



Inductively Coupled Plasma Optical Emission Spectrophotometer (ICPOES)

Technical and Operation Training

Melbourne TrACEES Platform



TrACEES Platform

Trace Analysis for
Chemical, Earth and
Environmental Sciences

Doc No. WT-ICPOES-v1.0

Created by: Hongzhan Di

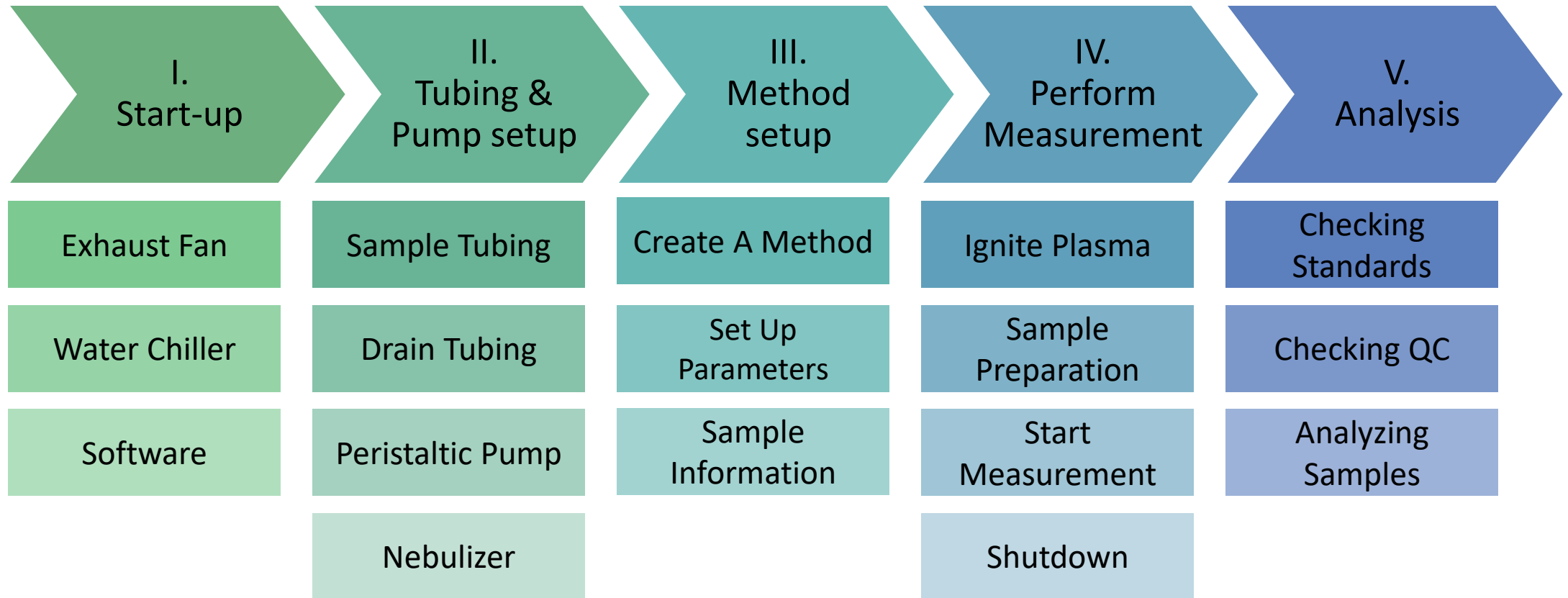
Reviewed by: Ellanor Goyne

Approved by: Alex Duan

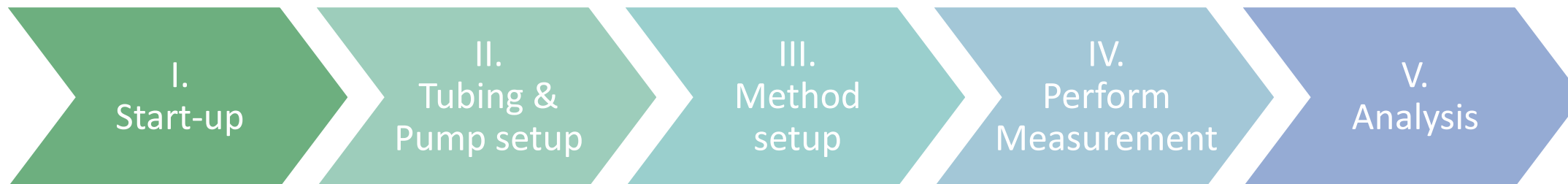
2023, Version 1.0



ICP-OES Operation Flow



ICP-OES Operation Flow



Exhaust fan

Switch on
exhaust fan next
to laboratory
entrance



