as solids or samples that contain % levels of carbon. The SSM-5000A requires high purity O2 to operate. Set the instrument pressure to 200kPa and the flow meter to 0.5 L/min and ensure that the drain pot is filled with water.

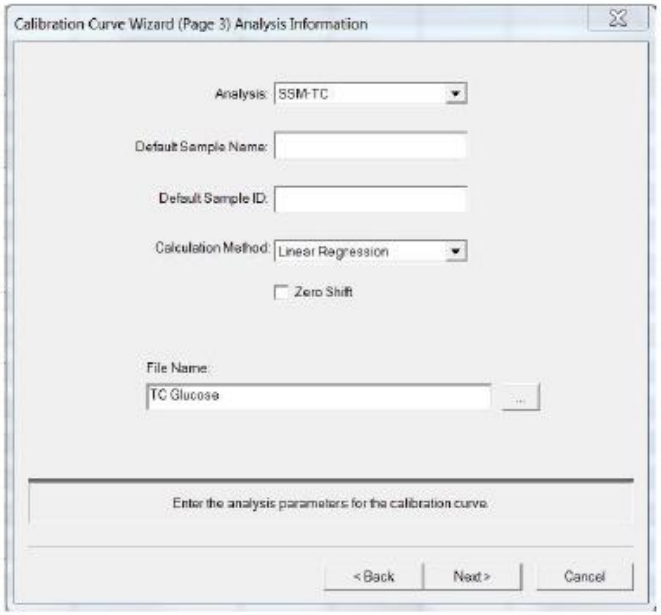
1. From the File View window select the "New" button to open the Calibration Curve Wizard. Select the system and enter any comments as needed. Click Next to continue.



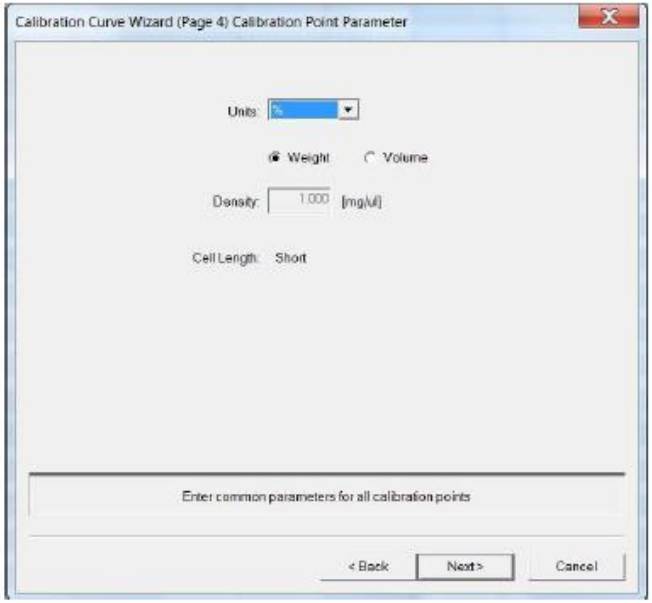


Solid Sample Module Analysis

2. Select the analysis type, choose a sample name and enter an appropriate file name. Click Next to continue.

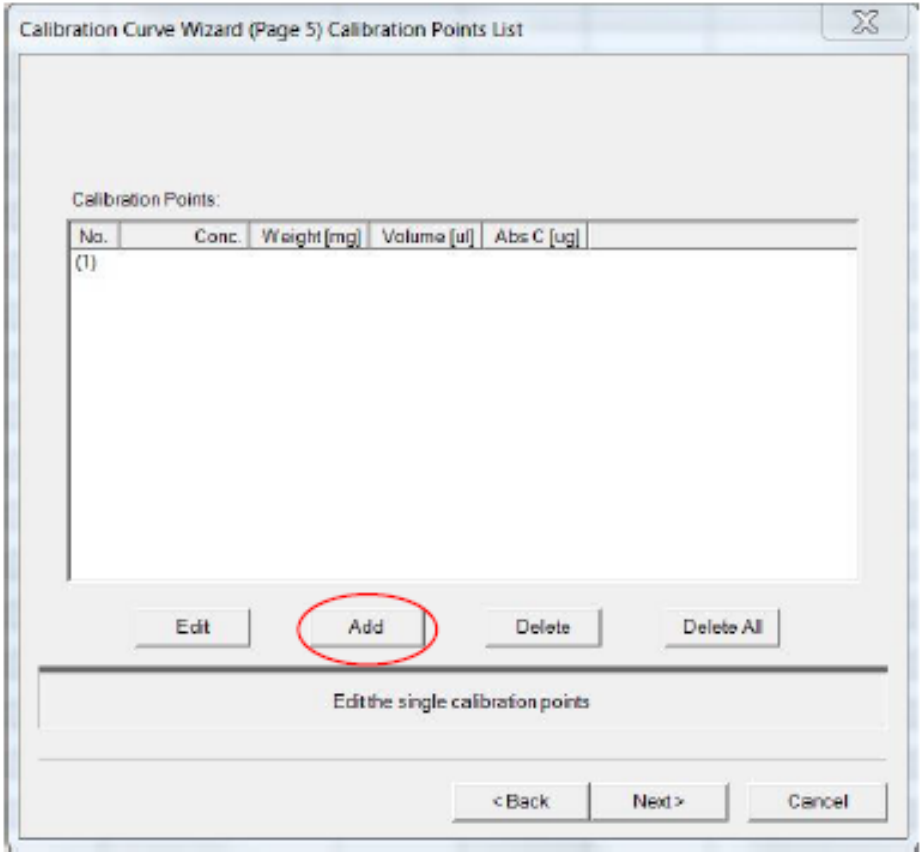


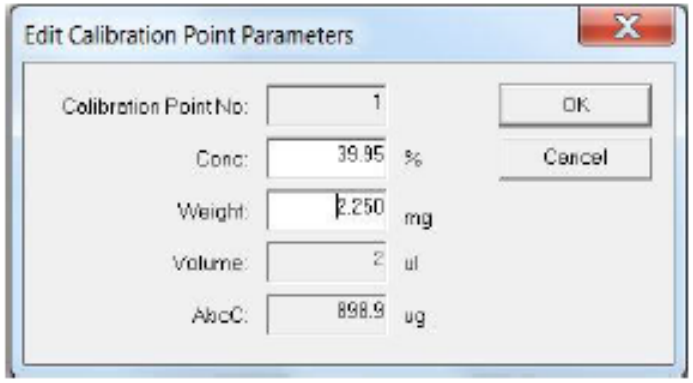
3. Select the unit, in this case %, whether the sample is measured in weight or volume and make sure the cell length is labeled appropriately for the concentration of carbon being measured. Short for high concentrations (% levels) and long for ppm levels. Click Next to continue.



Solid Sample Module Analysis

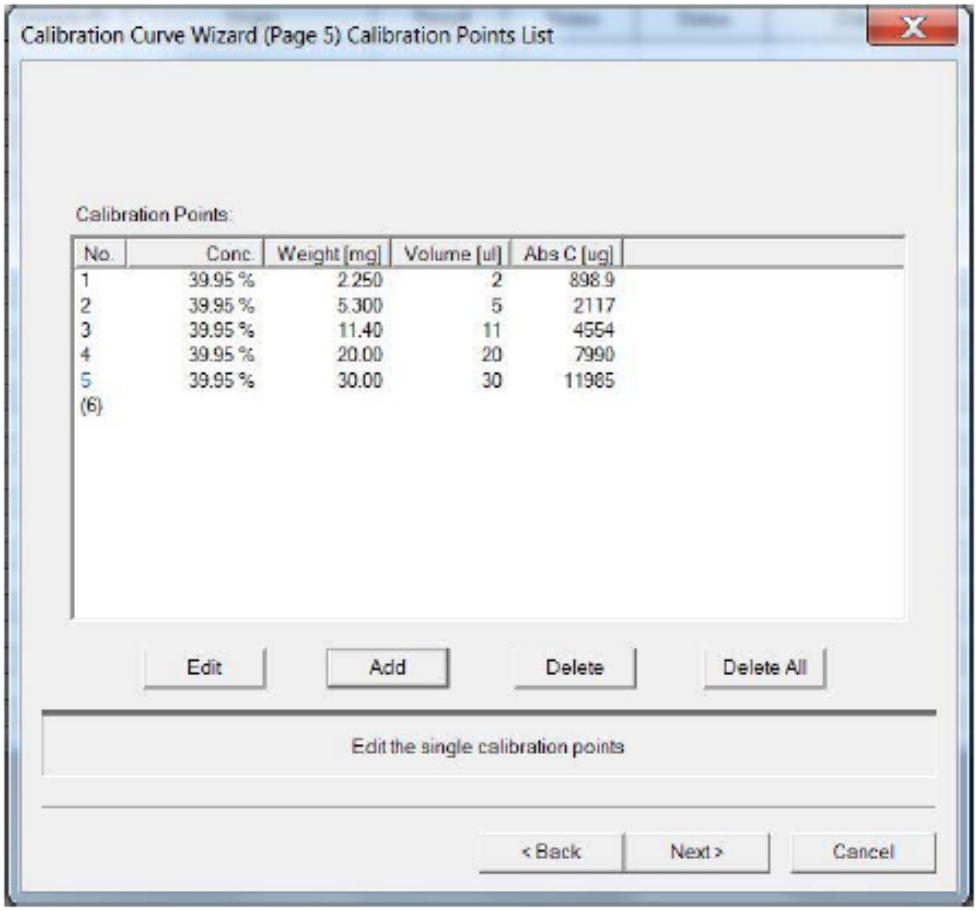
4. The Calibration Points List is where the concentration of carbon in the standard and the standard weight are entered. This will be done for each point in the curve. Press the Add button to open the Edit Calibration Point Parameters window.





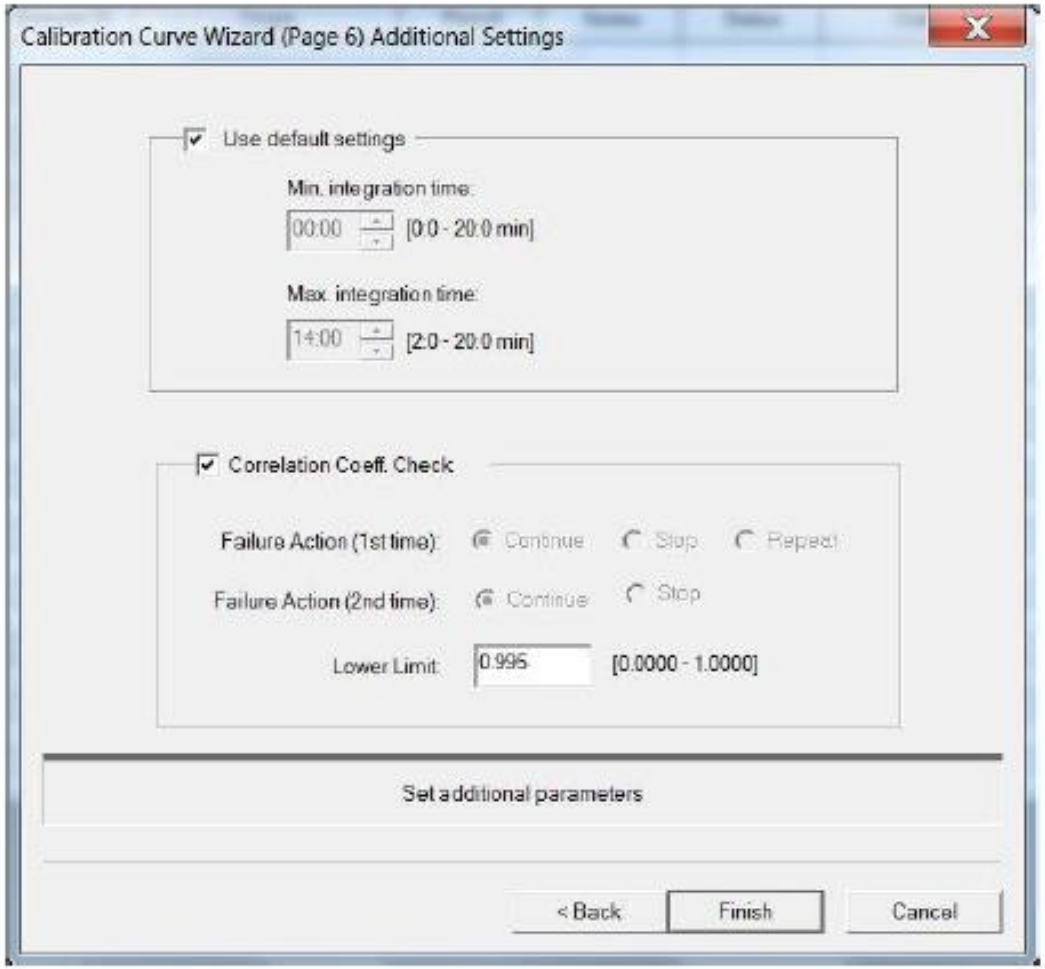
Solid Sample Module Analysis

5. Here we are using Glucose as a standard. The concentration of Carbon in Glucose is 39.95%. Weigh a small amount of Glucose into a sample boat and enter the weight. Continue this process until all points of the curve are entered. The Calibration Points List will look like this. Click Next to continue.



Solid Sample Module Analysis

6. In the Additional Parameters window check the Use Default Settings box. If desired also check the Correlation Coefficient Check box. Click Finish to complete the process.

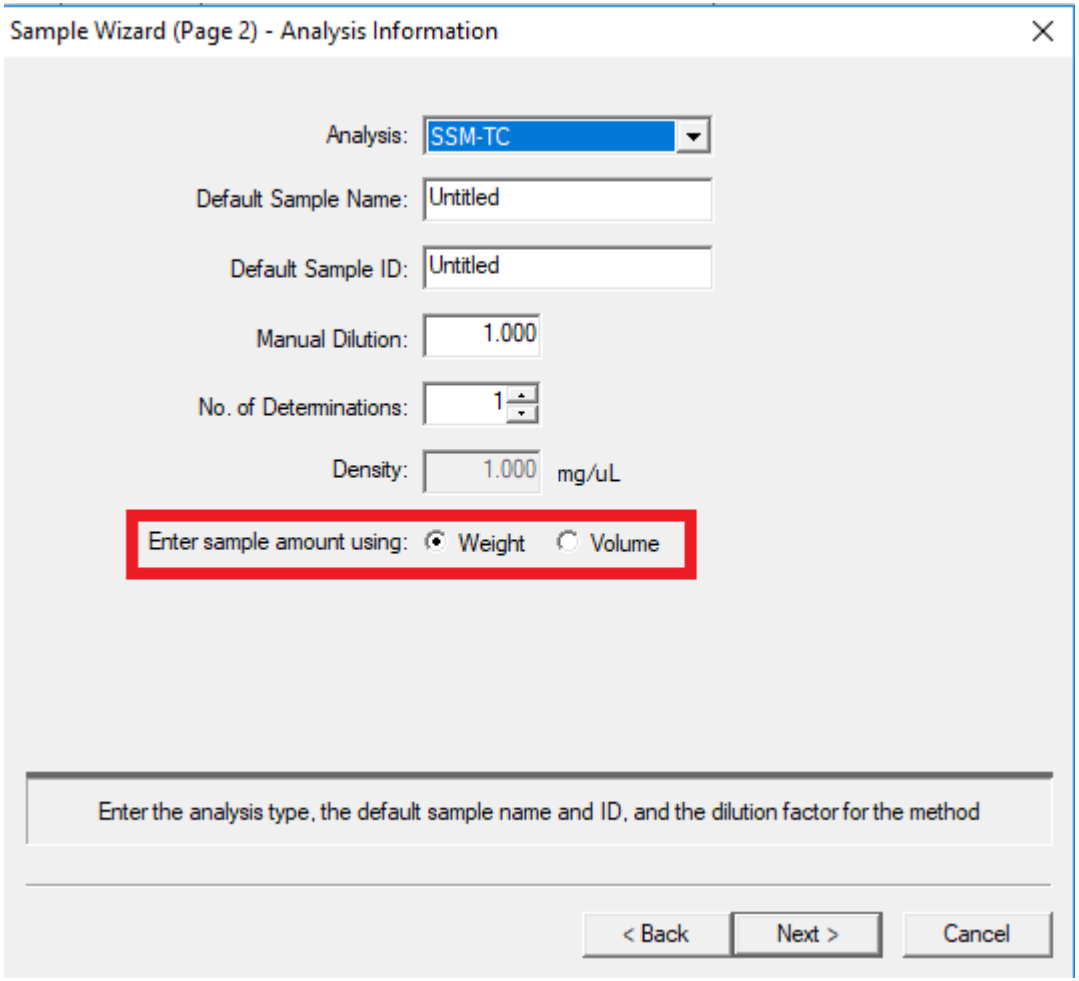


Solid Sample Module Analysis

Once a calibration curve for solid samples has been created, samples can be entered into the sample table just like for liquid samples.

See Creating a Sample Table

Note that sample amount can either be entered by weight or by volume.

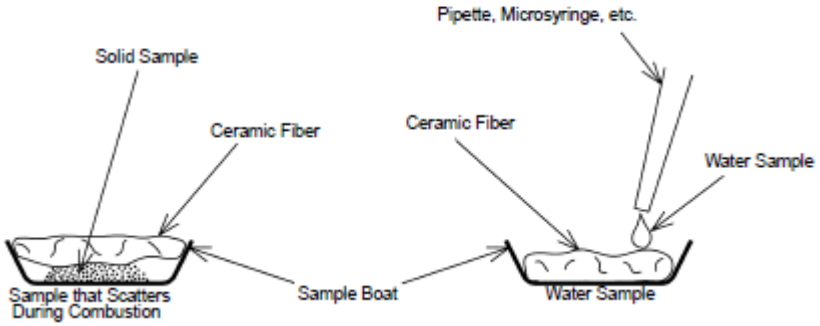


Solid Sample Module Analysis

Samples are then weighed or pipetted into the ceramic sample boats. The ceramic boats are then placed on the sample boat holder in the sample ports in preparation for measurement

Note: The maximum measurable mass of a liquid sample is 0.5g for TC and 0.3g for IC.

For TC analysis of a liquid sample, the sample can be impregnated into ceramic fiber placed on the bottom of the sample boat. Ceramic fiber is not used for IC analyses.



After sealing the sample boat into the sample port chamber you need to wait 3-5 minutes before starting analysis. This is the time it takes for the air trapped in the system to work through the detector. Failure to do so will result in false peaks from the CO2 in the air compromising measurement of the sample.

Solid Sample Module Analysis

3.1.6 Sample Additives

Sample additives are used with some samples to accelerate combustion or thermal decomposition reactions.

The required amount of sample additive and its effect vary according to the type of sample. Therefore, determine the optimum additive conditions to suit the actual sample.

Use the following commercially available sample additives for the analysis of organic elements, as necessary.

Tungsten Oxide (WO_3)

If a sample contains alkaline metals or alkaline earth metals, carbonates are generated along with carbon or carbon dioxide during combustion. Formation of these carbonates results in lower carbon values because the carbonates do not oxidize during combustion. To prevent this problem, tungsten oxide powder is spread over the entire sample.

Tungsten oxide can also be used as a sample additive for samples containing phosphorous, as they are often inflammable.

Vanadium Pentoxide (V_2O_5)

Vanadium pentoxide is used in the same manner as tungsten oxide for samples containing alkaline metals or alkaline earth metals.

Total Nitrogen Module (TNM-L)

With the optional TNM-L total nitrogen module, the analysis of the total nitrogen in a sample can be performed. Please note that it can only measure TOTAL nitrogen and cannot speciate out the different forms of nitrogen present.

It is possible to perform carbon and nitrogen measurements simultaneously with one sample injection

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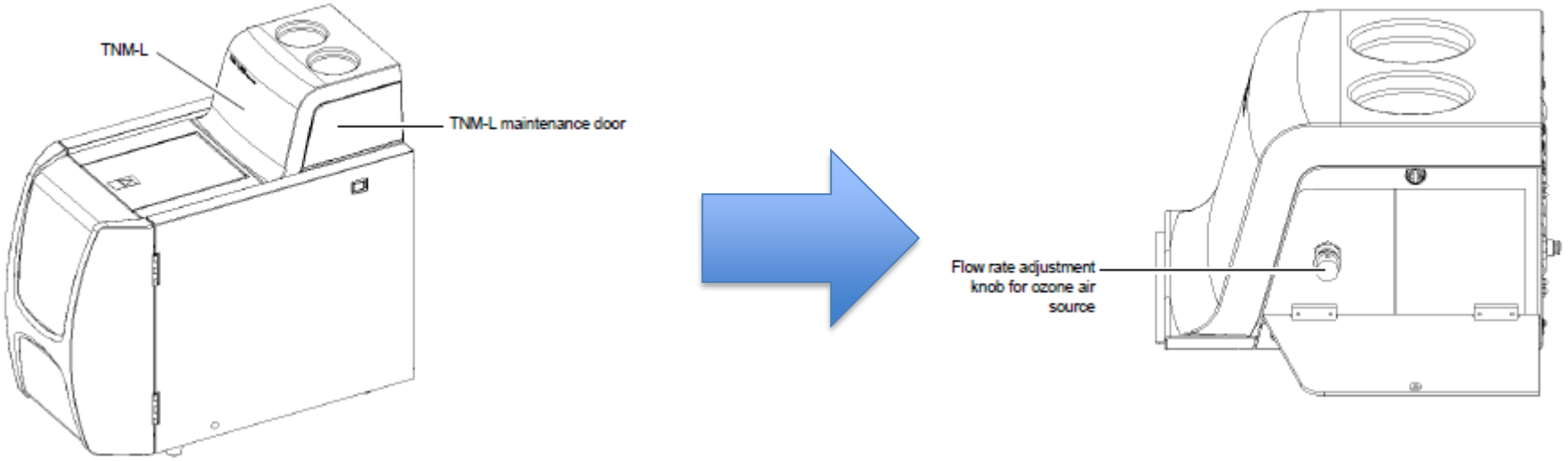
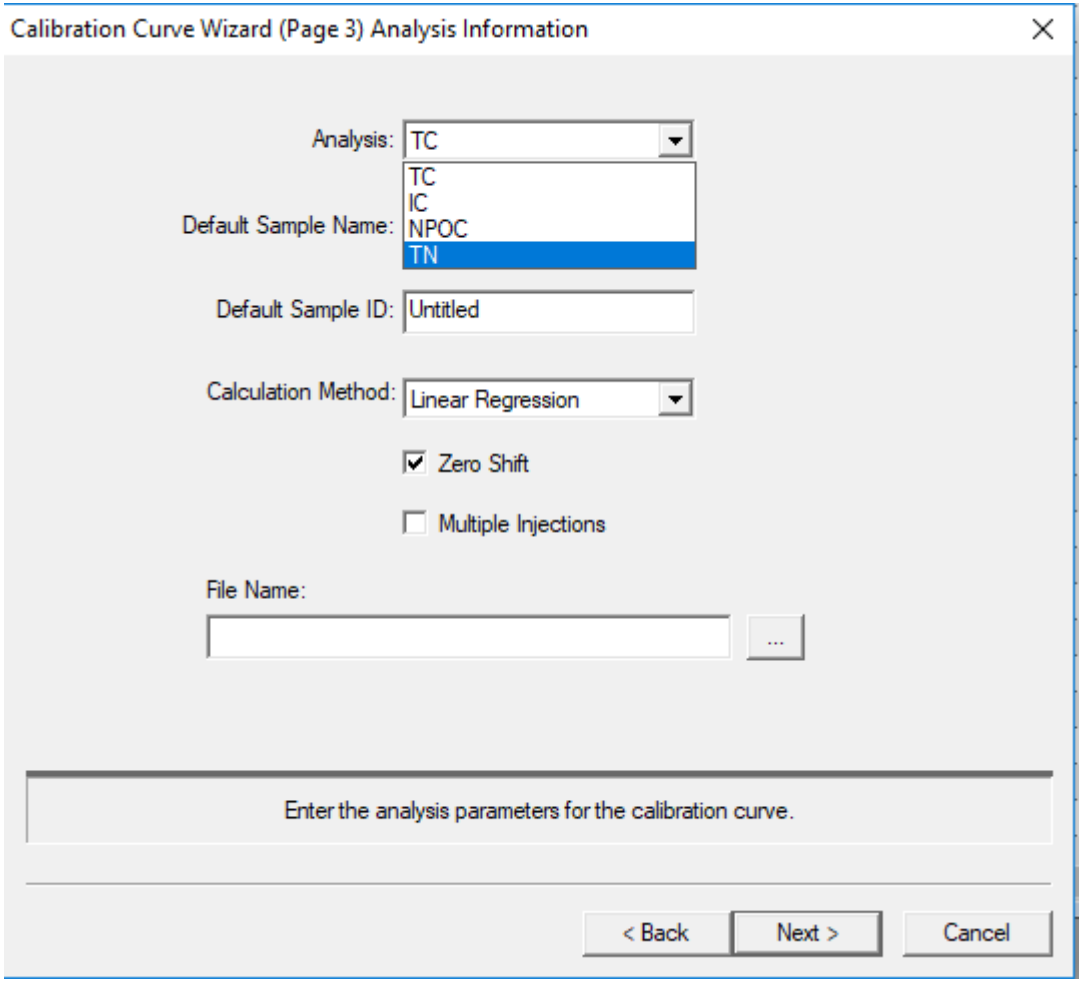
Total Nitrogen Module (TNM-L)



The supply gas pressure is displayed on the TN tab in the Background Monitor window. Watch the display while turning the carrier gas pressure adjustment knob on the pressure adjustment unit (inside the door on the right side of the TNM-L) to attain a supply gas pressure of 200 kPa.