Cooling water
supply

Turn on the
water chiller
located on the
floor under the
instrument

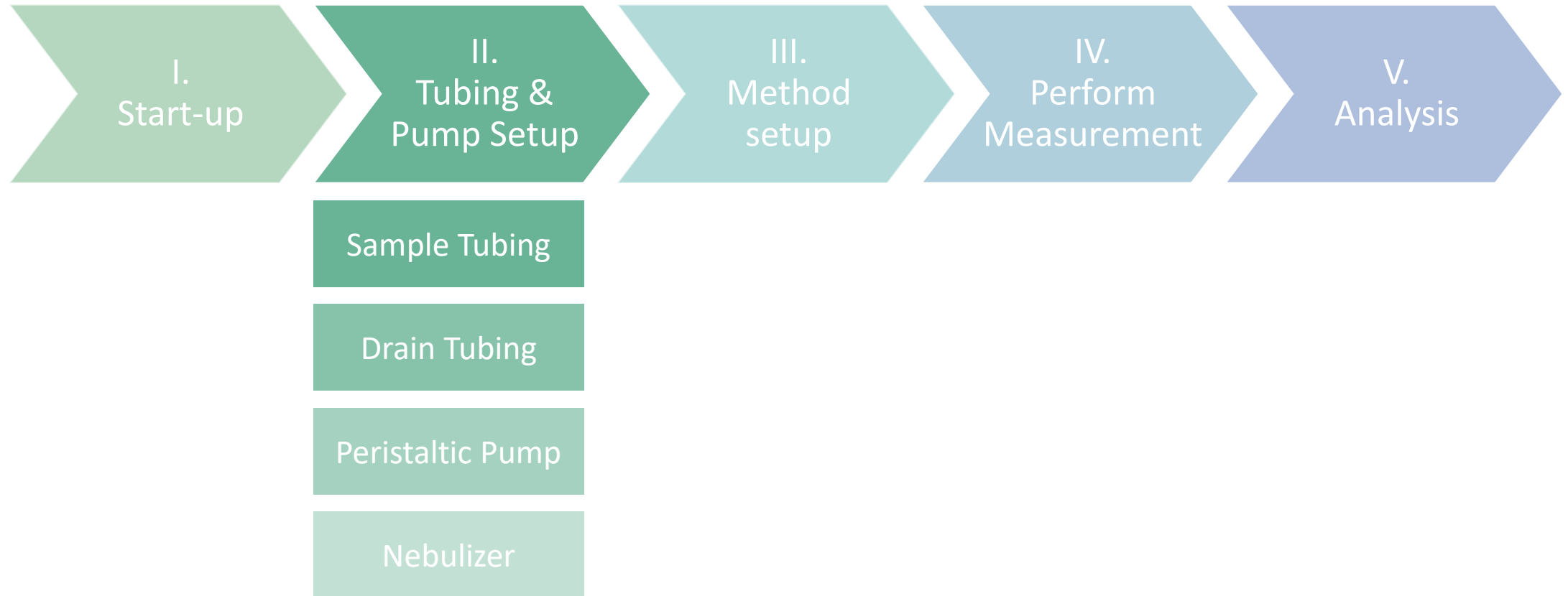


Computer
and software

Double click on
"ICPExpert" on
the desktop to
open software



ICP-OES Operation Flow



li. Tubing & Pump Setup

Sample tubing

Sample
Solution

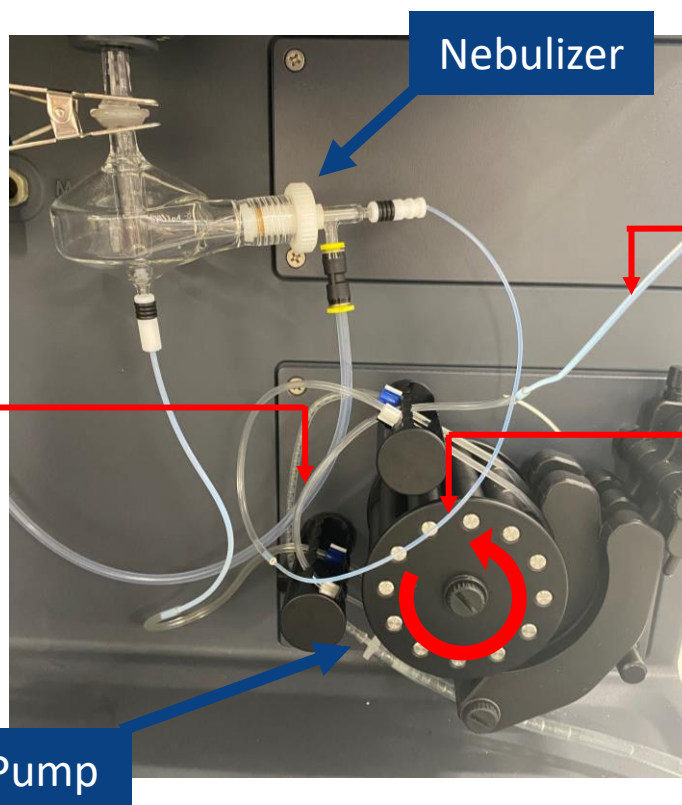
Sample
Tubing

Peristaltic
Pump

Sample
Tubing

Nebulizer

- *White colour clips;*
- *This end comes from sample tubing into the peristaltic pump.*



- ***Sample solution** is injected to **sample tubing** via autosampler*

- *White colour clips;*
- *The sample tubing exits the **peristaltic pump** and injects sample into the **nebulizer**.*

Peristaltic Pump

li. Tubing & Pump Setup

Drain tubing

Nebulizer

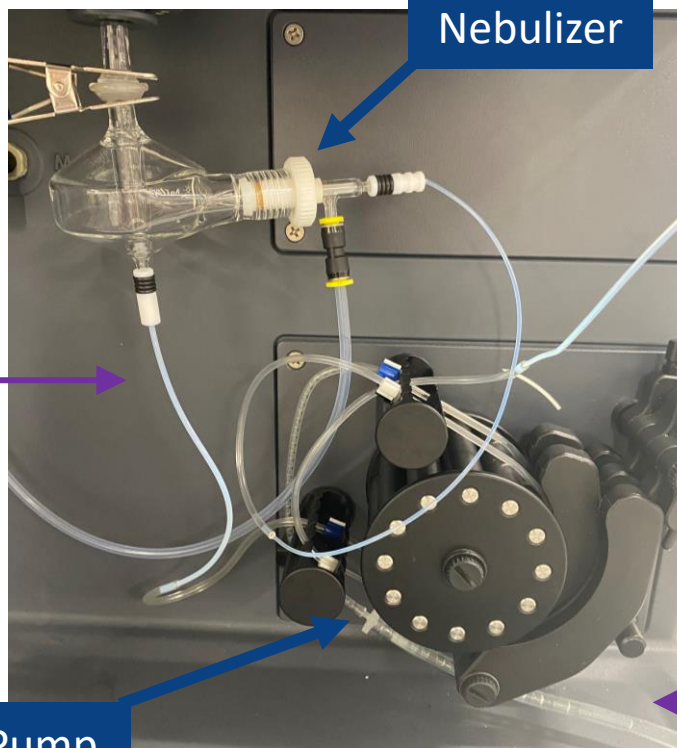
Drain
Tubing

Peristaltic
Pump

Drain
Tubing

Waste

- *Blue colour clips;*
- *This end drains waste from the **nebulizer**.*



- *Blue colour clips;*
- *This end brings waste into the **waste carboy** located below the instrument.*
NOTE: Check waste level before starting any analysis