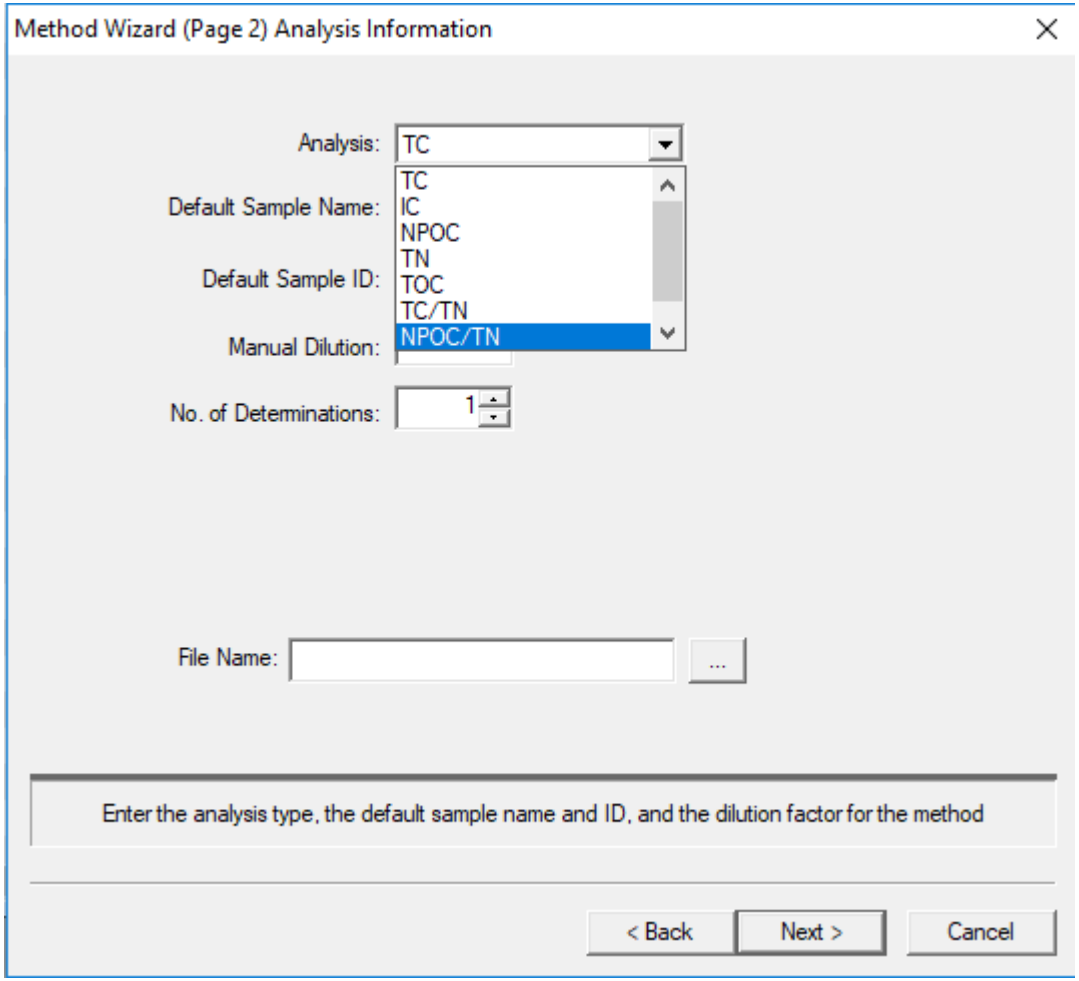
Total Nitrogen Module (TNM-L)

To create a calibration curve for total nitrogen analysis follow the instructions for creating a calibration curve. On page 3 of the calibration curve window make sure to select 'TN' form the drop down tab for 'Analysis'.



Total Nitrogen Module (TNM-L)

To measure carbon and nitrogen simultaneously, a method will need to be created. Follow the directions for creating a method and on page2 of the Method Wizard, use the drop down tab for ‘Analysis’ to select either ‘TC/TN’ or ‘NPOC/TN’. Follow the wizard and on the next pages select the correct calibration curves and properties for carbon analysis and nitrogen analysis.

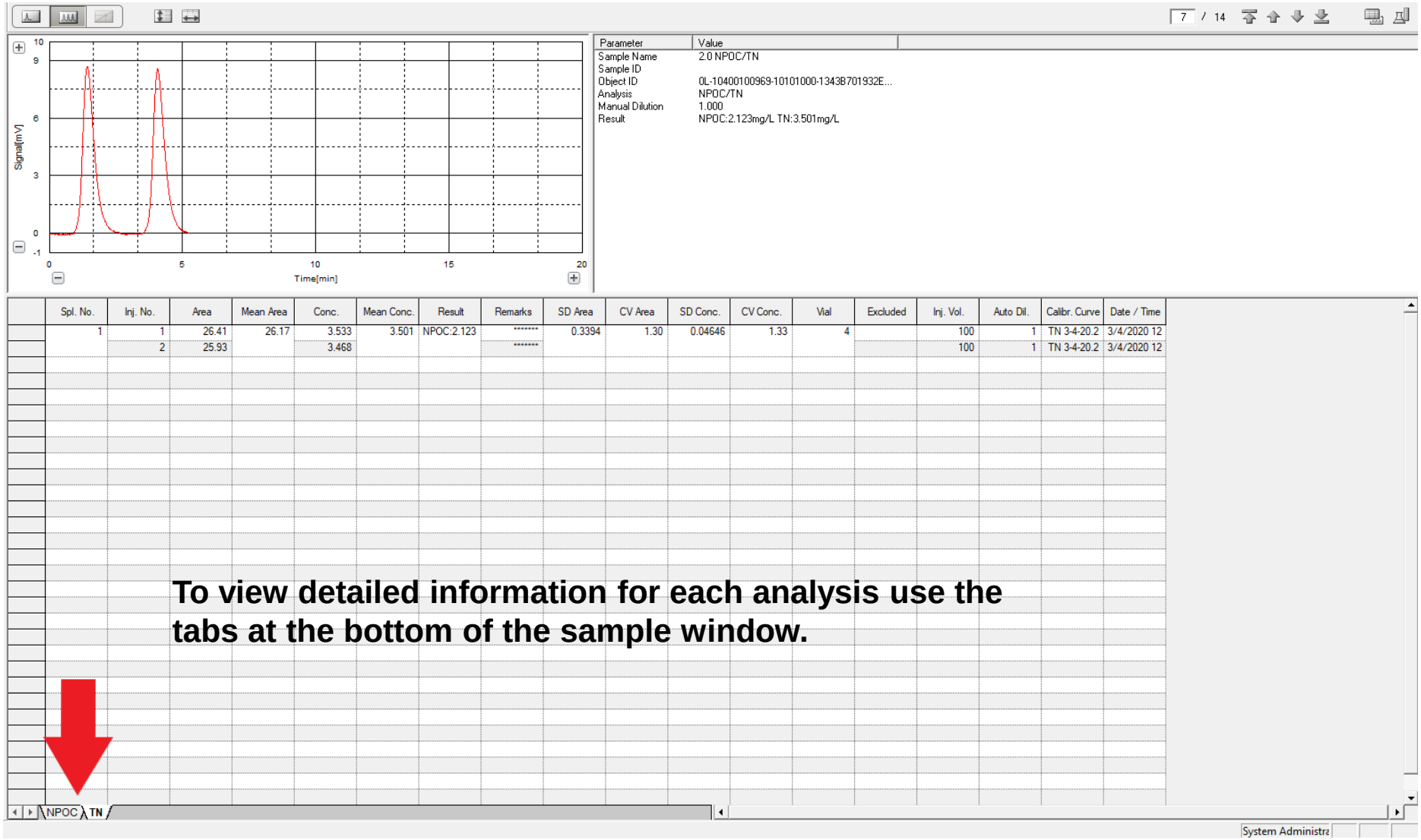


Total Nitrogen Module (TNM-L)

When samples are measured using a method for both carbon and nitrogen, the instrument will perform one injection and measure both carbon and nitrogen simultaneously. Results for both analysis will be displayed in the 'Result' of the Sample Table.

	Type	Analysis	Sample Name	Sample ID	Origin	Result
1	Unknown	TN	DI Flush		TN 3-2-20.2020_03_02_15_06_12.cal	TN:-0.1455mg/L
2	Standard	TN	TN curve with 100 ul volume		TN 3-4-20.2020_03_04_09_07_56.cal	
3	Unknown	TN	4.0 cal std chk (from same cal vial)		TN 3-4-20.2020_03_04_09_07_56.cal	TN:3.757mg/L
4	Unknown	NPOC/TN	Blank		NPOC-TN Method 2-26-2020.met	NPOC:0.01210mg/L TN:-0.08153mg/L
5	Unknown	NPOC/TN	2.0 NPOC		NPOC-TN Method 2-26-2020.met	NPOC:2.330mg/L TN:-0.08153mg/L
6	Unknown	NPOC/TN	2.0 TN		NPOC-TN Method 2-26-2020.met	NPOC:0.2148mg/L TN:3.507mg/L
7	Unknown	NPOC/TN	2.0 NPOC/TN		NPOC-TN Method 2-26-2020.met	NPOC:2.123mg/L TN:3.501mg/L
8	Unknown	NPOC/TN	2.0 CCV TN remade		NPOC-TN Method 2-26-2020.met	NPOC:0.05197mg/L TN:4.842mg/L
9	Unknown	TN	2.0 CCV TN old curve (before PM and		TN 8-1-19.2019_08_01_13_37_03.cal	TN:1.778mg/L
10	Unknown	TN	2.0 CCV TN		TN 3-4-20.2020_03_04_09_07_56.cal	TN:3.189mg/L
11	Standard	TN	TN curve with 40 ul volume		TN 3-4-20 B.2020_03_04_13_58_10.cal	
12	Unknown	TN	2.0 CCV TN		TN 3-4-20 B.cal	TN:0.00557mg/L
13	Unknown	NPOC/TN	2.0 CCV TN (on Method)		NPOC-TN Method 2-26-2020.met	NPOC:0.1362mg/L TN:2.835mg/L
14	Unknown	TN	2.0 CCV TN (on cal curve)		TN 3-4-20 B.2020_03_04_13_58_10.cal	TN:3.186mg/L

Total Nitrogen Module (TNM-L)



Pharmaceutical Water Testing

Running USP-643 with the TOC-L

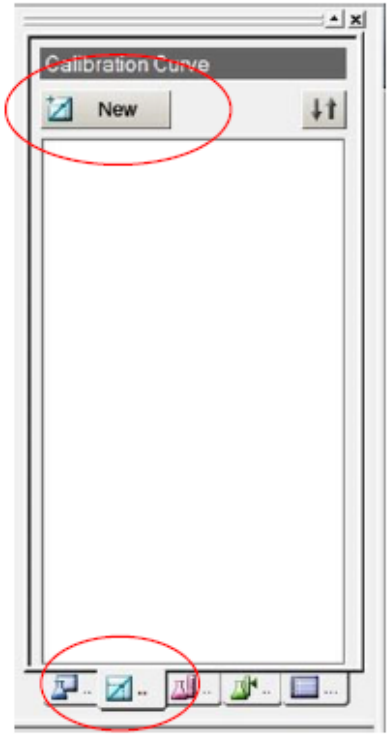
USP-643 is a method guideline established by the United States Pharmacopoeia for the analysis of high purity water and water for injection. The TOC-L is a combustion type TOC and is well suited for performing this system suitability analysis. The system suitability test uses Sucrose, an easily oxidized compound and 1,4 Benzoquinone, a more difficult compound to oxidize. Reagent water is measured and subtracted from each result and a % recovery calculated. The response for 1,4 Benzoquinone should be 100% +/- 15% that of Sucrose.

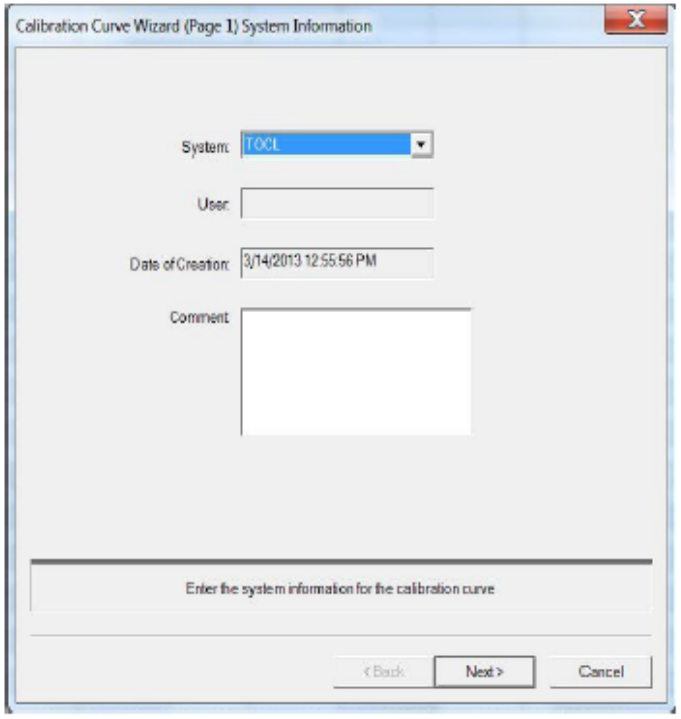
The basic steps in this process are to create a USP specific calibration curve. Next create a USP specific method that references the USP calibration curve. Create a sample table, populate it with a calibration curve and then add both the system suitability and unknown samples.

Pharmaceutical Water Testing

Creating the USP Calibration Curve

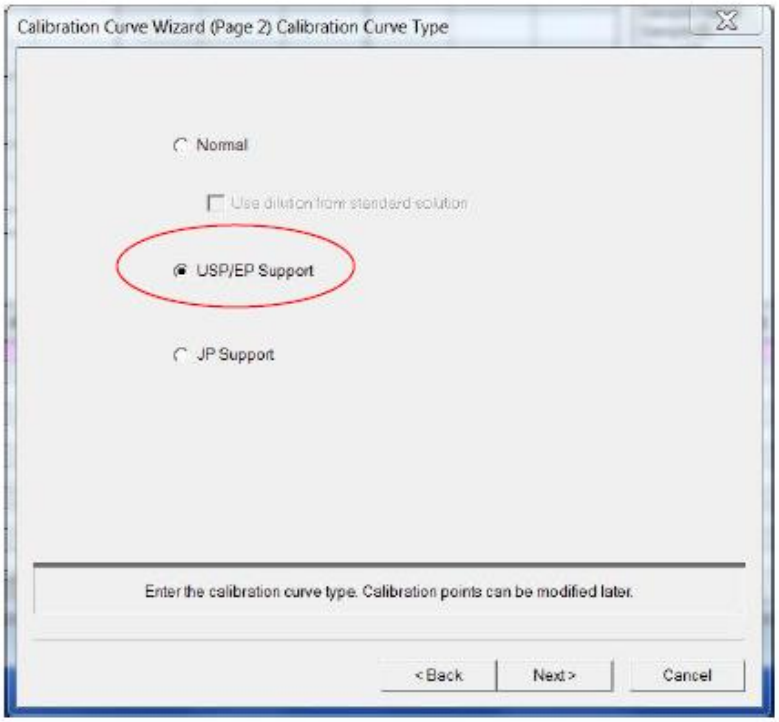
1. From the File View window select the "New" button to open the Calibration Curve Wizard. Select the system and enter any comments as needed. Click Next to continue.



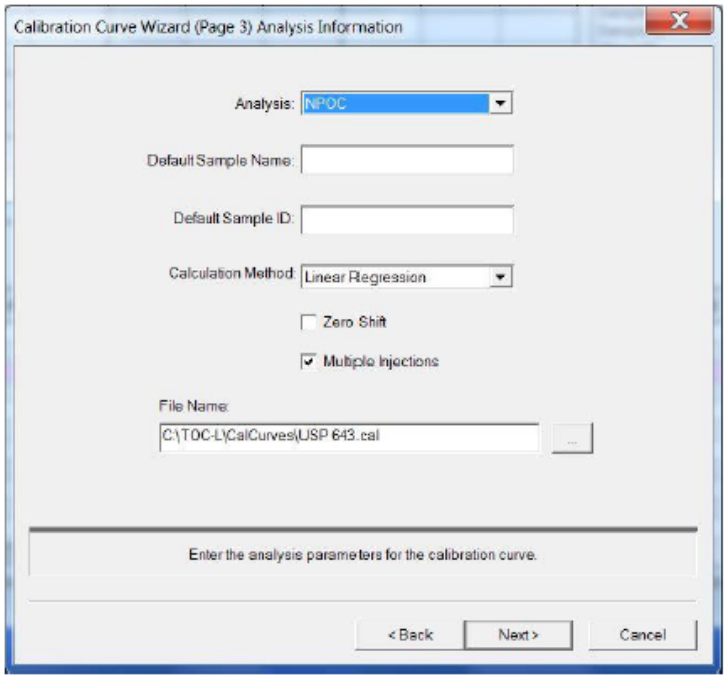


Pharmaceutical Water Testing

2. Select USP/EP Support to activate the USP functions. Click Next to continue.

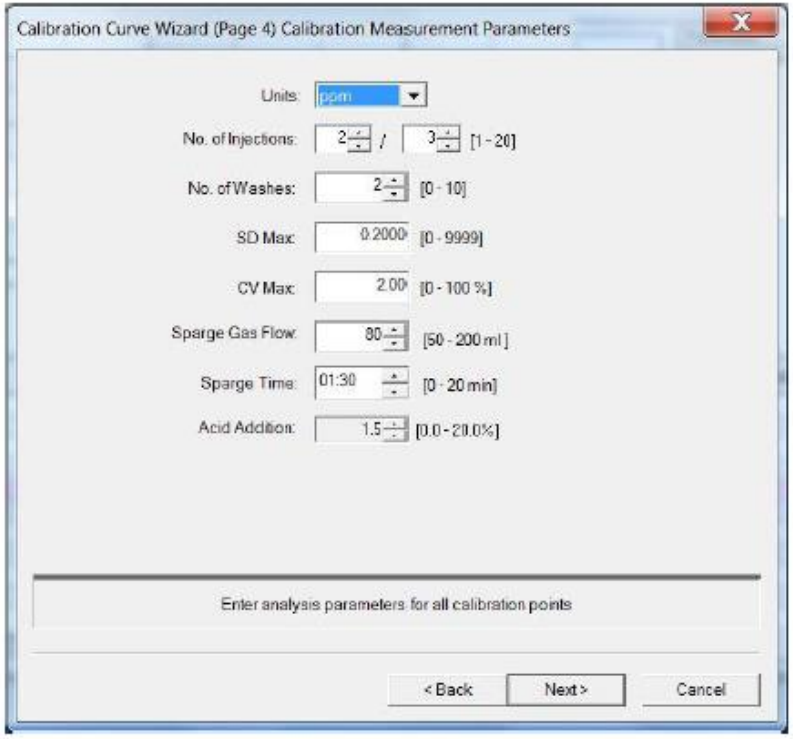


3. Choose the analysis type, uncheck Zero Shift, check Multiple Injections and enter an appropriate file name. Click Next to continue.

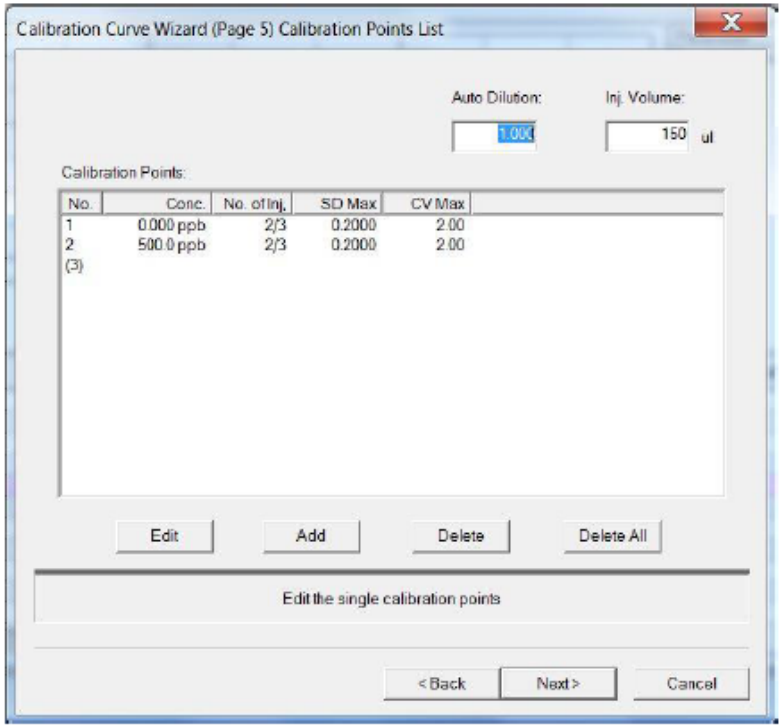


Pharmaceutical Water Testing

4. Select the desired units and any other settings as needed. Click Next to continue.

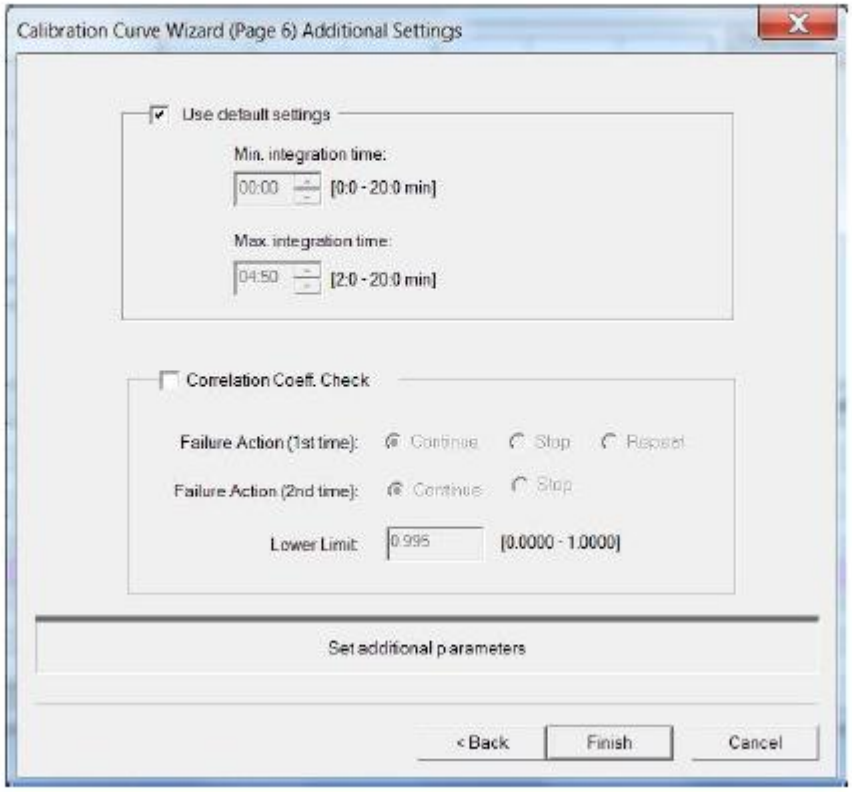


5. The Calibration Points List is automatically populated to reflect USP method and the injection volume is set accordingly. Click Next to continue.



Pharmaceutical Water Testing

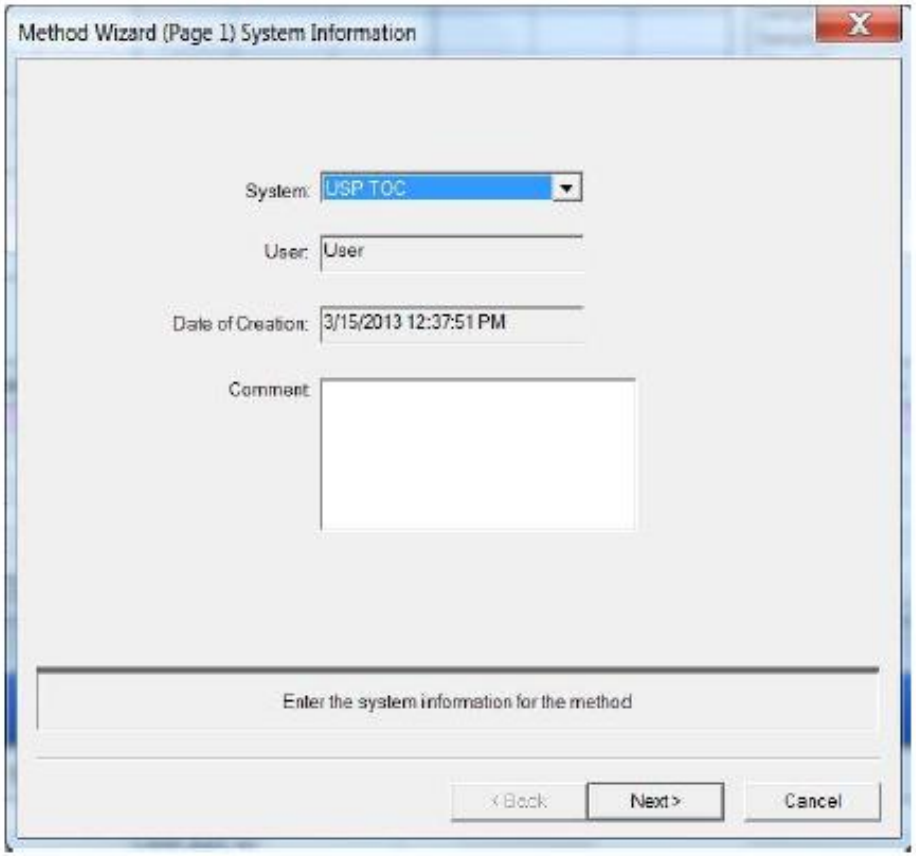
6. Ensure that the Use Default Settings box is checked. Click Finish to complete the process.



Pharmaceutical Water Testing

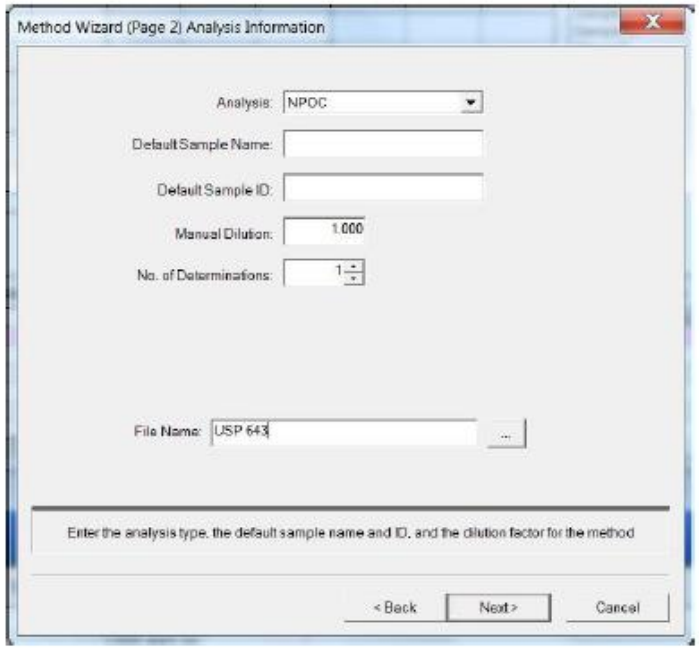
Creating the USP Method

1. From the File View window select the "New" button to open the Method Wizard. Select the system and enter any comments as needed. Click Next to continue.

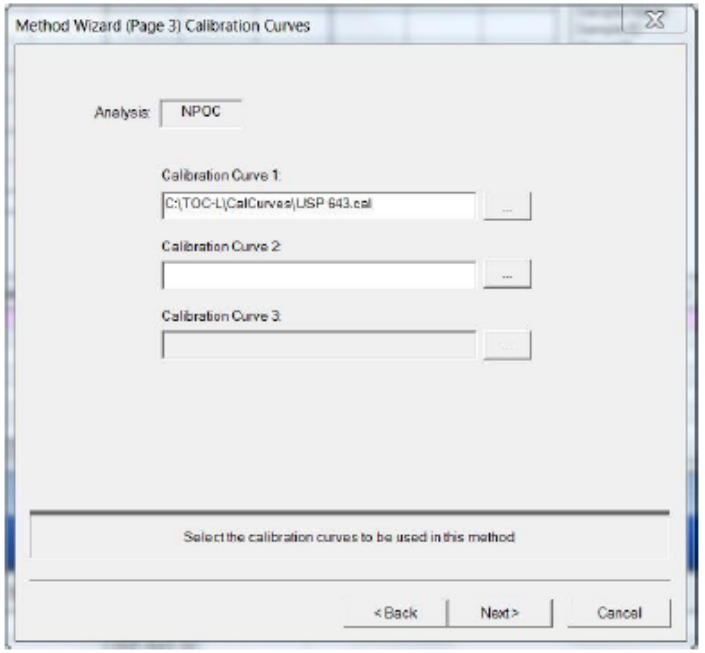


Pharmaceutical Water Testing

2. Select the analysis type and choose an appropriate file name. Click Next to continue.

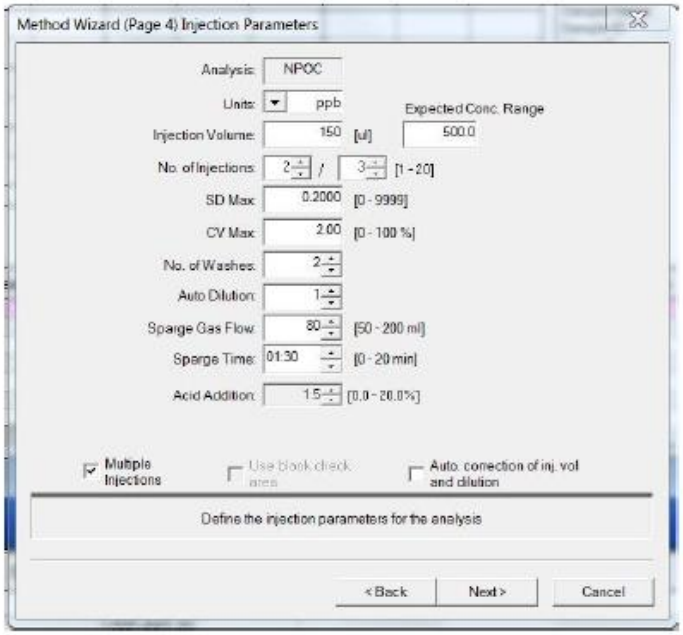


3. Select the appropriate calibration curve file for Calibration Curve 1. Click Next to continue.

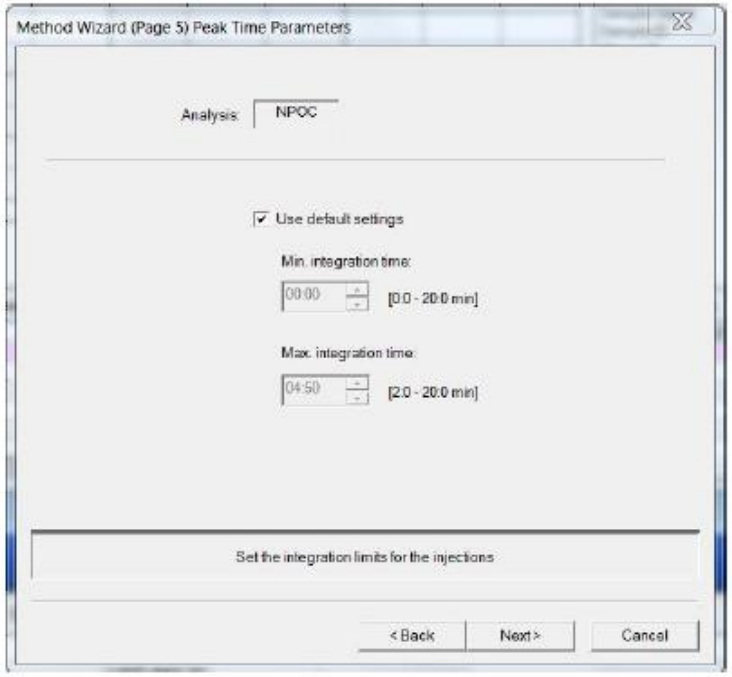


Pharmaceutical Water Testing

4. Select the desired units and any other appropriate changes. Click Next to continue.

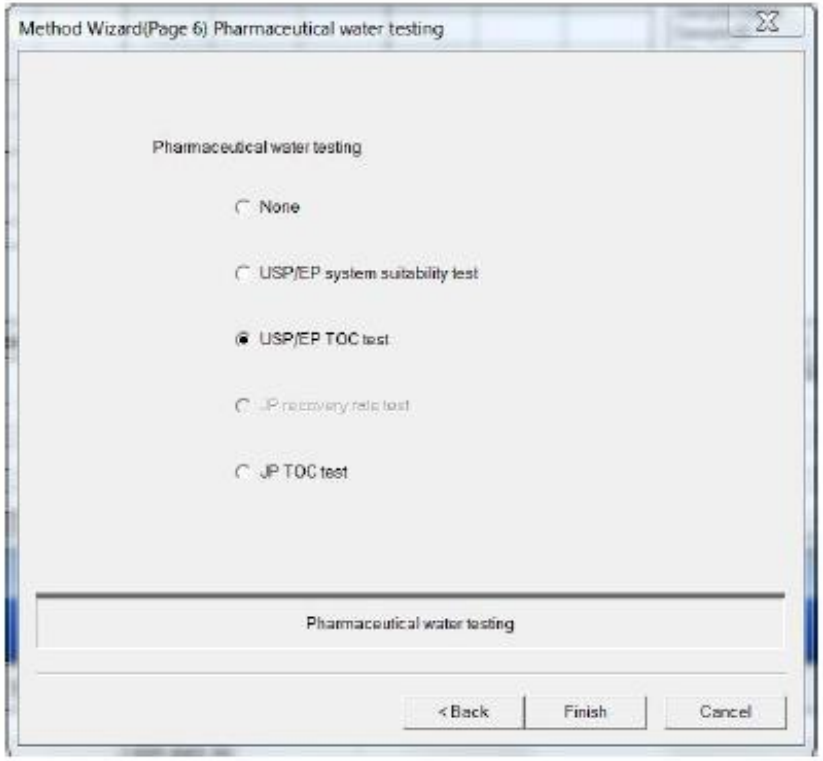


5. Ensure that the Use Default Settings box is checked. Click Next to continue.



Pharmaceutical Water Testing

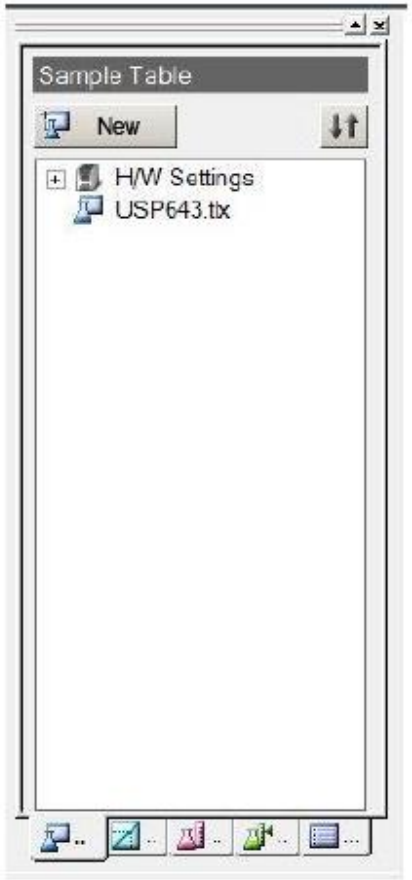
6. Check the USP/EP TOC Test box to activate the function. Click Finish to complete the process.

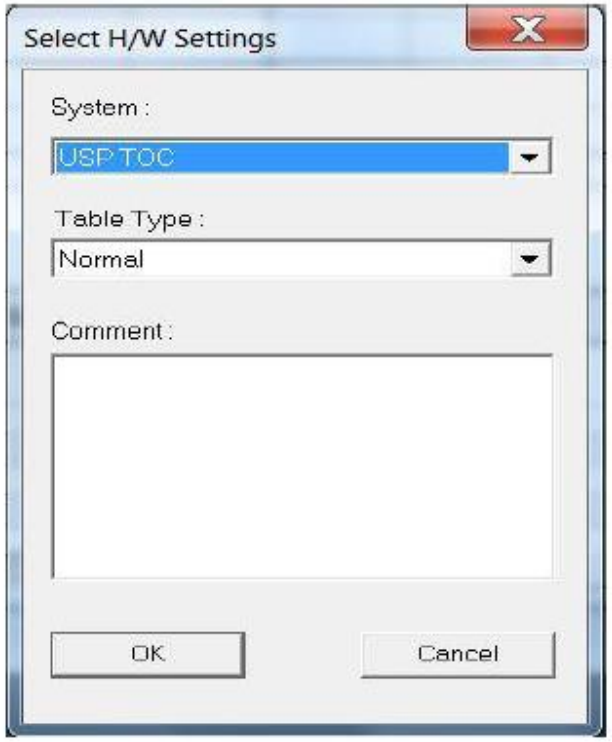


Pharmaceutical Water Testing

Creating a USP Sample Table

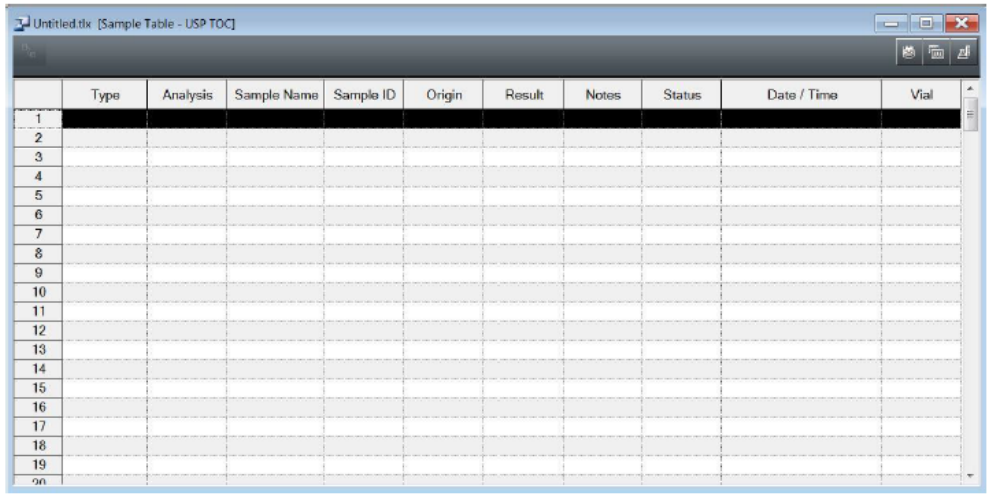
1. To create a working sample table for running USP samples several steps must be taken. From the File View window select the "New" button to open a new Sample Table. Select the system and click OK to open the blank sample table.



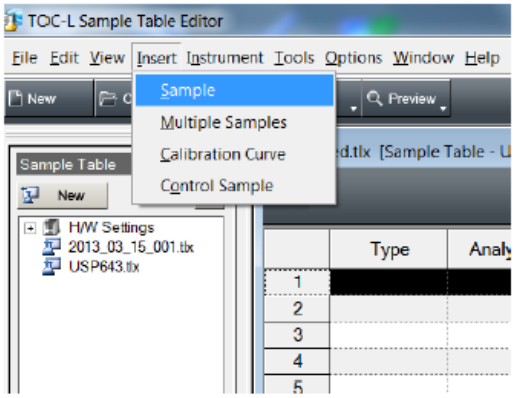


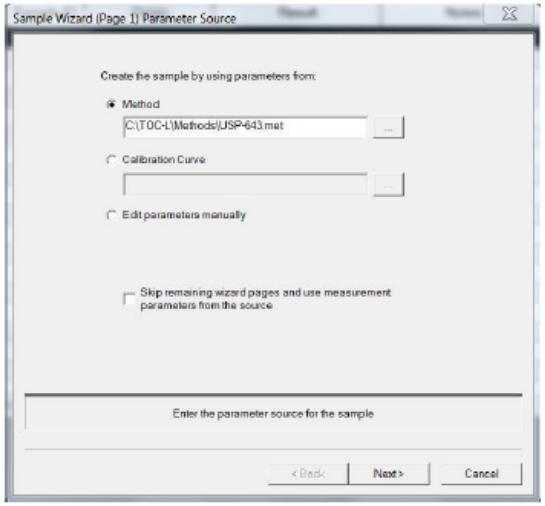
Pharmaceutical Water Testing

2. A blank sample should be added as the first sample run to precondition the system before calibrating. Click on the number 1 to highlight the line.



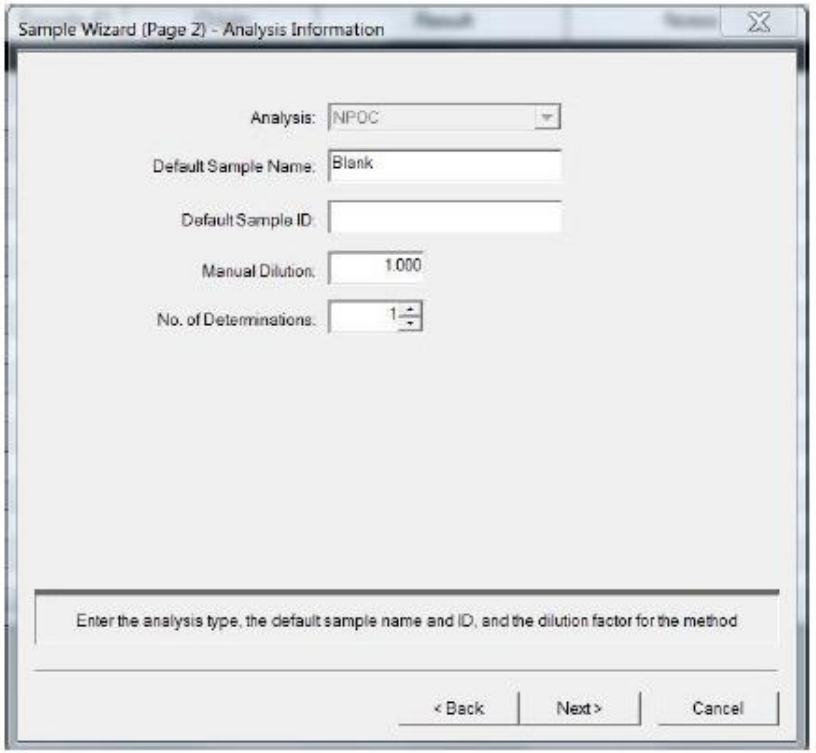
3. From the Menu bar click Insert/Sample. Proceed to page 1 of the Sample Table Wizard, choose the Method radio button and select the appropriate Method file. Click Next to continue.