Peristaltic Pump

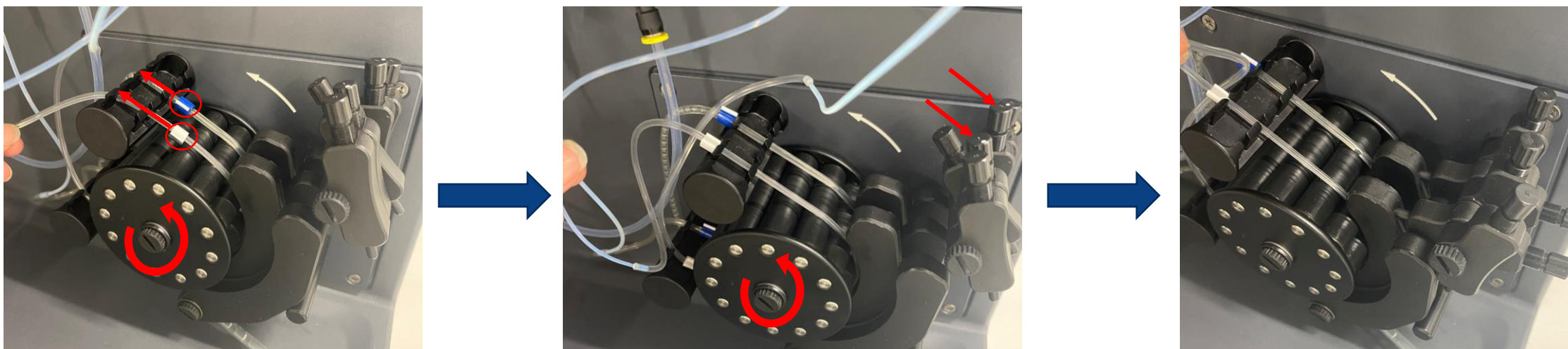
II. Tubing & Pump Setup

Peristaltic pump setup

Step 1.1. The tubing is wrapped around the pump in an **anti-clockwise** direction; The hook points need to be holed inline with the slots in the direction of the red arrows

- *The second slot from the front: Sample tubing;*
- *The second slot from the back: Drain tubing.*

Step 1.2. Swing the 2nd and 4th clamp levers in the direction of the red arrows to apply tension to the clamps.

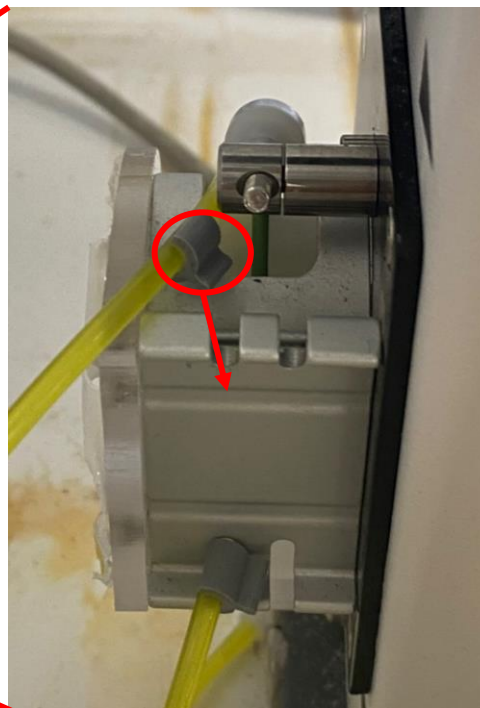
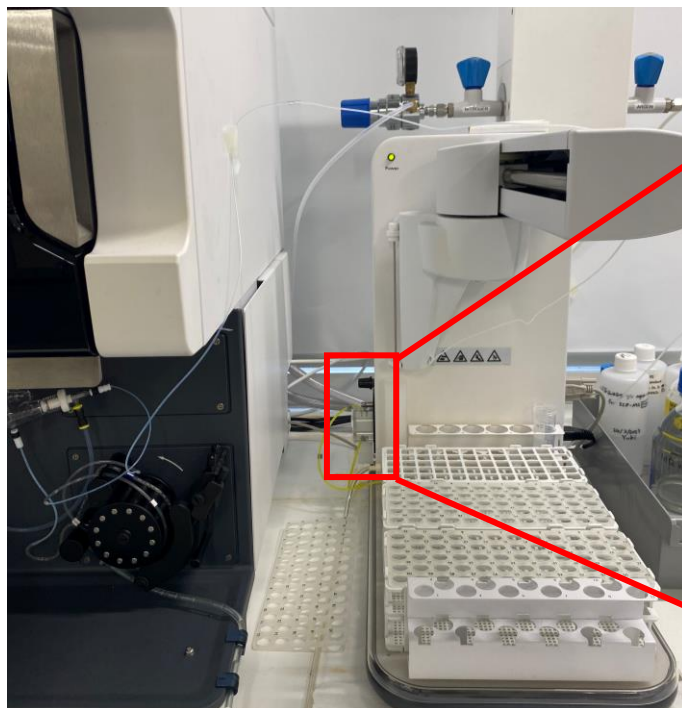


II. Tubing & Pump Setup

Autosampler pump setup

Step 1.1. To the back left of the autosampler there is a second pump. The blue hook points need to be moved into position in the direction of the red arrows