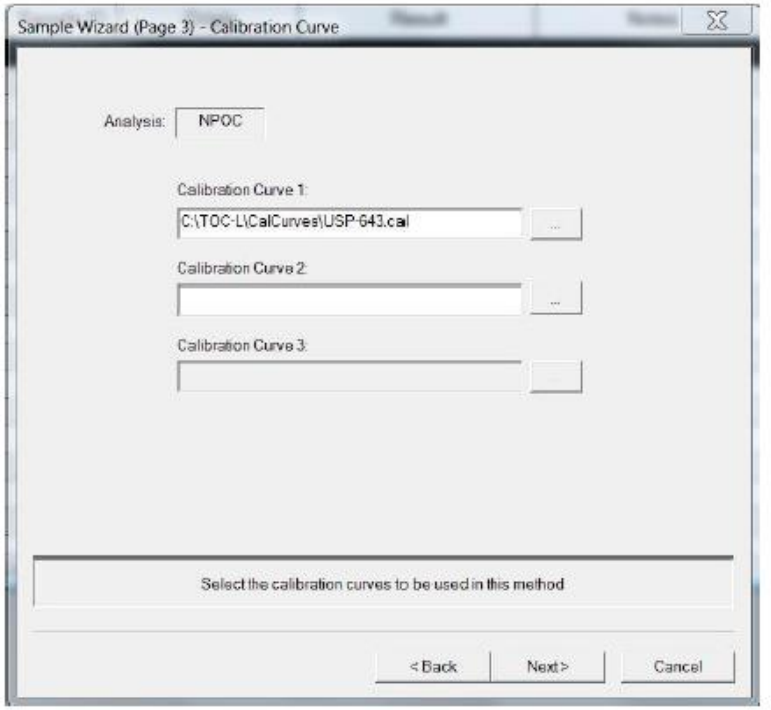


Pharmaceutical Water Testing

4. Enter a sample name and click Next to continue.

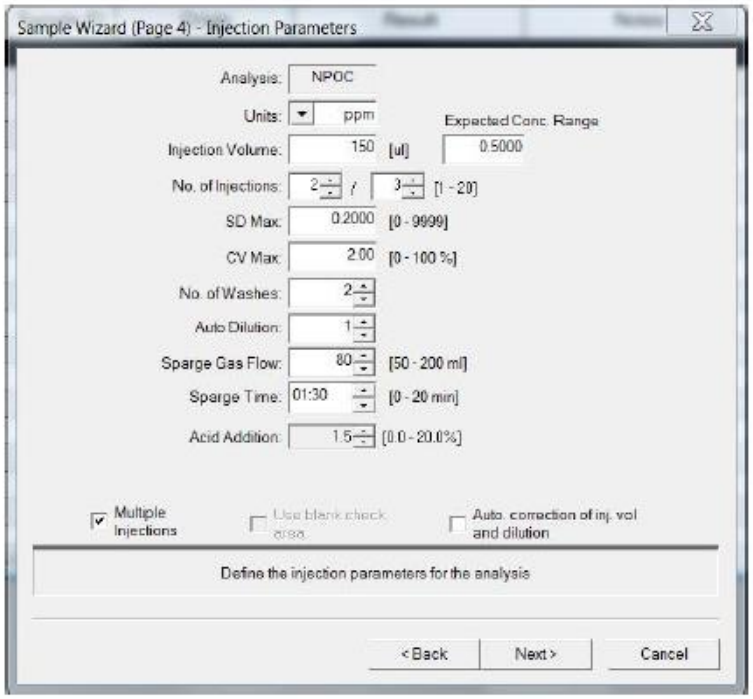


5. The calibration file name should be populated based on the Method file chosen previously. Click Next to continue.

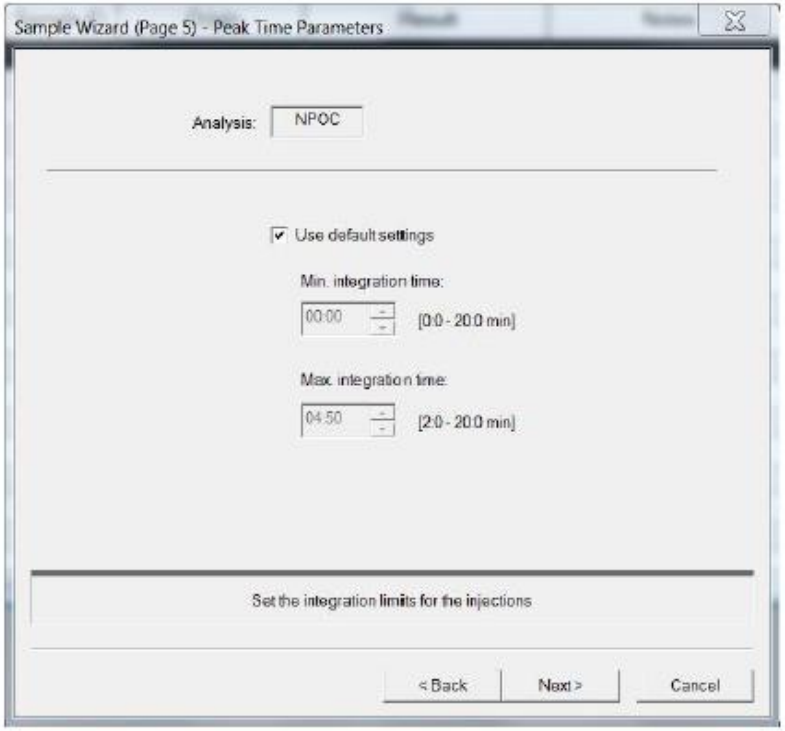


Pharmaceutical Water Testing

6. The Injection Parameters page is populated based on the contents in the Method file. Make any desired changes and click Next to continue.

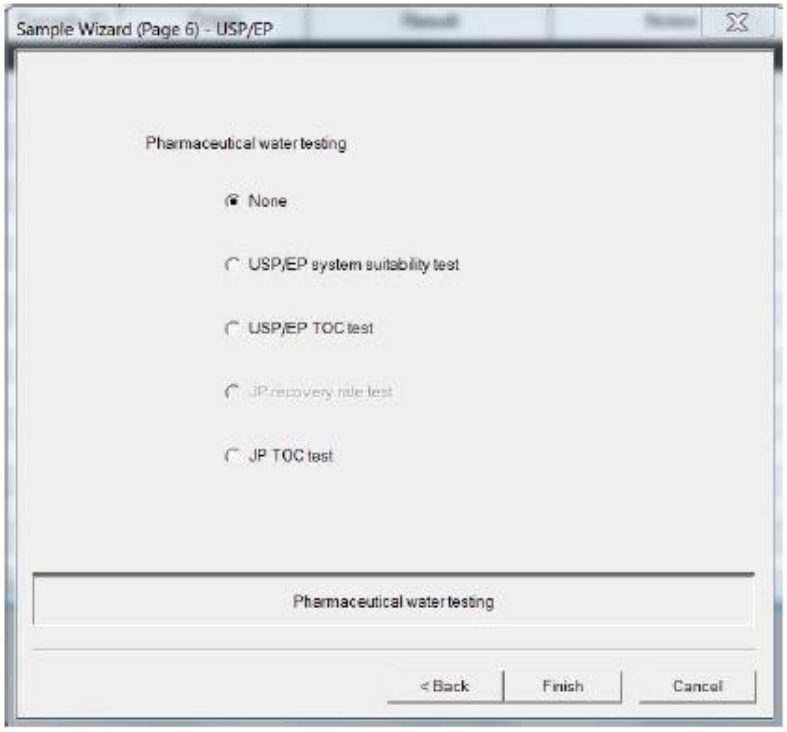


7. The Peak Time window opens with default parameters. Click Next to continue.

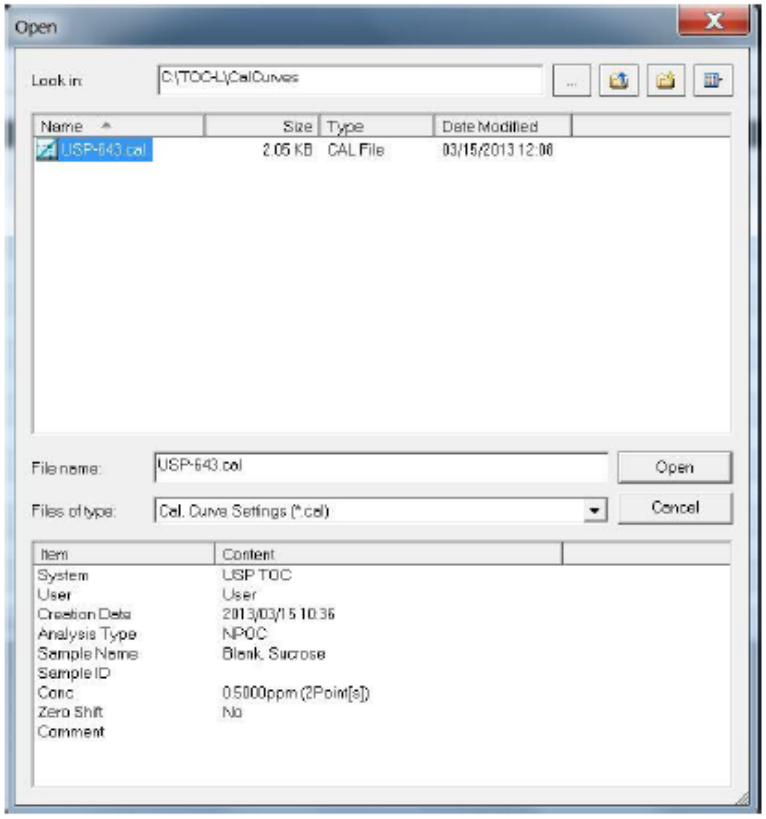


Pharmaceutical Water Testing

8. In the USP/EP window check None and click Finish to complete the process.

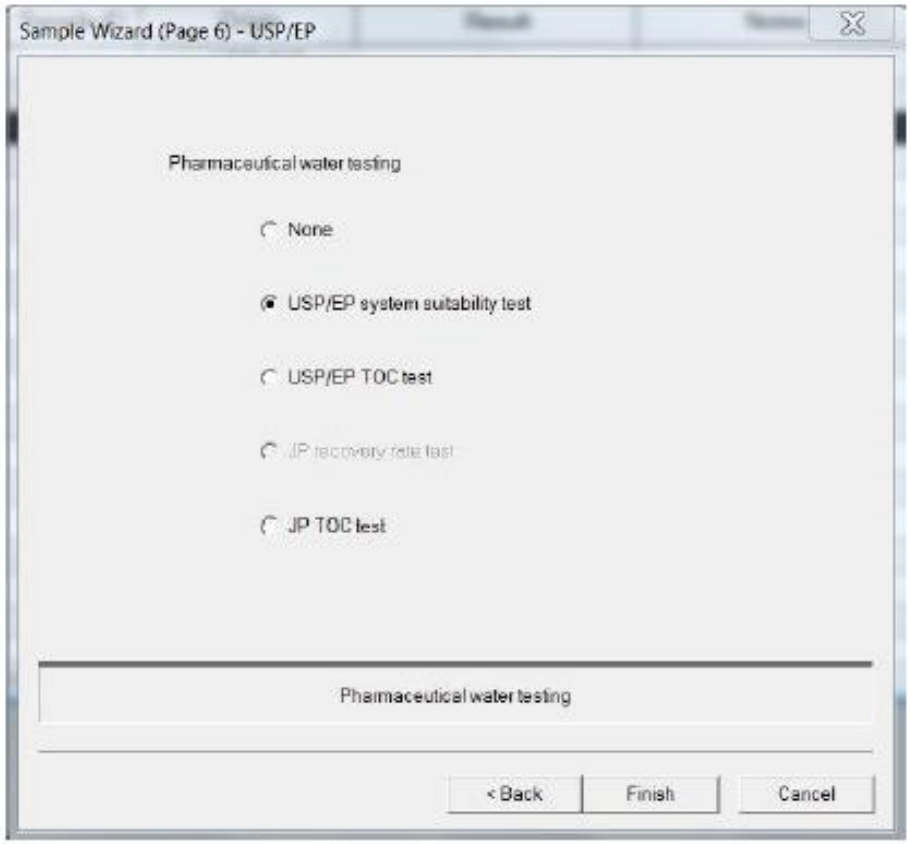


9. Highlight line number 2 and input a USP suitable calibration curve.



Pharmaceutical Water Testing

10. For line number 3 input a USP system suitability sample. Use the Insert Sample functior as normal, enter 1,4 Benzoquinone as the sample name but at page 6 choose USP/EP system suitability test. Click Finish to complete the process.



Pharmaceutical Water Testing

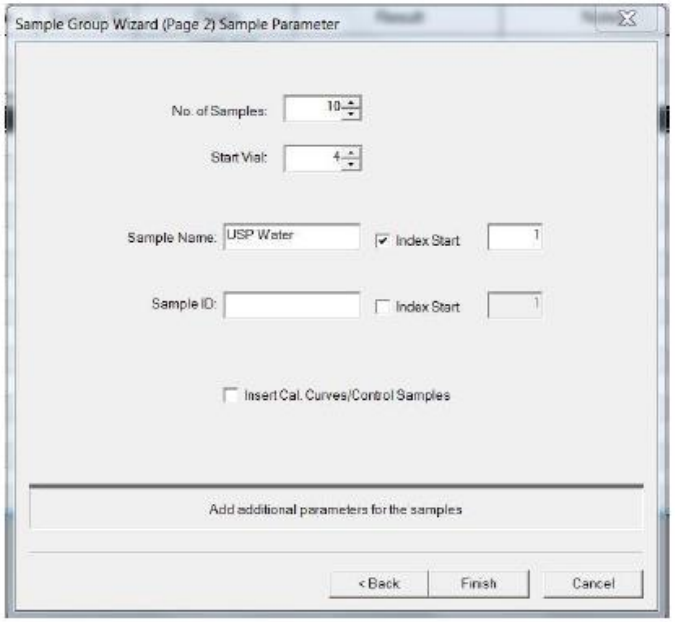
11. The sample table should now look like the following.

Untitled.tlx [Sample Table - USP TOC]

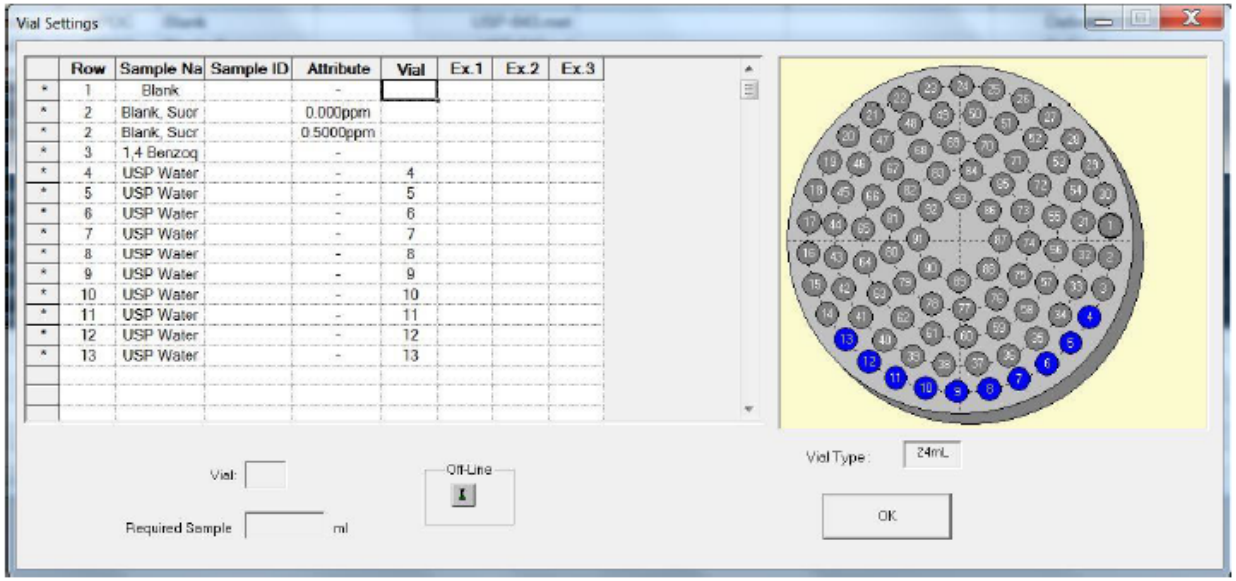
	Type	Analysis	Sample Name	Sample ID	Origin	Result	Notes	Status	Date / Ti
1*	Unknown	NPOC	Blank		USP-643.met			Defined	
2*	Standard	NPOC	Blank, Sucrose		USP-643.cal			Defined	
3*	Unknown	NPOC	1,4 Benzoquinone		USP-643.met			Defined	
4									
5									
6									
7									
8									
9									
10									
11									

Pharmaceutical Water Testing

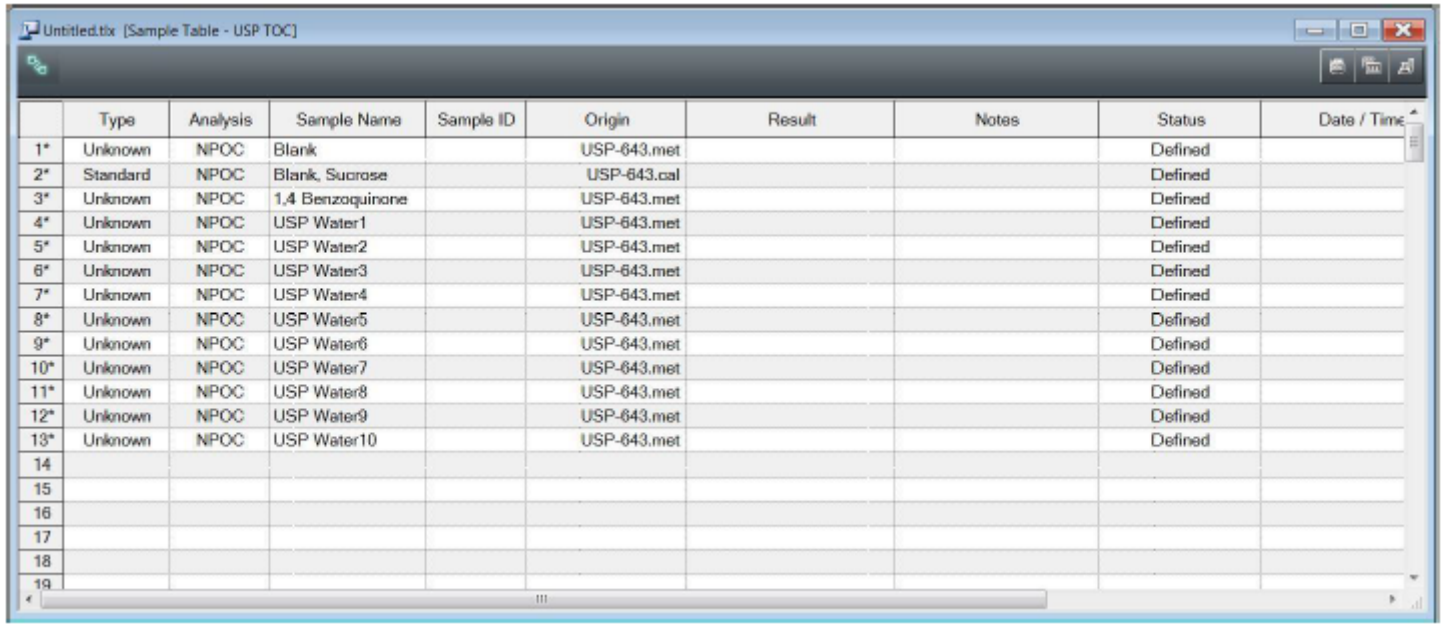
12. Highlight line number four and Insert Multiple Samples. Set the desired number of samples, the start vial number and any sample names. Click Finish to complete the process.



13. The Vial Settings window should open automatically. Ensure that all vials are set accordingly in both the auto sampler and Sample Table. Click OK to continue. The sample table is ready to run.



Pharmaceutical Water Testing



	Type	Analysis	Sample Name	Sample ID	Origin	Result	Notes	Status	Date / Time
1*	Unknown	NPOC	Blank		USP-643.met			Defined	
2*	Standard	NPOC	Blank, Sucrose		USP-643.cal			Defined	
3*	Unknown	NPOC	1,4 Benzoquinone		USP-643.met			Defined	
4*	Unknown	NPOC	USP Water1		USP-643.met			Defined	
5*	Unknown	NPOC	USP Water2		USP-643.met			Defined	
6*	Unknown	NPOC	USP Water3		USP-643.met			Defined	
7*	Unknown	NPOC	USP Water4		USP-643.met			Defined	
8*	Unknown	NPOC	USP Water5		USP-643.met			Defined	
9*	Unknown	NPOC	USP Water6		USP-643.met			Defined	
10*	Unknown	NPOC	USP Water7		USP-643.met			Defined	
11*	Unknown	NPOC	USP Water8		USP-643.met			Defined	
12*	Unknown	NPOC	USP Water9		USP-643.met			Defined	
13*	Unknown	NPOC	USP Water10		USP-643.met			Defined	
14									
15									
16									
17									
18									
19									

USP Notes

- It is always recommended to add a blank sample to the first line of the sample table. This helps pre-condition the TOC catalyst in preparation for calibration and running general samples.
- The USP system suitability test calculates whether the results are within the +/- 15% pass/fail range but does not stop the sample table operation in case of failure. Adding a suitable control sample after the system suitability test allows the software to stop the sample table if there is an out of control situation.

Cleaning Validation with the SSM

In the pharmaceutical industry, "cleaning validation" where residues on manufacturing equipment are quantitated to verify whether the amount is within tolerance, has been a valuable criterion for quality control and safety. To perform cleaning validation by Shimadzu TOC analyzer, the following three methods are available depending on selected sampling and analysis methods.

- Swab Sampling - Direct Combustion Method
- Swab Sampling - Water Extraction-TOC Analysis Method
- Rinse Sampling - TOC Analysis Method

For sampling methods, there are the swab method and the rinse method, and the swab method is suitable for accurately evaluating the amount of residues. For analysis method, the direct combustion method can achieve an effective, rapid, and easy-to-perform analysis since it does not require any pretreatment process, such as sample extraction from swab materials. With these reasons, the swab sampling-direct combustion method using Shimadzu TOC analyzer is significantly effective as a method for cleaning validation.

In past Application News which introduced the swab sampling – direct combustion method, a piece of quartz glass filter paper was used as the swab sampling material. When using this material, however, the method sometimes required re-cleaning of the equipment since fragments of the filter paper were left on surfaces. To avoid this difficulty, the method here uses Easy wiper, made of quartz glass fibers which leaves no fragments on swabbed surfaces.

Here, we introduce the method using Easy wiper as the swab sampling material in the analysis of residual pharmaceutical products and their constituent substances.

Y.Ikezawa, M.Tanaka

Fig. 1 Easy Wiper

Preparation of Residue Sample

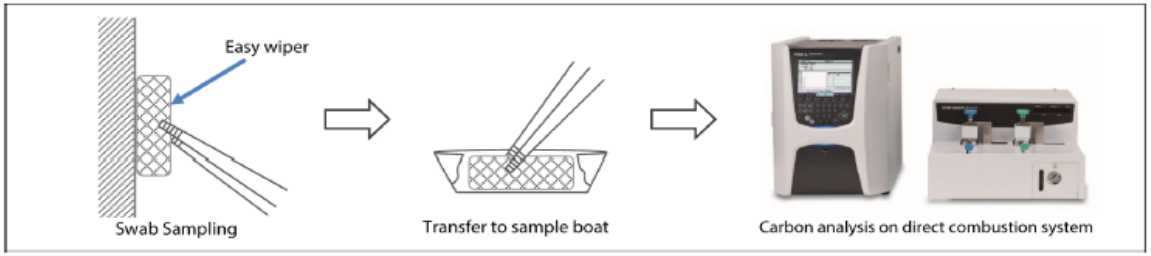
In order to evaluate the recovery rate in the cleaning validation with Easy wiper, residue analysis samples were created by allowing various pharmaceutical products and their constituents to adhere to a stainless plate. Water-soluble substances and water-insoluble substances were used for the sample (see Table 1).