- The tubing needs to line up with the left slot

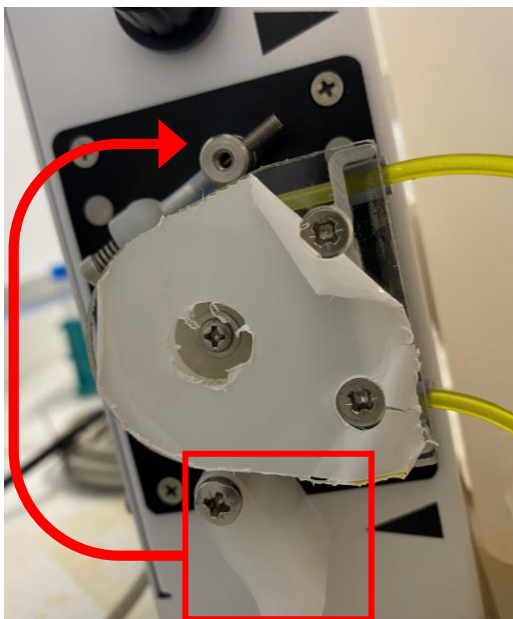


II. Tubing & Pump Setup

Autosampler pump setup

Step 1.2. The tubing needs to be clamped down by swinging the clamp lever from underneath at the back to over the top then lifting the top lever and clamping it down.