The water-soluble substances and water-insoluble substances were dissolved with water and organic solvents listed in Table 1, respectively, and the solution concentrations were adjusted to 2000 mgC/L (=carbon concentration of 2000 mg/L). Then 100 µL of each solution was applied to a 5 cm × 5 cm square area, and the solvent was dried to produce residue analysis samples. Here, the carbon amount of each sample should be 200 µg.

Note that Rinderon ointment and Vaseline were prepared by previously obtaining the carbon amount on Shimadzu Total Organic Analyzer with Solid Sample Combustion Unit.

Table 1 Sample Type

Substance Name	Solubility in Water	Solvent Used for Solution Preparation
Tranexamic acid	Soluble	Water
Anhydrous caffeine	Soluble	Water
Isopropylantipyrine	Insoluble	Ethanol
Nifedipine	Insoluble	Acetone
Rinderon ointment	Insoluble	Acetone
Vaseline	Insoluble	Diethyl ether



Cleaning Validation with the SSM

Swab Sampling – Direct Combustion Method

In the swab sampling – direct combustion method, the internal surface of manufacturing equipment is swabbed with Easy wiper made of inorganic quartz glass fibers, and the swab is analyzed on the direct combustion system. See Fig. 2. The Easy wiper with which the residual substances are adhered is placed in the sample boat, and directly analyzed on the direct combustion system combining the Solid Sample Combustion Unit SSM-5000A and TOC analyzer.

In order to evaluate the recovery rate of various substances in this method, the stainless plate where the sample was adhered was swabbed with Easy wiper, and the swab was placed in a sample boat on SSM-5000A to perform TC analysis. A part of the analysis data is shown in Fig. 3.

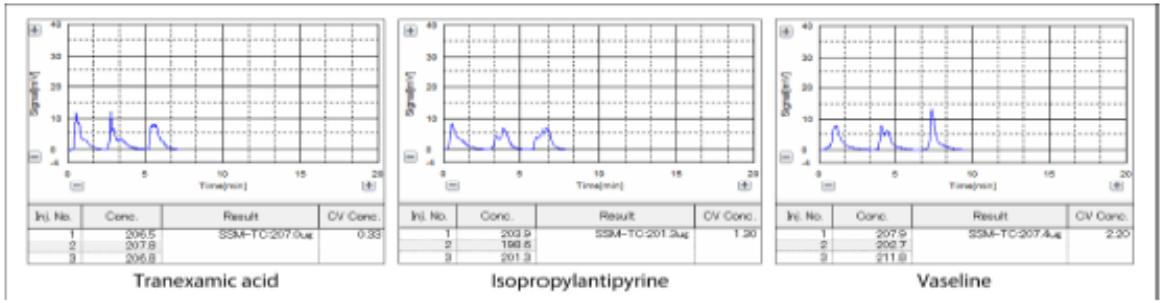
Since the carbon amount of the residue analysis sample is 200 µg, the TC value when the whole sample is swabbed should be 200 µg. Note that in order to evaluate a blank sample, a stainless plate with no substance adhered was swabbed and the swab was also analyzed in the same way. The blank value was then subtracted from each TC value, and the obtained value was compared with the theoretical value 200 µg to obtain the recovery rate. The results are shown in Table 3. High recovery rates of almost 100 % were obtained for all substances, whether they were water-soluble or water-insoluble.

Table 2 Measurement Condition

Analyzer	: Shimadzu Total Organic Carbon Analyzer TOC-L$_{CPH}$+Solid Sample Combustion Unit SSM-5000A (IC circuit bypath using system with cell switching valve set)
Cell Length	: Short cell
SSM carrier gas	: 400 mL/min oxygen gas
Analyzed Item	: TC (Total Carbon)
Calibration curve	: 1-point calibration curve using 30 µL of 1 %C aqueous glucose solution
Swab Material	: *Easy wiper made of quartz glass fiber (Heated at 600 °C for 15 minutes)*
Swabbing Method	: Add 200 µL of pure water to Easy wiper, and hold it with tweezers to swab the surface.

Table 3 Measurement Results of Swab Sampling – Direct Combustion Method with Easy Wiper

Substance Name	TC Concentration [µgC]	Recovery Rate [(TOC Conc.-Blank)/Theoretical Conc.]
Blank	0	–
Tranexamic acid	207	104 %
Anhydrous caffeine	207	104 %
Isopropylantipyrine	201	101 %
Nifedipine	208	104 %
Rinderon ointment	193	96.7 %
Vaseline	207	104 %



No Peaks

Probably the most common issue with the TOC-L is when user's see no peaks during analysis or no instrument response. This problem it almost always caused by one of two things:

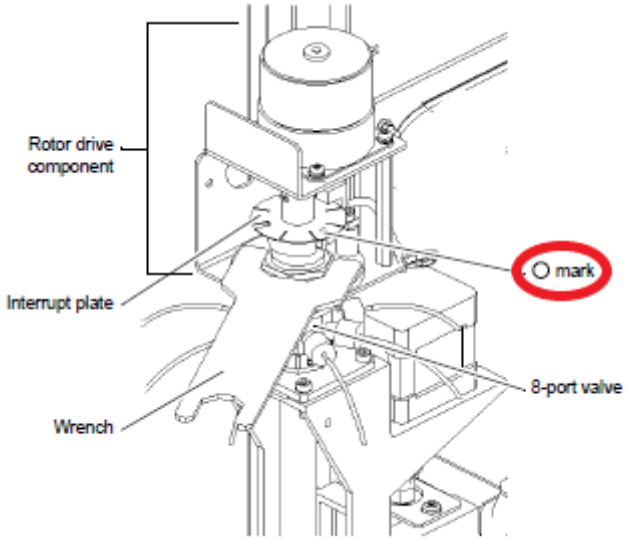
1. No sample being injected into the combustion tube

2. A Leak of carrier gas somewhere inside the instrument causing no sample to be carried to the detector.

No Peaks

To check to make sure sample is being injected into the combustion tube, start a sample analysis and open the front door of the TOC and watch what the instrument does.

Watch the 8-port valve and the syringe to make sure the instrument is pulling in sample and then injecting it into the combustion tube via port-7 of the 8-port valve. You can tell which port the valve is on by watching the “O” mark on the valve. Whichever port the “O” lines up with is the port it is currently on.

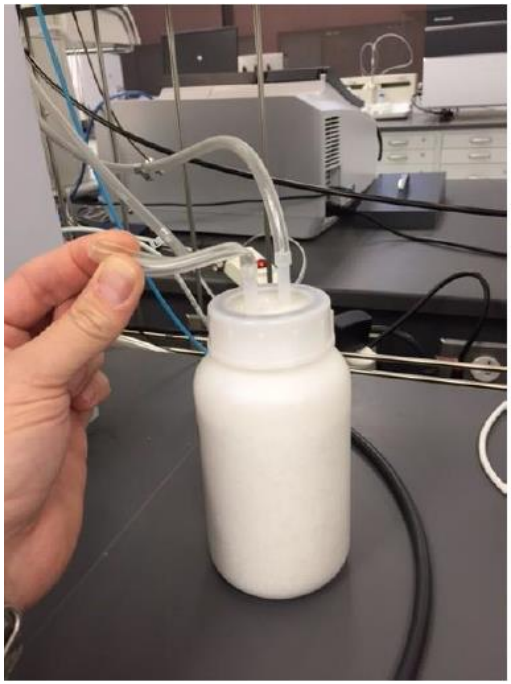


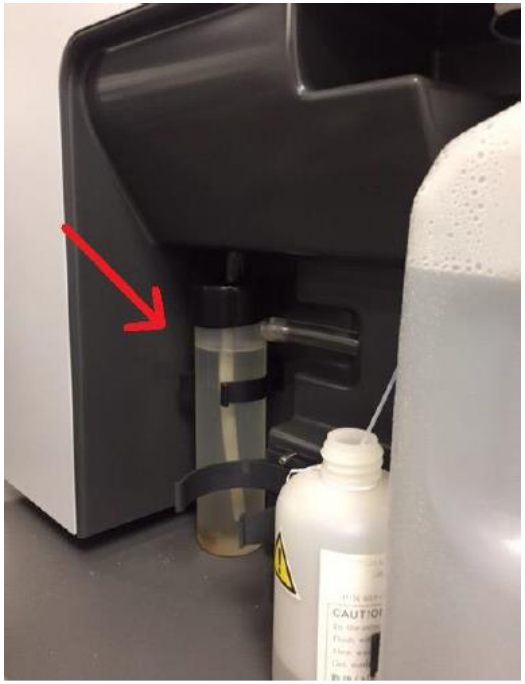
Open the lid on top and locate the top of the combustion tube that sticks up out of the furnace. Make sure that when the slider moves forward over the combustion tube you see sample being injected into the combustion tube. If no sample is being pulled up and injected into the combustion tube:

1. Check the auto-sampler tubing and make sure it is not clogged or pinched
2. Replace the ASI auto-sampler needle if it is clogged
3. Check the 8-port valve and if necessary replace the 8-port valve rotor.
4. Check the syringe and if needed replace the syringe, and/or plunger tip.

No Peaks

To perform a leak check, pinch the “L” tubing going to the CO2 scrubber blocking any flow of gas that may be going through that tube. While pinching the tube, check for bubbling in the drain pot on the left hand side of the instrument.



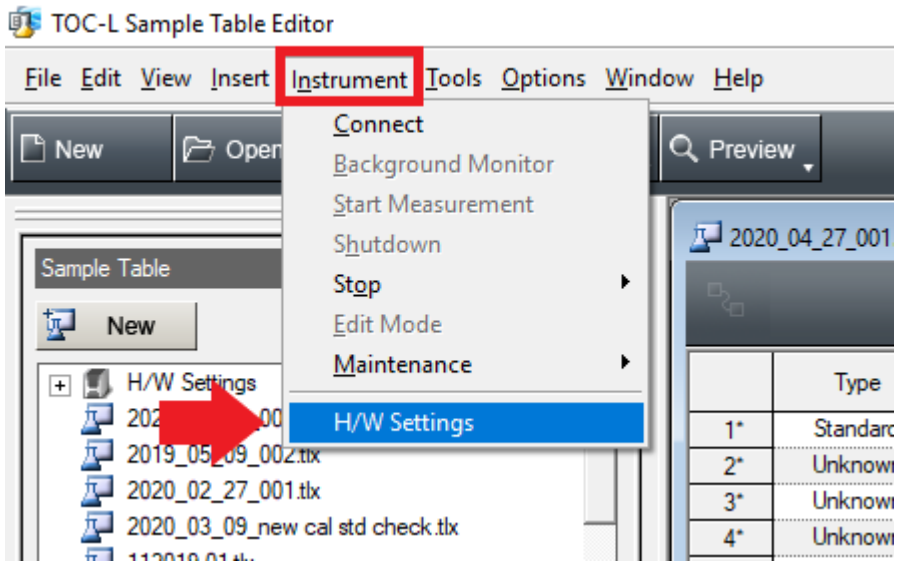


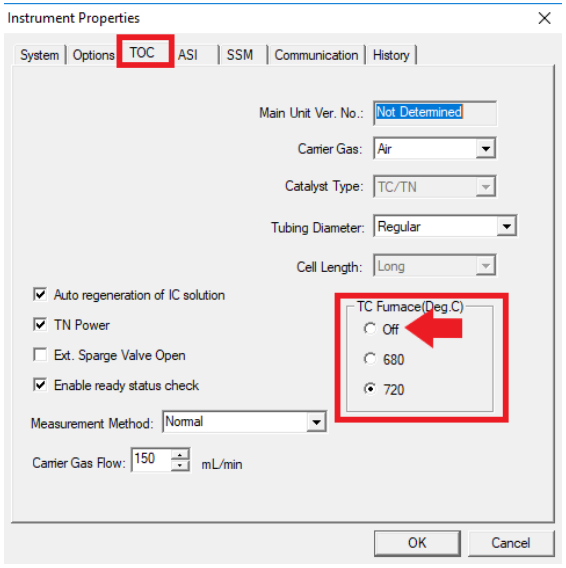
If there is **NO** bubbling in the drain pot then there is a gross leak of carrier gas somewhere in the system. If there **IS** bubbling in the drain pot then there is no leak of carrier gas in the system.

No Peaks

The most common spots for leaks of carrier gas are in or around the combustion tube. If it is determined there is a leak of carrier gas:

1. shut off the furnace and allow it to cool before proceeding.





1. Remove the combustion tube and check to make sure it is not cracked or broken and replace if necessary.

2. Replace the o-rings in the TC Injection block.

High Baseline Position

A high baseline is any baseline position above 200 mV. When the baseline is above 200 mV the instrument will give a 'NOT READY' status and will not allow a run to be started. At this point action needs to be taken to lower the baseline below 200 mV. There are several things that can cause a high baseline including:

1. A leak of carrier gas. The TOC measures CO2 so if there is a leak of carrier gas, no clean gas is flowing through the detector so it becomes filled with ambient air. The instrument will then measure the CO2 in ambient air resulting in a high baseline position.

2. A dirty detector cell. The detector cell is gold lined for maximum transmission of light to the detector. Over time the gold lining can become dirty, corroded, or even scratched, leading to a high baseline. In this scenario the baseline will usually creep up over time and when it gets to 200 mV, will no longer give the 'READY' status. Please contact Shimadzu for technical assistance.

3. Contaminated carrier gas. Carrier gas that contains high levels of CO2 or other hydrocarbons can lead to a high baseline. If using compressed cylinders, it is possible to get the occasional bad cylinder of air. If using a TOC gas generator, check the filters and scrubbers on the generator and replace if necessary.

4. A faulty detector or light source. This is the least common scenario as the detector and light source are quite robust typically lasting for ten years or more. This scenario usually results in a baseline being so high or so low that it cannot even be seen on the background monitor page. Please contact Shimadzu for technical assistance.

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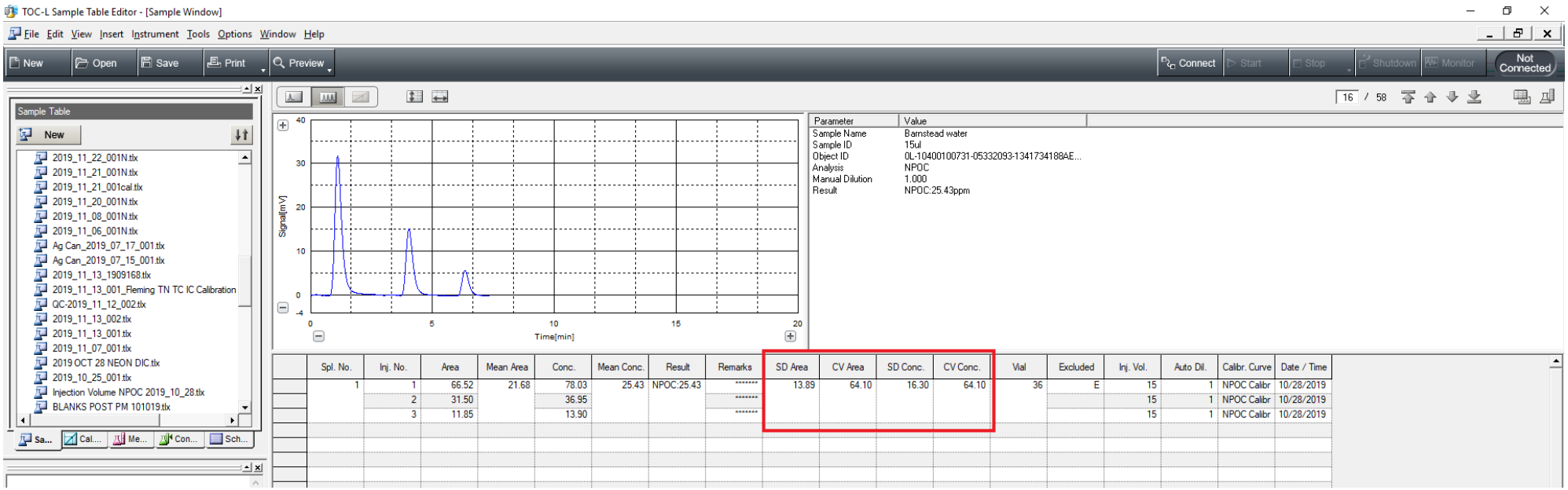
Poor Reproducibility

It is common in TOC analysis to perform multiple injections for each sample and then average those results together for the final result. Replicate injections should have low CV (RSD) values, and low SD values. When these values become large, action should be taken.



Poor Reproducibility

There are many things that can cause poor reproducibility including:

1. A worn out syringe and/or plunger tip
2. Clogged or leaking 8-port valve/rotor
3. Clogged or kinked injection line (port 7 tubing)
4. Worn out or leaking o-rings
5. Cracked or broken combustion tube
6. Poor injection into the combustion tube

Poor Recovery of Control Samples / Check Standards

Low Recovery of Control Samples or Check Standards could be caused by:

1. Bad or consumed catalyst. Remove the combustion tube and replace the catalyst.
2. Clogged ASI needle or ASI tubing. Check the needle and tubing and replace if necessary.
3. Worn out syringe or syringe plunger tip. Check and replace if necessary.
4. A leak of carrier gas. Check for a leak of carrier gas.
5. Worn out o-rings. Check and replace o-rings as needed.
6. Improperly prepped Calibration Standards. Check the calibration standards and re-prep if necessary. Then re-run the calibration curve.

Poor Recovery of Control Samples / Check Standards

High recovery of Control Samples or Check Standards is much less common than low recovery however High recovery could be caused by:

1. Instrument contamination. If instrument contamination is suspected, remove and clean these parts:
   - The ASI needle
   - The syringe
   - The 8-port valve rotor
   - The combustion tube
   - The TC injection tubing

2. Instrument Carry-over. If Carry-over between sample is suspected, increase the number of washes and/or ASI needle rinses.

3. Improperly prepped Calibration Standards. Check the calibration standards and re-prep if necessary. Then re-run the calibration curve.

High Blank Values

High blank values can be caused by many things, including:

1. Contamination in the instrument. If instrument contamination is suspected, remove and clean these parts:
   - The ASI needle
   - The syringe
   - The 8-port valve rotor
   - The combustion tube
   - The TC injection tubing

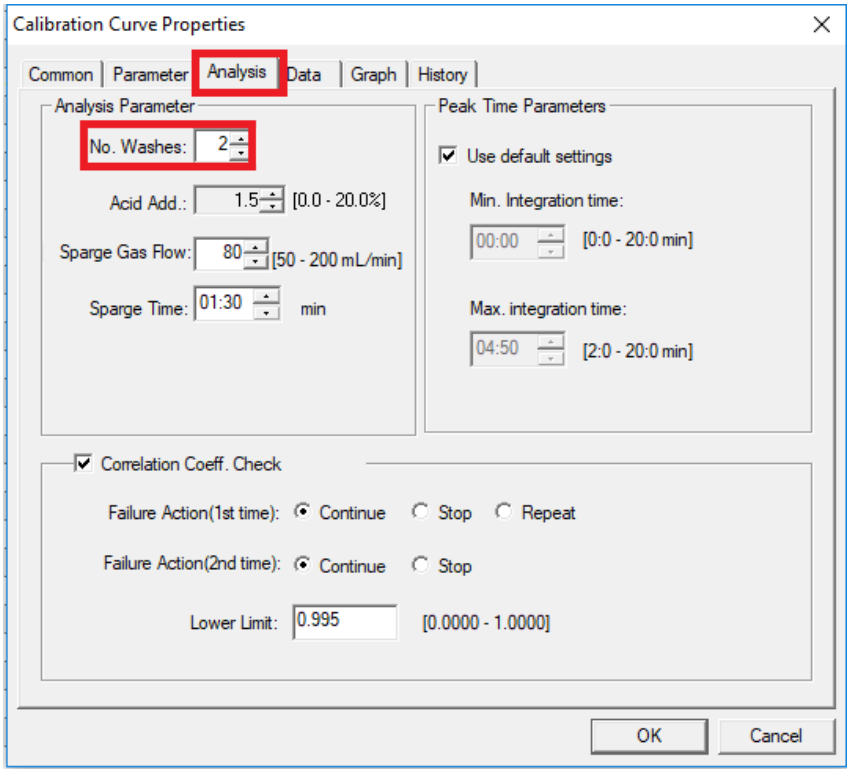
2. Contaminated DI water. Replace the dilution water bottle and ASI rinse bottle with freshly collected DI water.

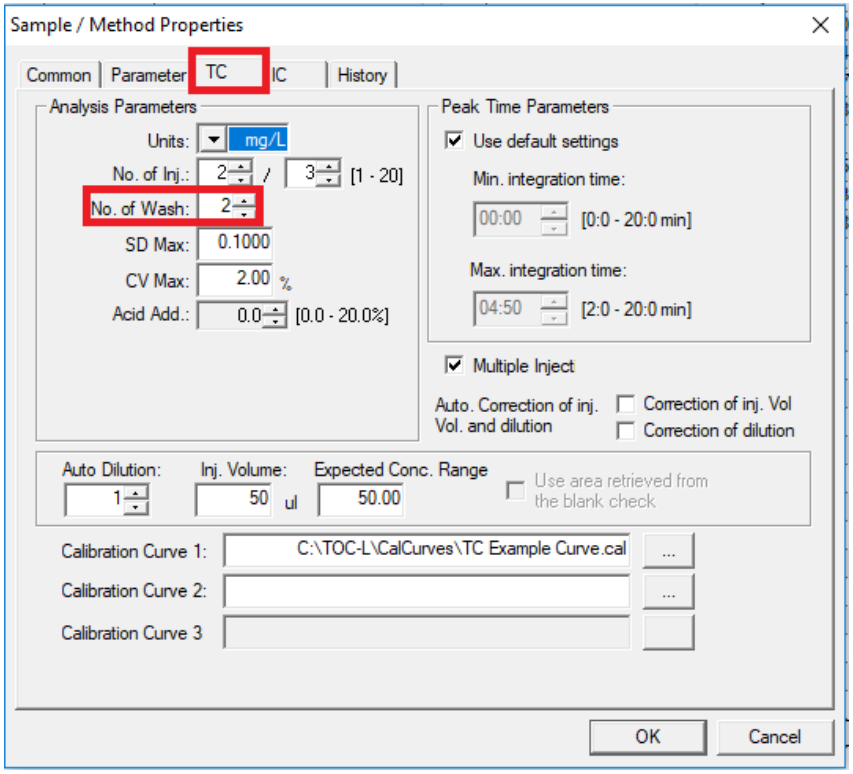
3. Contaminated acid. Replace the acid with a new or freshly prepped acid.

4. Not enough ASI needle rinses or syringe /flow line washes. Click to the next pages for instructions on where to increase these values.

High Blank Values

In the TOC-L software, ‘Washes’ refers to the number of times the instrument washes the syringe, valve, and flow lines with **SAMPLE** prior to analysis. These values can be changed by opening either an existing calibration curve file or method file.

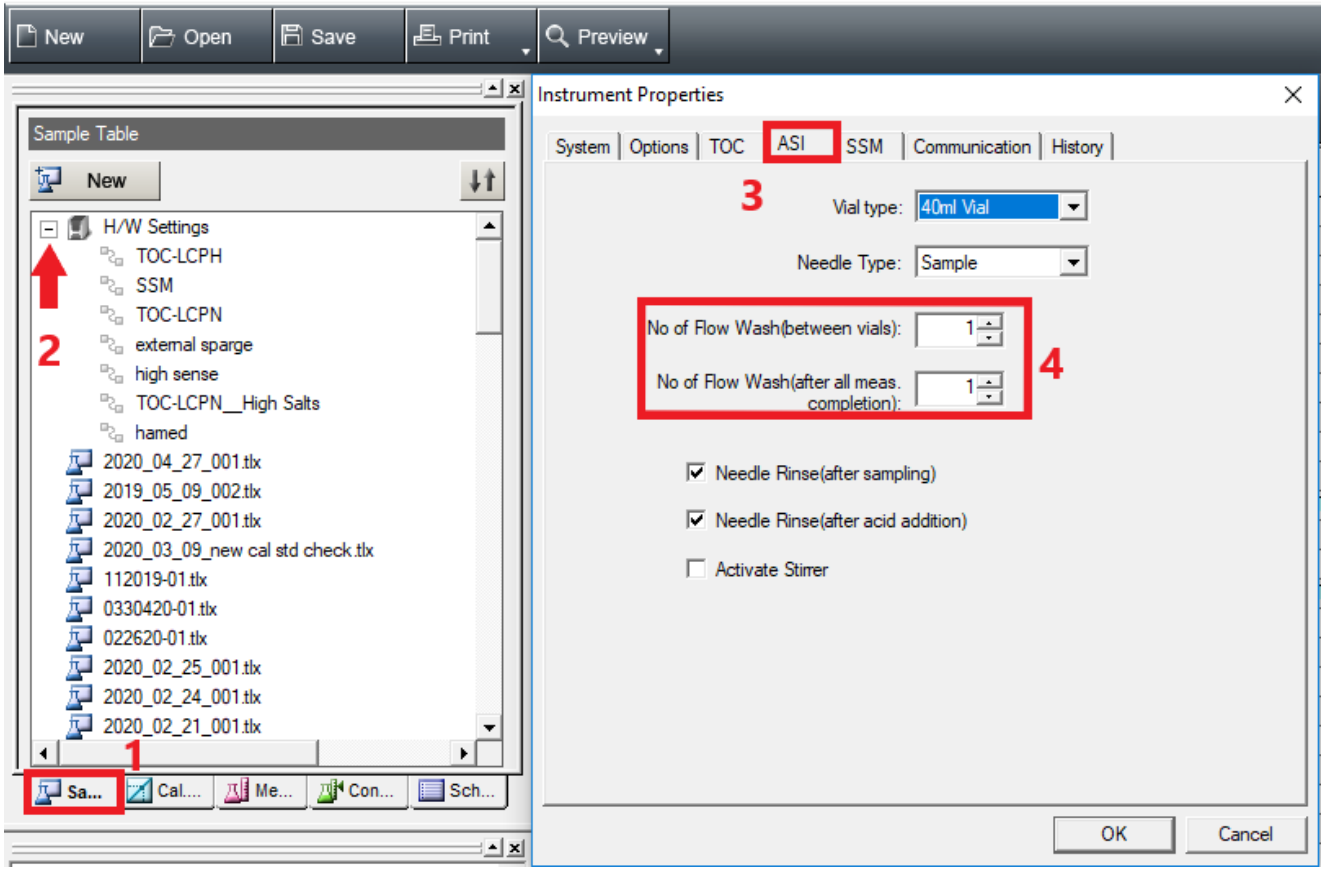




High Blank Values

In the Hardware Settings, the number of times the auto-sampler rinses the needle with DI rinse water can be set. Increase this value for really dirty samples or if needle contamination and carryover is suspected.

1. Open the list of saved sample tables.
2. Expand the list of Hardware (H/W) Settings and select the desired configuration.
3. Select the 'ASI' tab to view the auto-sampler settings.
4. Change the number of needle washes to the desired amount.



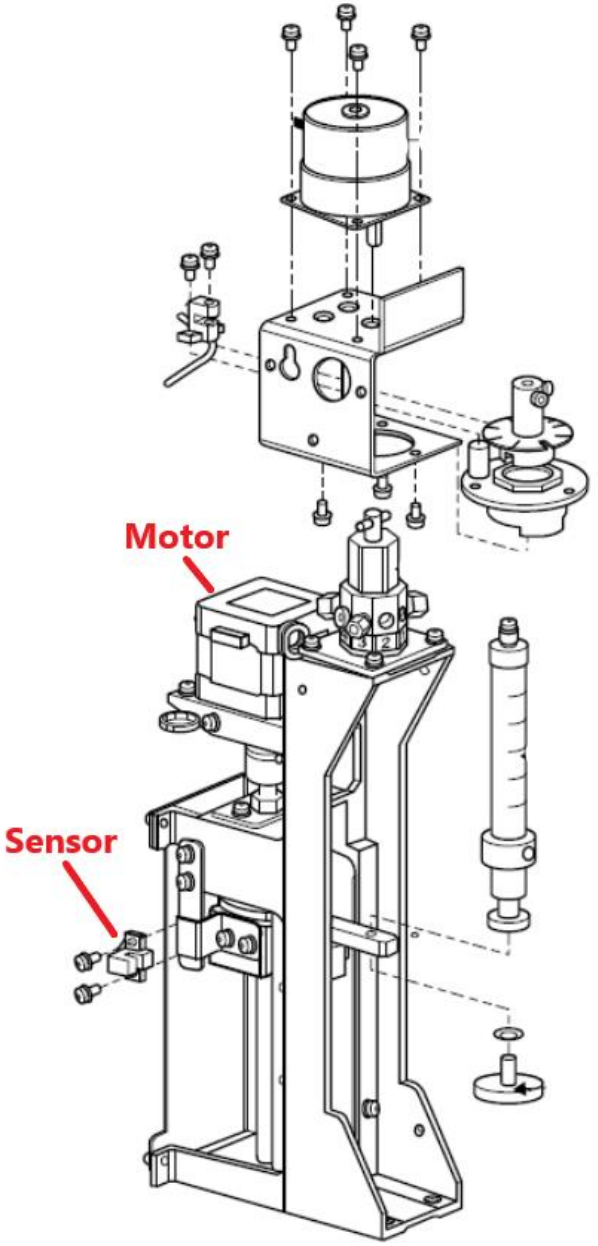
ERROR: Syringe Pulse Count / Syringe Pump

Either of these error messages signifies an issue with the syringe pump drive in the TOC.

1. Restart the instrument.

2. During the Instrument 'Initialization' open the front door and watch what the syringe drive does.
   - If the syringe never moves up or down and the same error message is generated again, it is usually due to a faulty syringe pump motor. Replace the motor.
   - If the syringe moves up and down a few times and then gives the same error message, it is usually due to a faulty photo-sensor. Replace the photo-sensor.

3. Contact Shimadzu Tech Support for current part numbers and prices for the motor an sensor.



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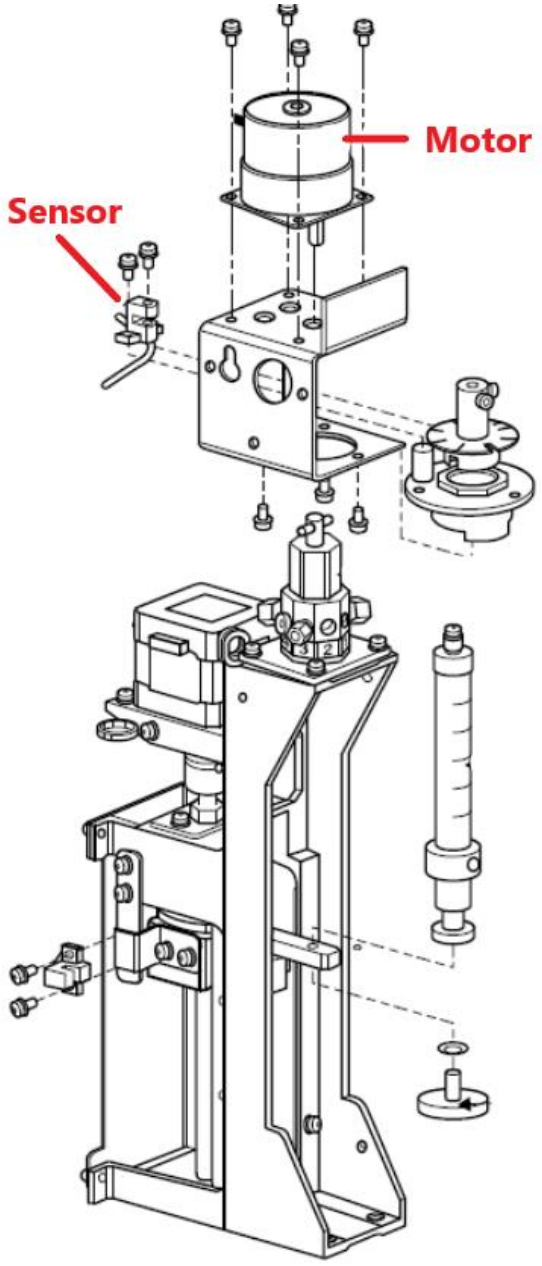
ERROR: 8port Valve Pulse Count / 8port Valve Position

Either of these error messages signifies an issue with the 8-port Valve drive in the TOC.

1. Restart the instrument.

2. During the Instrument 'Initialization' open the front door and watch what the 8-port valve does.
   - If the valve never rotates and the same error message is generated again, it is usually due to a faulty 8-port valve motor. Replace the motor.
   - If the valve rotates a few times and then gives the same error message, it is usually due to a faulty photo-sensor. Replace the photo-sensor

3. Contact Shimadzu Tech Support for current part numbers and prices for the motor an sensor.



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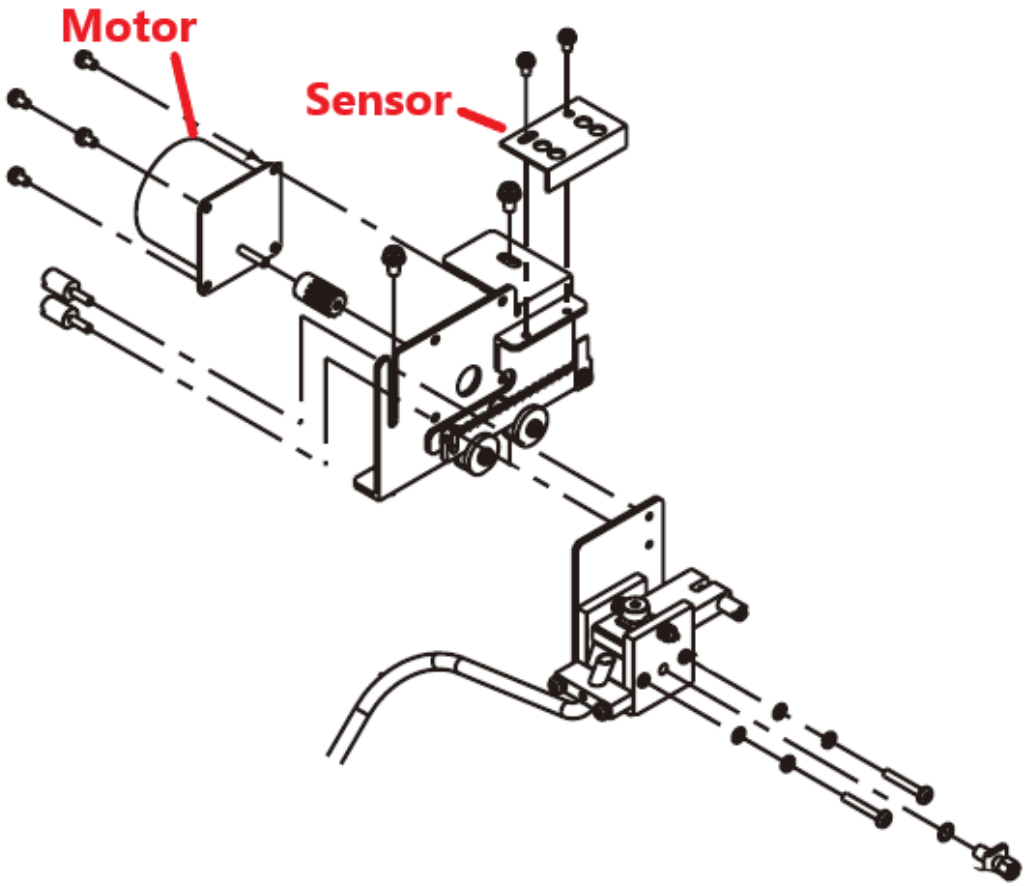
ERROR: TC / IC Slider Position

Either of these error messages signifies an issue with the TC or IC slider drive mechanism.

1. Restart the instrument.

2. During the Instrument 'Initialization' open the front door and watch what the slider does.
   - If the slider never moves forward or backward and the same error message is generated again, it is usually due to a faulty slider motor. Replace the motor.
   - If the slider moves forward and backward a few times and then gives the same error message, it is usually due to a faulty photo-sensor. Replace the sensor.

3. Contact Shimadzu Tech Support for current part numbers and prices for the motor an sensor.



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ERROR: ASI Arm Vertical Pulse Count / ASI Arm Vertical Position

Either of these error messages signifies an issue with the ASI arm in the auto-sampler in relation to the up and down movement.

1. Restart the instrument

2. Start an analysis and watch the ASI auto-sampler arm.
   - If the arm never moves up or down and the same error message is generated again, it is usually due to a faulty ASI motor.
   - If the arm moves up and down a few times and then gives the same error message, it is usually due to a faulty photo-sensor.

3. Contact Shimadzu Service. Removing the covers to get down to the motor drives in the ASI is difficult and requires a trained Shimadzu Service Engineer.

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ERROR: ASI Arm Horizontal Pulse Count / ASI Arm Horizontal Position

Either of these error messages signifies an issue with the ASI arm in the auto-sampler in relation to the forward and backward movement.

1. Restart the instrument

2. Start an analysis and watch the ASI auto-sampler arm.
   - If the arm never moves forward or backward and the same error message is generated again, it is usually due to a faulty ASI motor.
   - If the arm moves forward and backward a few times and then gives the same error message, it is usually due to a faulty photo-sensor.

3. Contact Shimadzu Service. Removing the covers to get down to the motor drives in the ASI is difficult and requires a trained Shimadzu Service Engineer.

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ERROR: ASI Tray Pulse Count / ASI Tray Position

Either of these error messages signifies an issue with the sample tray in the auto-sampler.

1. Restart the instrument

2. Start an analysis and watch the ASI auto-sampler tray.
   - If the tray never rotates and the same error message is generated again, it is usually due to a faulty ASI motor.
   - If the rotates around a few times and then gives the same error message, it is usually due to a faulty photo-sensor.

3. Contact Shimadzu Service. Removing the covers to get down to the motor drives in the ASI is difficult and requires a trained Shimadzu Service Engineer.

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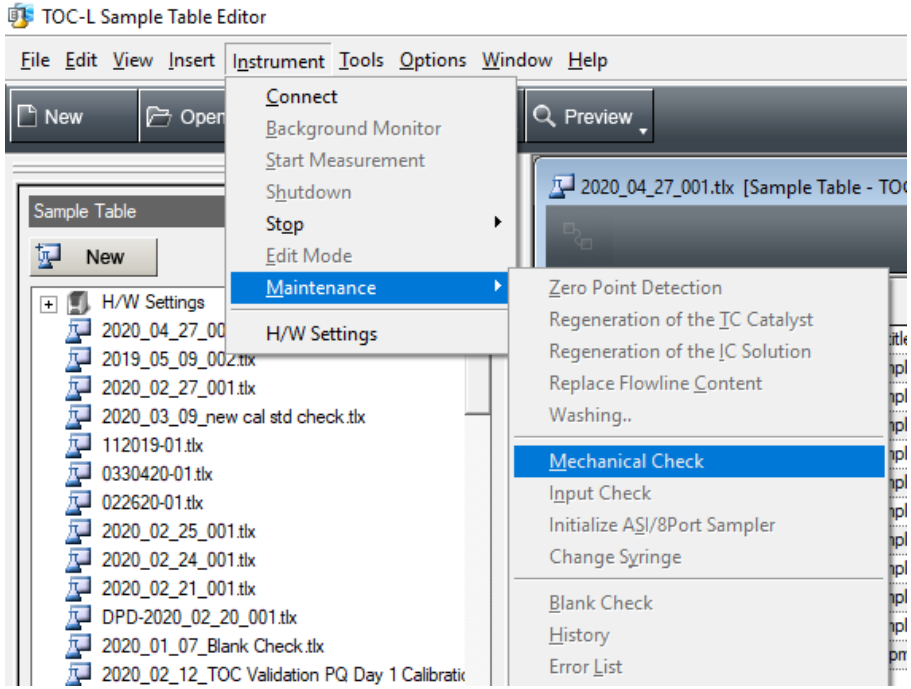
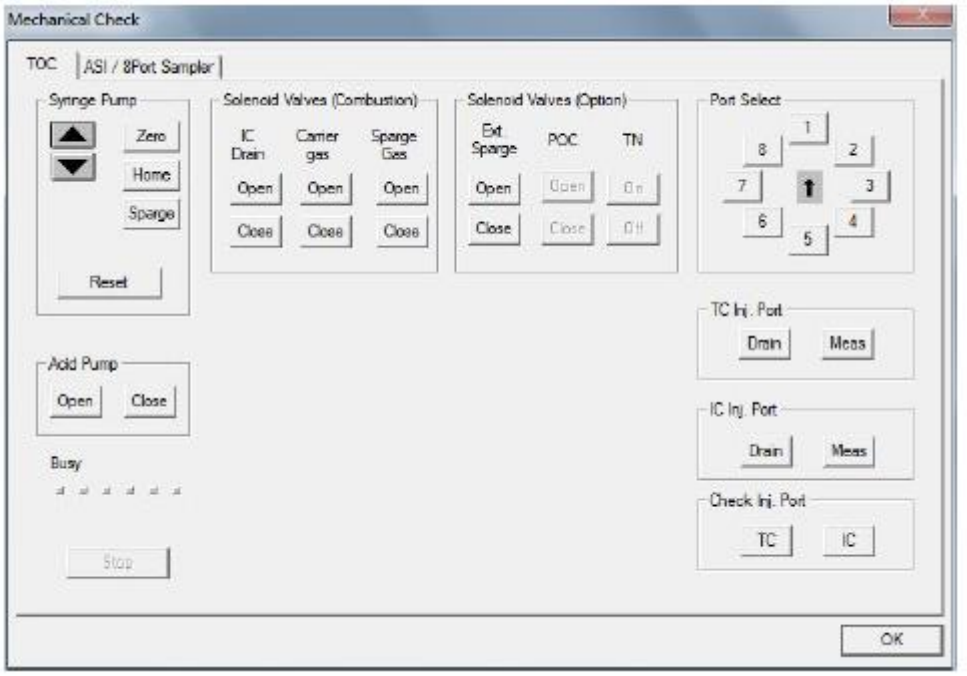
Mechanical Check

Mechanical check allows users to check all the mechanical functions of the TOC-L. It can be very useful for troubleshooting and diagnosing mechanical issues. To access the mechanical check, select 'Instrument' > 'Maintenance' > 'Mechanical Check'.





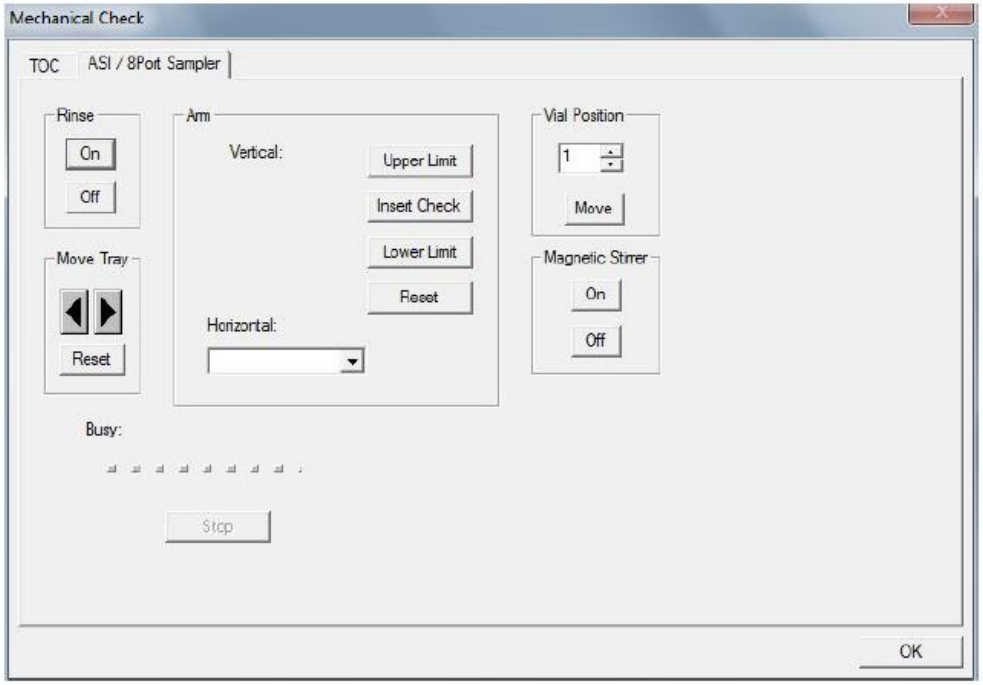
Mechanical Check

Item	Function
Syringe Pump	Click the buttons to move the syringe pump to the Zero, Home and Sparge positions. Click the Reset button to move the plunger to the highest position.
Solenoid Valves (Combustion and Option)	Use the Open and Close buttons to open and close the following solenoid valves. • IC Drain • Carrier gas • Sparge Gas • POC
Port Select	Click a numbered button to open the flow line between the syringe and the injection port. The numbered buttons correspond to the port numbers of the 8-port valve.
Acid Pump	Click Open to turn the IC reagent delivery pump on, and Close to turn it off.
TC Inj. Port	Click Drain or Meas to move the TC slider to the drain or injection position.
IC Inj. Port	Click Drain or Meas to move the IC slider to the drain or injection position.

Mechanical Check

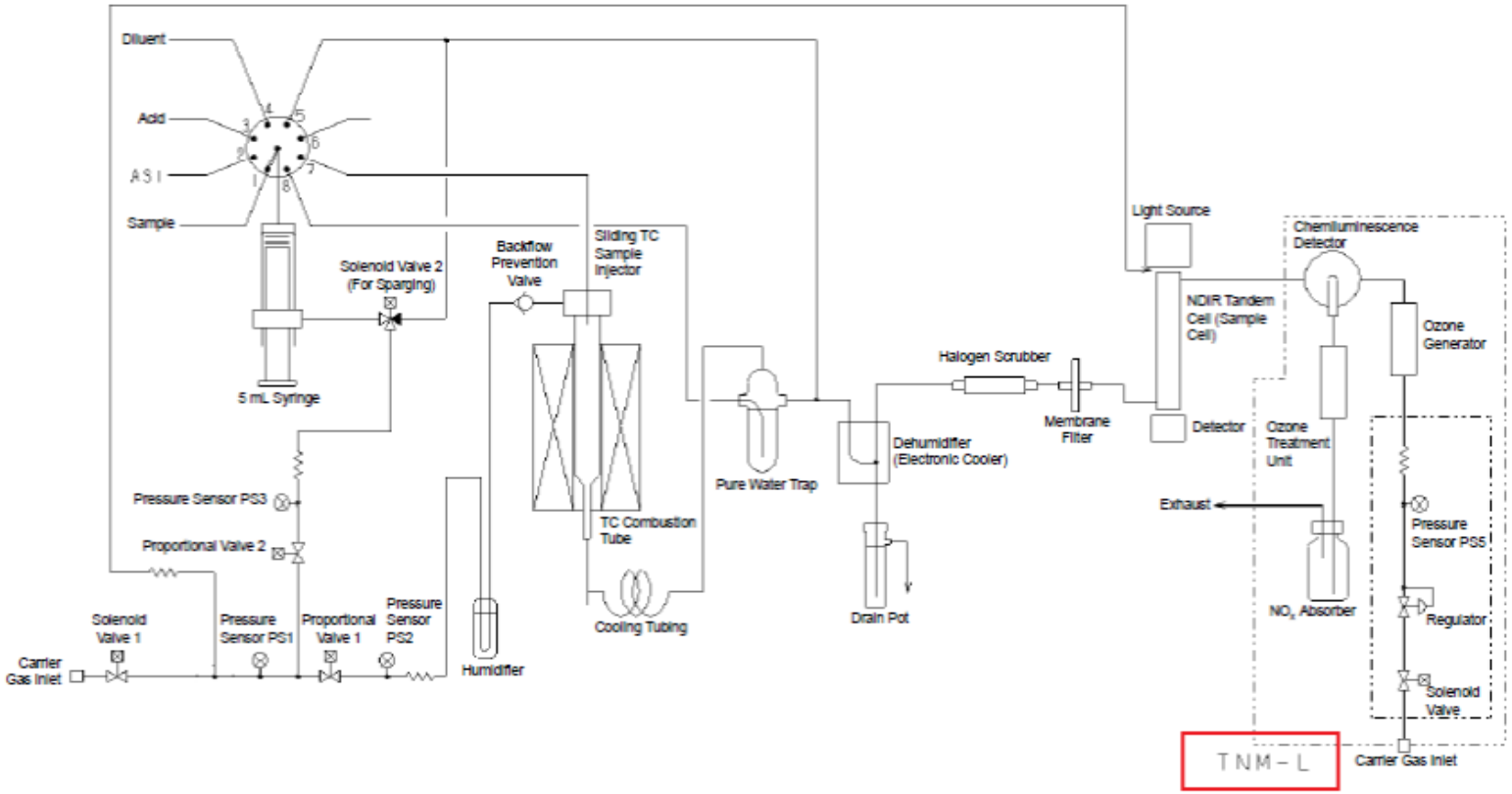
ASI/8 Port Sampler Tab

The enabled items will vary depending on the optional accessories connected.



Item	Function
Rinse	Click On and Off to start and stop the ASI-L flow line rinsing operation.
Move Tray	Click the left and right buttons to move the ASI-L vial rack left and right. Click Reset to return the rack to the home position.
Arm	Click the up and down buttons to move the arm up and down. Click Reset to return the arm to the home position. Use the Horizontal drop-down box to move the arm horizontally to the Rinse, Needle Change Pos, Drain Port or Vial 1st (to 5th) Line positions.
Vial Position	Enter a vial position number, and then click the Move button to move the arm to the specified position.
Magnetic Stirrer	Click On and Off to start and stop the magnetic stirrer operation.

Troubleshooting TNM-L Issues



Flow Diagram TOC-LCPN + TNM-L

Section 1 / 5

Troubleshooting TNM-L Issues

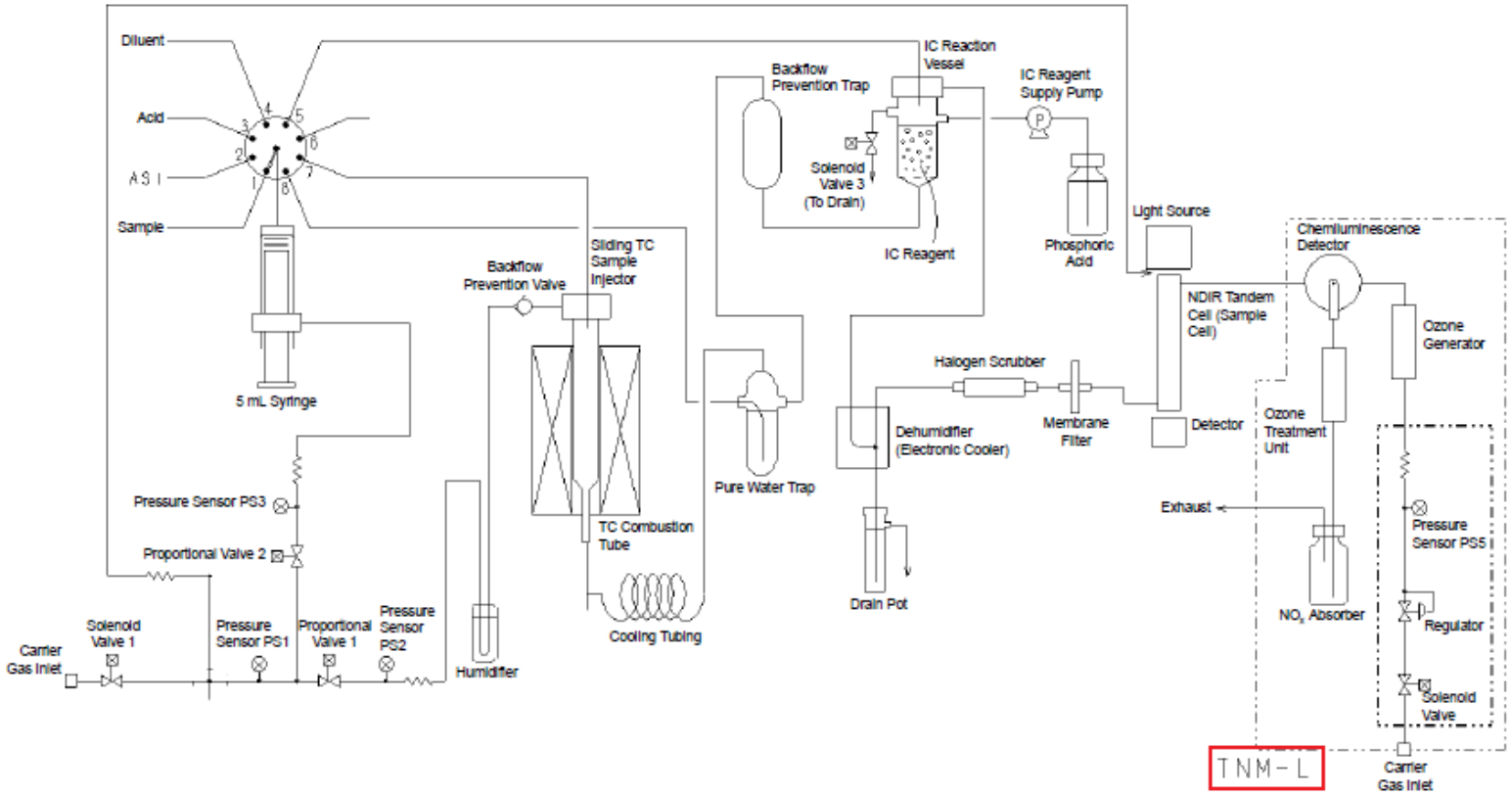


Figure 2.8 Flow Diagram TOC-LCPH + TNM-L

Troubleshooting TNM-L Issues

- **The TNM-L is downstream of the TOC-L. Sample passes through the TOC-L where carbon Is measured and then enters the TNM-L where nitrogen is measured.**

- **When troubles arise with nitrogen measurement such as no peaks or poor reproducibility it is first a good idea to check the carbon measurement.**

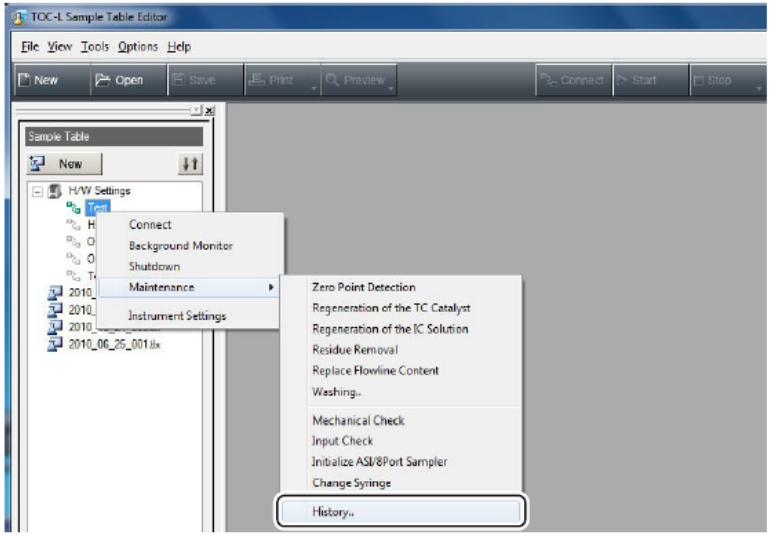
  - If carbon measurement is normal and looks good then the issue with the TNM-L measurement lies within the TNM-L so troubleshoot the TNM-L

  - If carbon measurement is not good then the issue with TNM-L measurement is likely a result of something within the TOC-L so troubleshoot the TOC-L

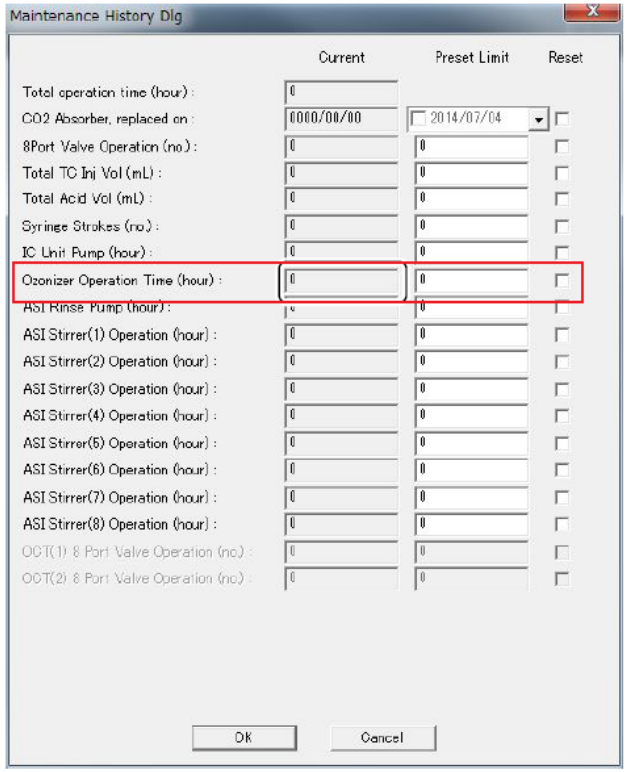
Troubleshooting TNM-L Issues

- **No peaks during for nitrogen**
  - Make sure air is being supplied to the TNM-L unit at 200 kPa
  - Make sure there is no leak of gas within the TNM-L
  - Check the hours on the Ozone Generator. The ozone generator does have a lifespan and if no ozone is being generated then there will be no peaks for nitrogen measurement. Replacement of ozone generator is performed by Shimadzu Service Engineer. Please contact your local Shimadzu Office.

Open the Sample Table tab of the file viewer. Right-click on the connected system configuration icon, and click Maintenance – History.



The Maintenance History Dlg window is displayed.

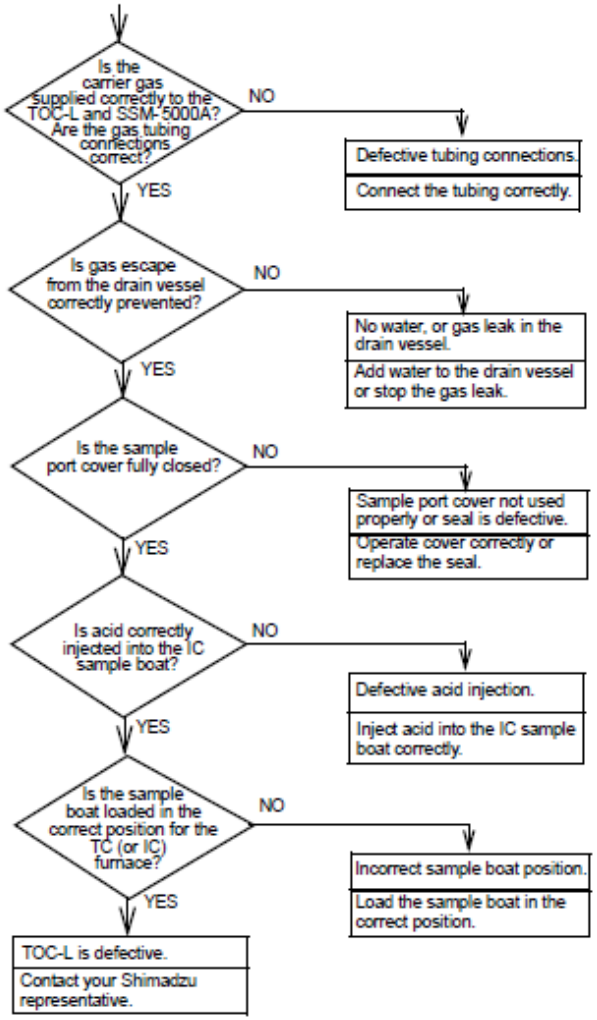


Troubleshooting TNM-L Issues

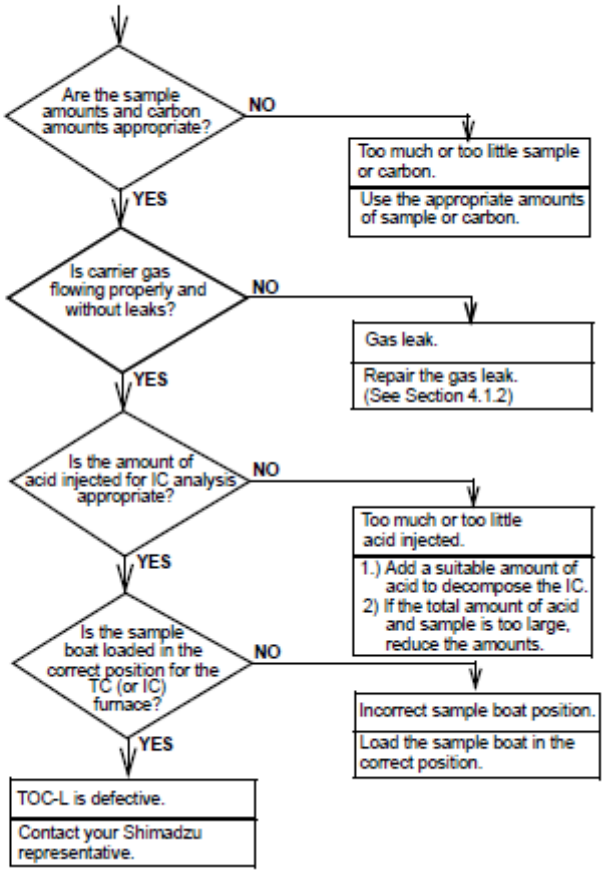
- **Poor reproducibility for nitrogen measurement**
  - Check for a leak of carrier gas within the TNM-L unit
  - Check black tubing that connects the detector of the TOC-L to the reactor on the TNM-L and make sure the connection is good at the TNM-L reactor.
  - Pull apart the TNM-L reactor assembly and inspect and/or replace the detector gasket.
    - See Replacing the TNM-L Detector Gasket

Troubleshooting SSM Issues

No Peaks Generated During Standard Measurement

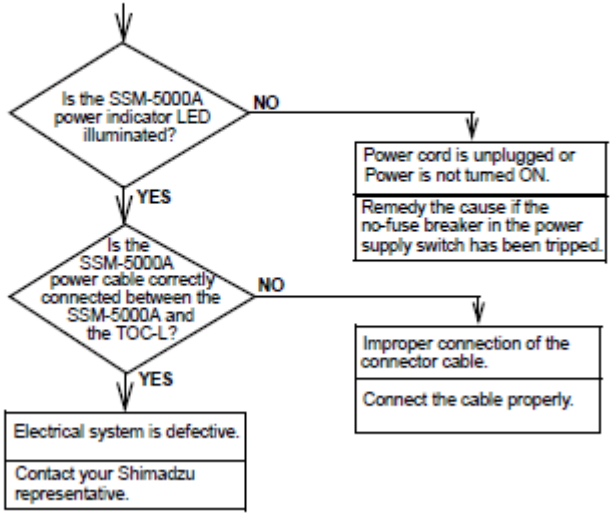


Repeatability is Poor Even for Standard Measurement

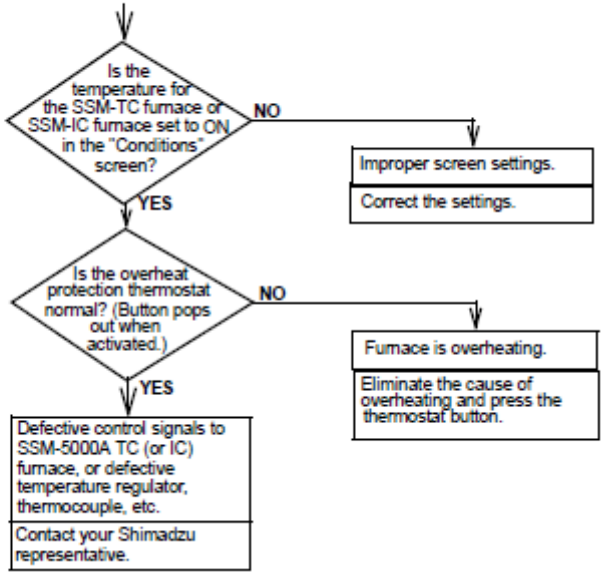


Troubleshooting SSM Issues

SSM-5000A Does Not Operate



TC or IC Furnace Does Not Reach Required Temperature



Principles of Analysis

Principles of Analysis

Two types of carbon are present in water: organic carbon and inorganic carbon. Organic carbon (TOC) bonds with hydrogen or oxygen to form organic compounds. Inorganic carbon (IC or TIC) is the structural basis for inorganic compounds such as gas carbonates and carbonate ions. Collectively the two forms of carbon are referred to as total carbon (TC) and the relationship between them is expressed TOC=TC-IC.

Nitrogen is also present in water in two types: organic and inorganic. The sum of these is referred to as total nitrogen (TN).

The principles underlying TC and TN analysis are explained in the following sections.

Principles of Analysis

Principles of TC (Total Carbon) Analysis

Sample is introduced into the TC combustion tube, which is filled with an oxidation catalyst and heated to 680°C. The sample is burned in the combustion tube and, as a result, the TC components in the sample are converted to carbon dioxide. Carrier gas, which flows at a rate of 150 mL/min to the combustion tube, carries the sample combustion products from the combustion tube to an electronic dehumidifier, where the gas is cooled and dehydrated. The gas then carries the sample combustion products through a halogen scrubber to remove chlorine and other halogens. Finally, the carrier gas delivers the sample combustion products to the cell of a non-dispersive infrared (NDIR) gas analyzer, where the carbon dioxide is detected. The NDIR outputs an analog detection signal that forms a peak; the peak area is measured by the TOC-Control L software.

The peak area is proportional to the TC concentration of the sample. A calibration curve equation that mathematically expresses the relationship between peak area and TC concentration can be generated by analyzing various concentrations of a TC standard solution. The TC concentration in a sample can be determined by analyzing the sample to obtain the peak area and then using the peak area in the calibration curve equation.

Principles of Analysis

Principles of IC (Inorganic Carbon) Analysis

Two methods for measuring IC using the TOC-L are available: analysis within the injection syringe and analysis using the optional IC reactor. In both methods, the measured IC consists of carbon derived from carbonates, hydrogen carbonates and dissolved carbon dioxide.

Defining IC

The IC measured by TOC analysis consists of the carbon contained in carbonates and in carbon dioxide dissolved in water. By acidifying the sample with a small amount of hydrochloric acid to obtain a pH less than 3, all carbonates are converted to carbon dioxide (CO_2) by the following reactions:

$$Me_2CO_3 + 2HCl \rightarrow CO_2 + 2MeCl + H_2O$$

$$MeHCO_3 + HCl \rightarrow CO_2 + MeCl + H_2O$$

Carbon dioxide and dissolved carbon dioxide in the sample are volatilized by bubbling (sparging) air or nitrogen gas that does not contain carbon dioxide through the sample.

Analysis Using the IC Reaction Vessel (H Type Instrument)

The TOC-L IC reactor kit is used to sparge the IC reaction solution (acidified reaction liquid) with carrier gas. Sample is injected into the IC reaction vessel and the IC in the sample is converted to carbon dioxide, which is volatilized by the sparging process and detected by the NDIR.

Analysis Within the Syringe (N Type Instrument)

The sample is acidified to pH 3 or lower in the syringe, using hydrochloric acid. The sample is sparged with carrier gas and the IC in the sample is converted to carbon dioxide and detected by the NDIR.

Principles of Analysis

Principles of NPOC (Non-Purgeable Organic Carbon) Analysis

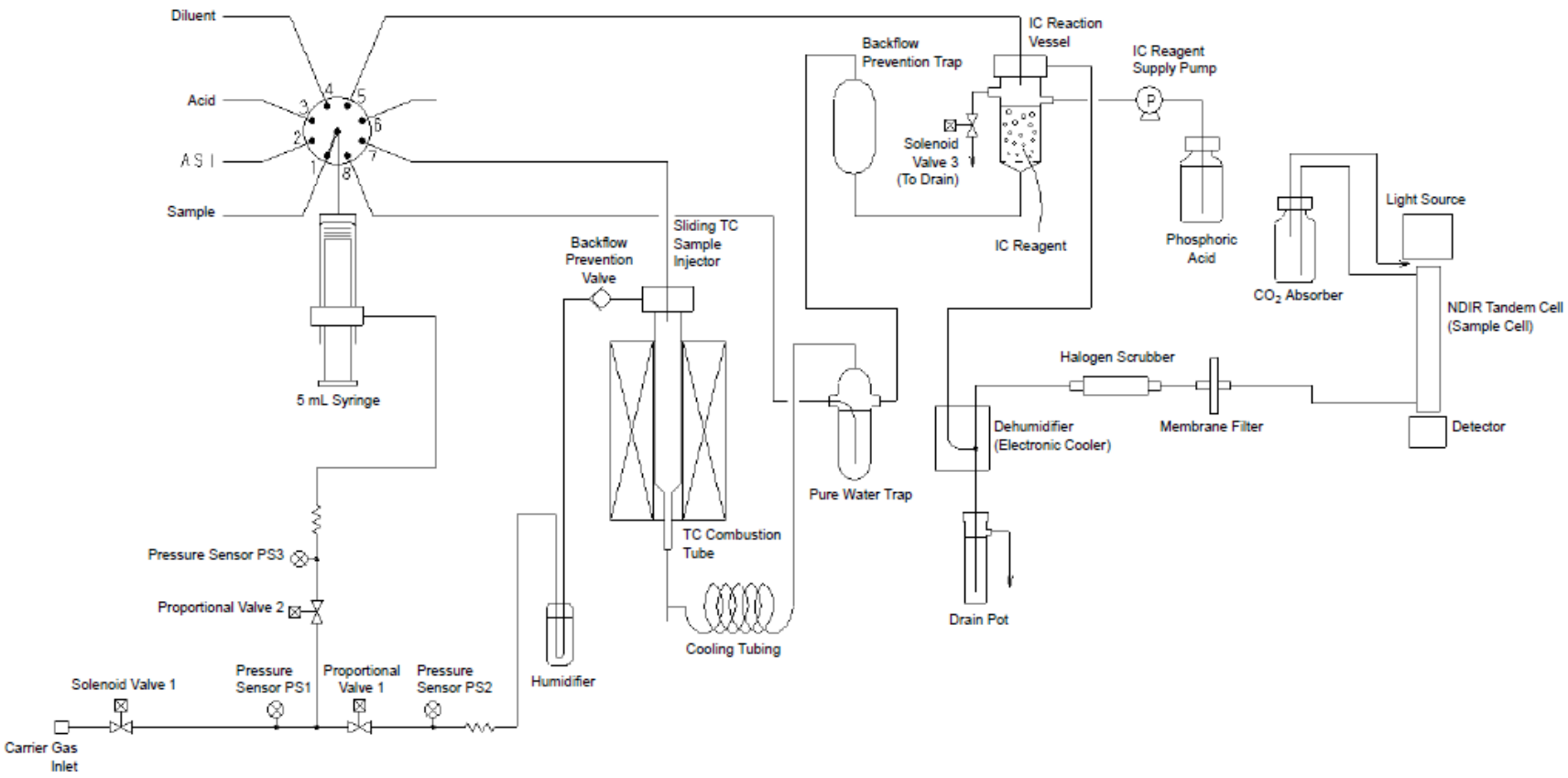
After acidifying the sample to pH 2 to 3, sparge gas is bubbled through the sample to eliminate the IC component. The remaining TC is measured to determine total organic carbon, and the result is generally referred to as TOC. However, in the TOC-L, this analysis value is referred to NPOC to distinguish it from the TOC value obtained by calculating the difference between TC and IC. NPOC stands for non-purgeable organic carbon and refers to organic carbon that is present in a sample in a non-volatile form.

NPOC and TOC (obtained by IC elimination) described in the TOC-related standard methods and referred to in water quality-related test methods (JIS, ASTM, EPA, EN) are identical. Purgeable organic substances in the sample can be lost during the sparging process. Consequently, when the sample contains purgeable organic substances, TOC should not be measured by the NPOC method. If the dissolved purgeable organic component in the water sample is large, the amount volatilized during sparging is relatively small. Generally, the amount of purgeable organic substances in natural environmental, public and purified water is small; as a result, NPOC can be referred to as TOC.

Table 8.1 • Residual Rate of Volatile Organic Substances using Sparging (Nitrogen Gas)

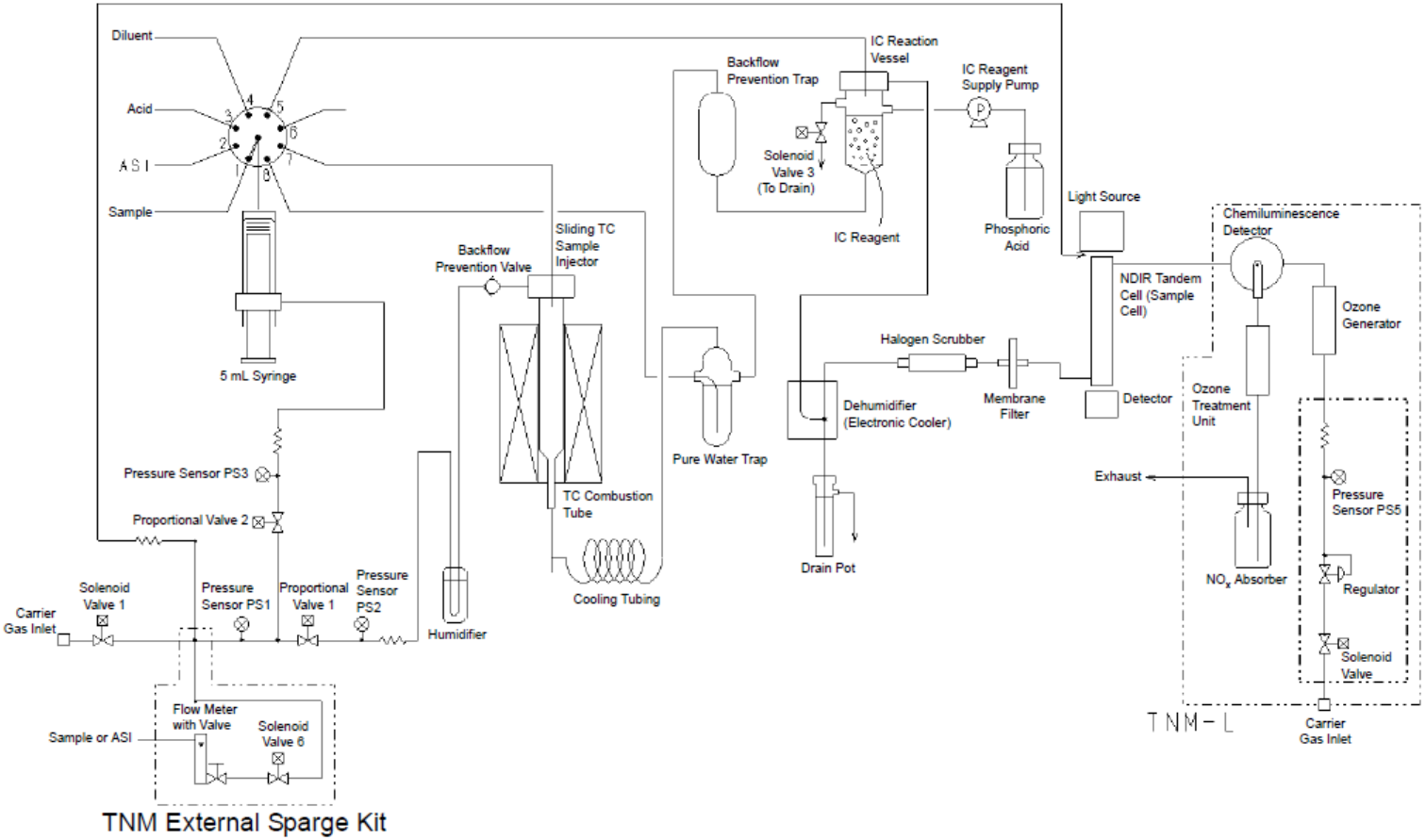
Organic Substance	Pre-Sparging Conc. (ppm)	Post-Sparging Conc. (ppm)	Residual Rate (%)
Methanol	117. 5	116	98.6
Ethanol	106.5	105	98.5
Isopropyl alcohol	129	127	98.5
n-butyl alcohol	117	115	98.3
Acetone	106	101	95.3
Acetaldehyde	130	117	90.0
Ethyl acetate	102	88	86.3
Tyrosine	117	116.5	99.5
Benzene	85	2.5	2.9
Cyclohexane	79	2	2.5

TOC-LC*H



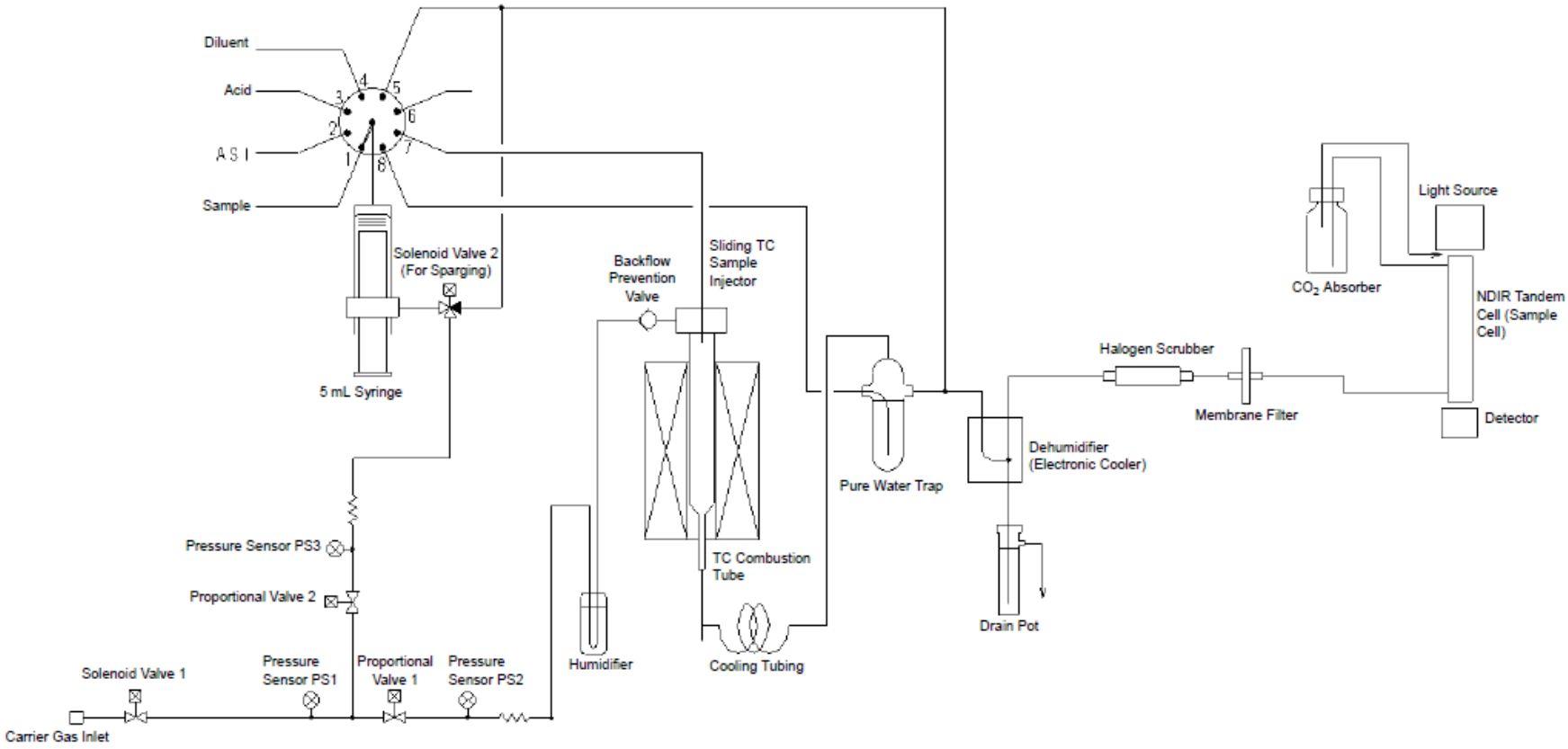
Flow Diagram (TOC-LCPH)

TOC-LC*H + TNM-L + Ext. Sparge Kit



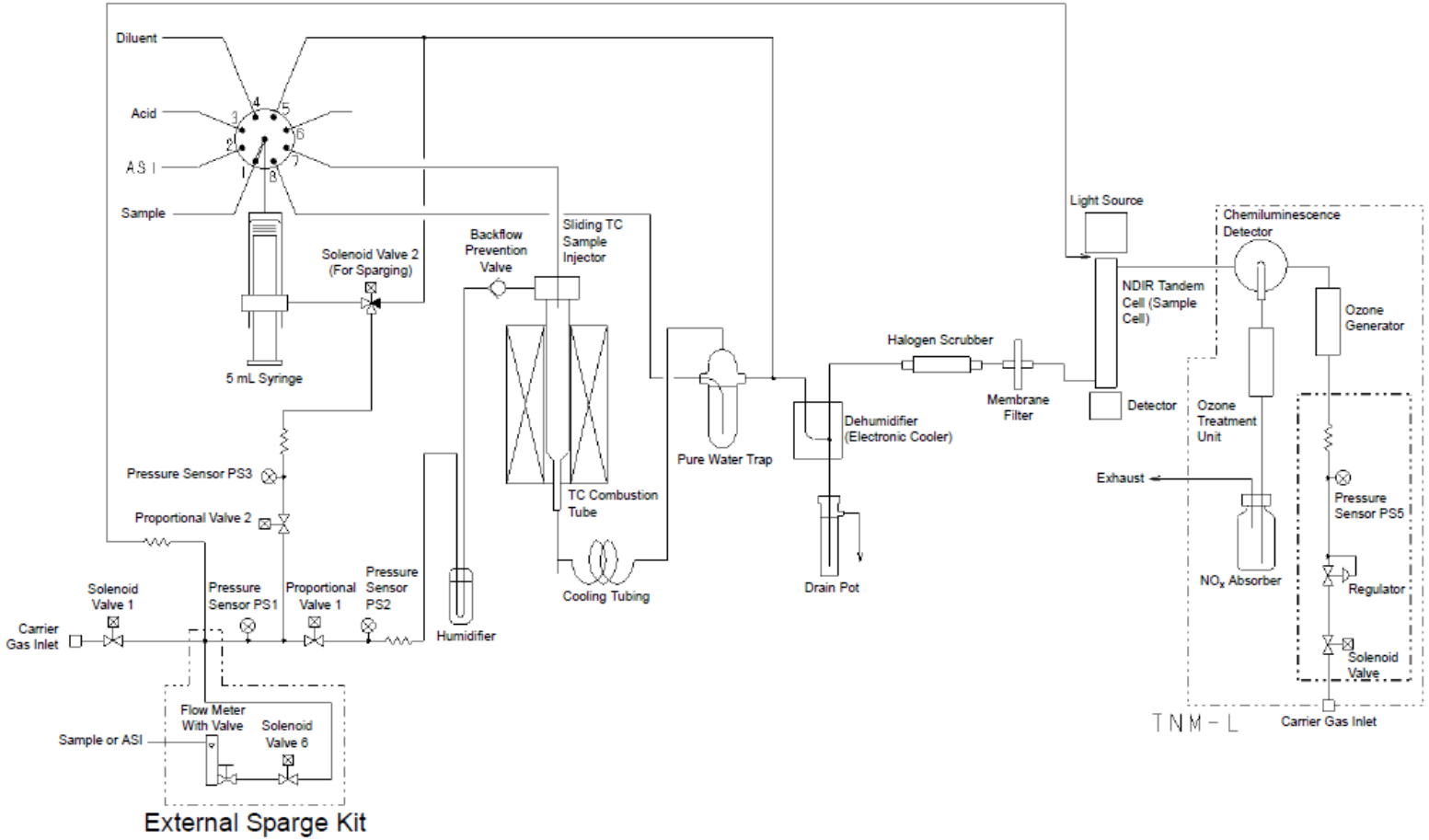
Flow Diagram (TOC-LCPH + TNM-L + External Sparge Kit)

TOC-LC*N



Flow Diagram (TOC-LCPN)

TOC-LC*N + TNM-L + Ext. Sparge Kit



Flow Diagram (TOC-LCPN + TNM-L + External Sparge Kit)

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Preparation of Standard Solutions

Preparation of Standard Solutions

Zero water refers to a standard solution with zero concentration of TC, IC, and TN, or the water used to prepare these standard solutions of various concentrations. Although zero water is absolutely free of carbon (TC) and nitrogen (TN) in theory, in practice, this type of water is difficult to attain. Even ultra-pure water attained using a high performance membrane technique or water that has been repeatedly distilled will have a TC content of about 10 µg/L, which will increase as the water moved and stored in a container. Carbon dioxide is present in the atmosphere at concentrations ranging from 300 to 500 mg/L. The carbon dioxide content of ambient air is even higher in a room with many people or where combustion equipment is used. Carbon dioxide in ambient air dissolves in water, taking the form of IC. The dissolved amount is generally about 0.2 mg/L, but depends on the water temperature and carbon dioxide content of the air in contact with the water

Not all water distillation and purification instruments incorporate mechanisms that prevent contact with air during water uptake, retention, or storage processes. For this reason, IC is present in most types of distilled and purified water. In some cases, the TC content of commercially available distilled water stored in plastic or glass containers is near 1 mg/L.

Note: The degree of purity required for zero water varies depending on the measurement range. For example, commercially available purified water is acceptable when measuring samples with TC concentrations of a few hundred mg/L. The quality of the zero water should be considered along with the measurement range.

Storage of Standards

Storage of Standard Solutions

The standard solutions undergo concentration changes, particularly when low-concentration solutions are stored even for short periods. As a result, high-concentration standard stock solutions (for example, 1000 mgC/L) should be stored in airtight containers in a cool, dark place. Glass bottles are suitable storage containers. Dilute the stock solution prior to each use.

Storage Time

The limitation on storage of standard solutions is about 2 months for 1000 mgC/L standard stock solutions and about 1 week for diluted standard solutions (for example, 100 mgC/L). The limitations are for cold storage in sealed containers.

Note: IC standard solution absorbs atmospheric carbon dioxide and undergoes concentration changes. As a result, it is particularly important to store IC standard solution in a sealed container.

Prepare fresh standard solutions:

- If reproducibility of analysis values is poor or concentration fluctuates.
- If contaminants, including even small amounts of dust, are present in the standard solution.

TC/NPOC Standard Prep

Preparation of TC Standard Solutions

1. Accurately weigh 2.125 g of reagent grade potassium hydrogen phthalate.
2. Transfer the weighed amount into a 1 L volumetric flask.
3. Add zero water to the 1 L mark, and stir the solution.

   The carbon concentration of the solution is 1000 mgC/L (1000 mgC/L = 1000 ppmC). This solution is the standard stock solution.

   Note:
   - The standard stock solution may be prepared at other concentrations.
   - Although this example describes the preparation of a 1000 mgC/L standard stock solution, the standard stock solution may be prepared at other concentrations within the range of solubility.

4. Accurately dilute this standard stock solution with zero water to prepare standard solutions at the required concentrations.

   Note:
   - The TC standard solution reagent need not be potassium hydrogen phthalate. Other substances (such as sucrose) are also acceptable depending on the application.
   - Although this example describes the preparation of a 1000 mgC/L standard stock solution, the standard stock solution may be prepared at other concentrations within the range of solubility.
   - A variety of official drying methods for standard substances are defined. Use a suitable method for drying, if required.

IC Standard Prep

Preparation of the IC Standard Solution

1. Accurately weigh 3.497 g of reagent grade sodium hydrogen carbonate and 4.412 g of sodium carbonate.
2. Transfer the weighed amount into a 1 L volumetric flask.
3. Add zero water up to the 1 L marker and mix well.

   The carbon concentration of this liquid solution is equivalent to 1000 mgC/L (1000 mgC/L = 1000 ppmC). Keep this solution as a standard stock solution.

The following procedure is the same as for preparing TC standard solution.

TN Standard Prep

Preparation of TN Standard Solution

1. Accurately weigh 7.219 g of reagent grade potassium nitrate.
2. Transfer the weighed material to a 1 L volumetric flask.
3. Add zero water up to the 1 L marker and mix well.

   The nitrogen concentration of this liquid solution is equivalent to 1000 mgN/L (1000 mgN/L = 1000 ppmN). Keep this solution as a standard stock solution.

The following procedure is the same as for preparing TC standard solution.

TC/TN Mixed Standard Prep

Preparation of 100 mgC/L TC - 100 mgN/L TN Mixed Standard Solution

1. Prepare 1 L of 1000 mgC/L TC standard solution using the preparation method described above.
2. Prepare 1 L of 1000 mgN/L TN standard solution using the preparation method described above.
3. Introduce 100 mL of each of the prepared standard solutions into a 1 L volumetric flask.
4. Add 50 mL of 1 M hydrochloric acid to the flask.
5. Add zero water up to the 1 L marker and mix well.

   The hydrochloric acid concentration will be about 0.05 M after dilution.

Hydrochloric Acid Solution

This section describes the preparation of acid used for IC removal in NPOC measurement and IC measurement with the N Type instrument.

Although the concentration of hydrochloric acid is normally 1 mol/L as described below, adjust the concentration according to the properties of the sample.

Preparation Procedure

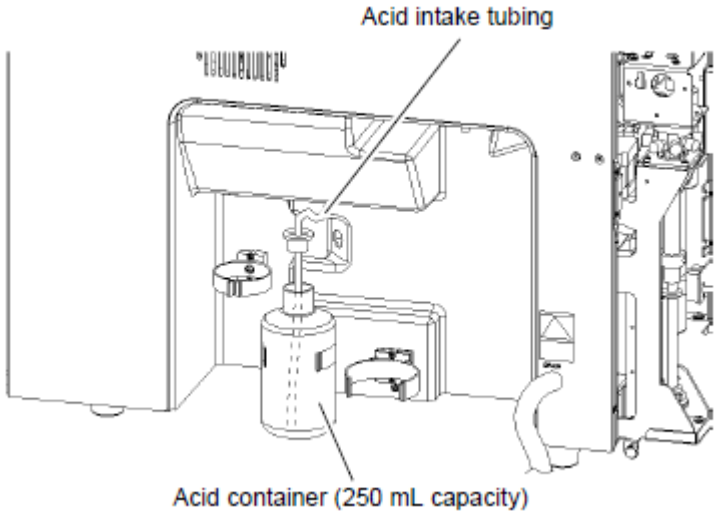
1. Dilute commercially available special grade concentrated hydrochloric acid (12 mol/L) with 12 parts pure water to prepare 1 mol/L hydrochloric acid. A concentration accuracy of approximately ±2 % is acceptable.
   The pure water used as the diluent must have a TOC concentration no greater than 0.5 mg/L. Since the TOC value of the diluent pure water can have a large effect on the analysis result with high sensitivity analysis (low TOC concentrations), it is necessary to use pure water having a very low TOC concentration.

CAUTION

Hydrochloric acid is a hazardous chemical. Use precautions to avoid contact when handling this substance.

2. Pour the prepared acid into the 250 mL acid container provided standard with the instrument, and secure the acid container in the mounting clip on the left side of the instrument.
3. Pass the acid intake tubing through the hole in the cap.
4. Secure the tubing, using a wire clamp about 100 mm from the end of the tubing, to prevent the tubing and cap from separating.
5. Place the cap on the container.

   Note: Verify that the tip of the acid intake tubing nearly reaches the bottom of the container.

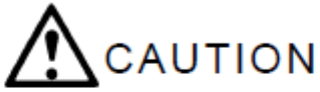


IC Reagent (Phosphoric Acid Solution)

Only for TOC-LC*H High sensitivity model Instruments

Procedure

1. Prepare the IC reagent by diluting 50 mL of commercially available 85 % phosphoric acid (ACS Reagent grade) with pure water to a final volume of 250 mL.
   Since the specific gravity of 85 % phosphoric acid is about 1.7, dilution results in a concentration of phosphoric acid of about 25 % (weight%) in the prepared IC reagent.
   A concentration accuracy of approximately ±2 % is acceptable.
2. Pour the prepared IC reagent into the 250 mL container provided standard with the instrument, and secure the container in the mounting clip on the left side of the instrument.
3. Pass the tip of the IC reagent tubing through the hole in the cap.
4. Secure the tubing, using a wire clamp at a point about 100 mm from the end of the tubing tip, to prevent the tubing and cap from separating.
5. Replace the cap on the container.



The IC reagent contains corrosive phosphoric acid. Always handle acid with care to prevent spills and contact with the skin.