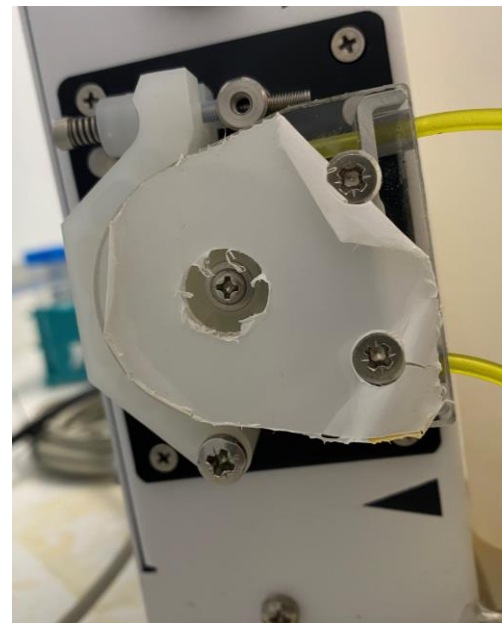
Side View



Front View



Side View

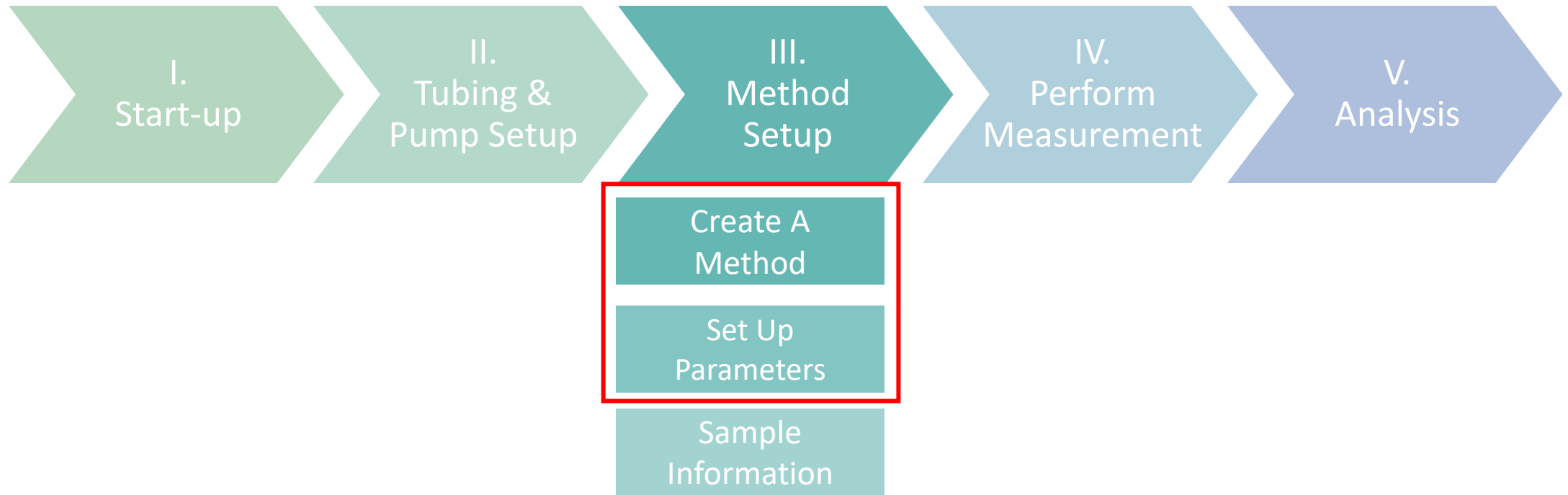


Front View



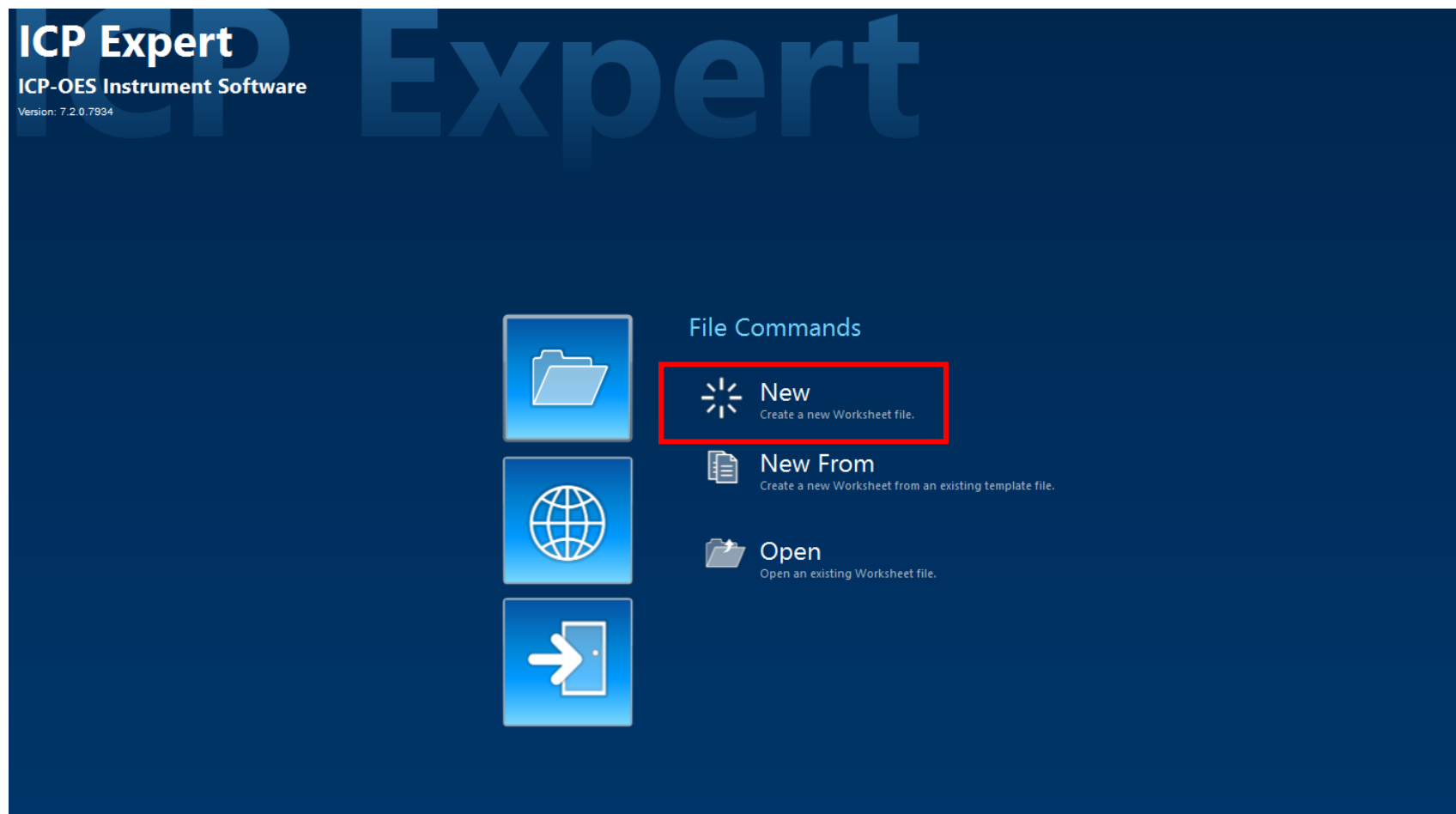


ICP-OES Operation Flow



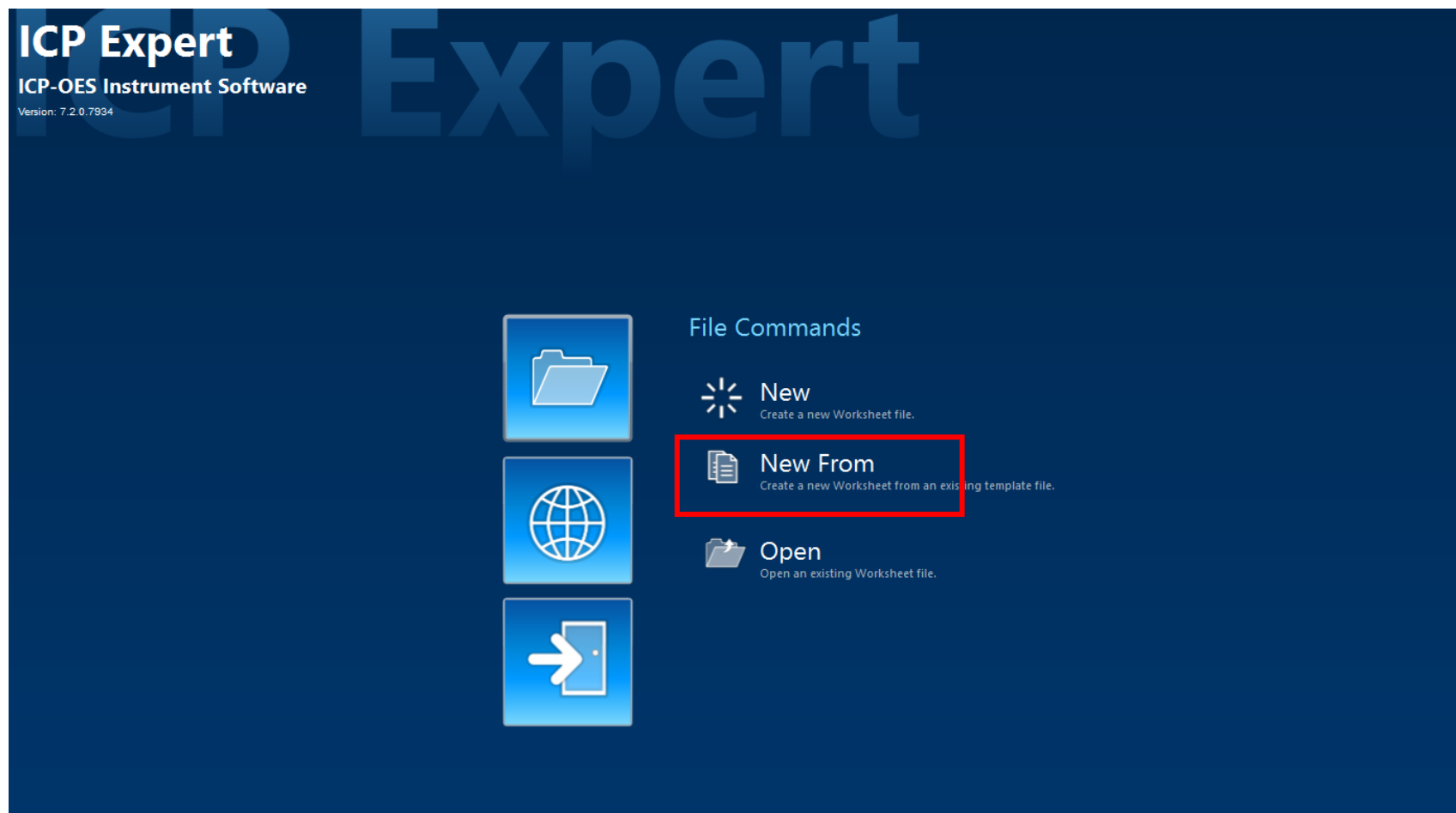
Step 1. Create a Method

Step 1 To create a workbook from new, click on '**New**' on the main screen



Step 1. Create a Method

Step 1 Alternatively to create a new workbook from a template select '**New from**' and select previously saved method



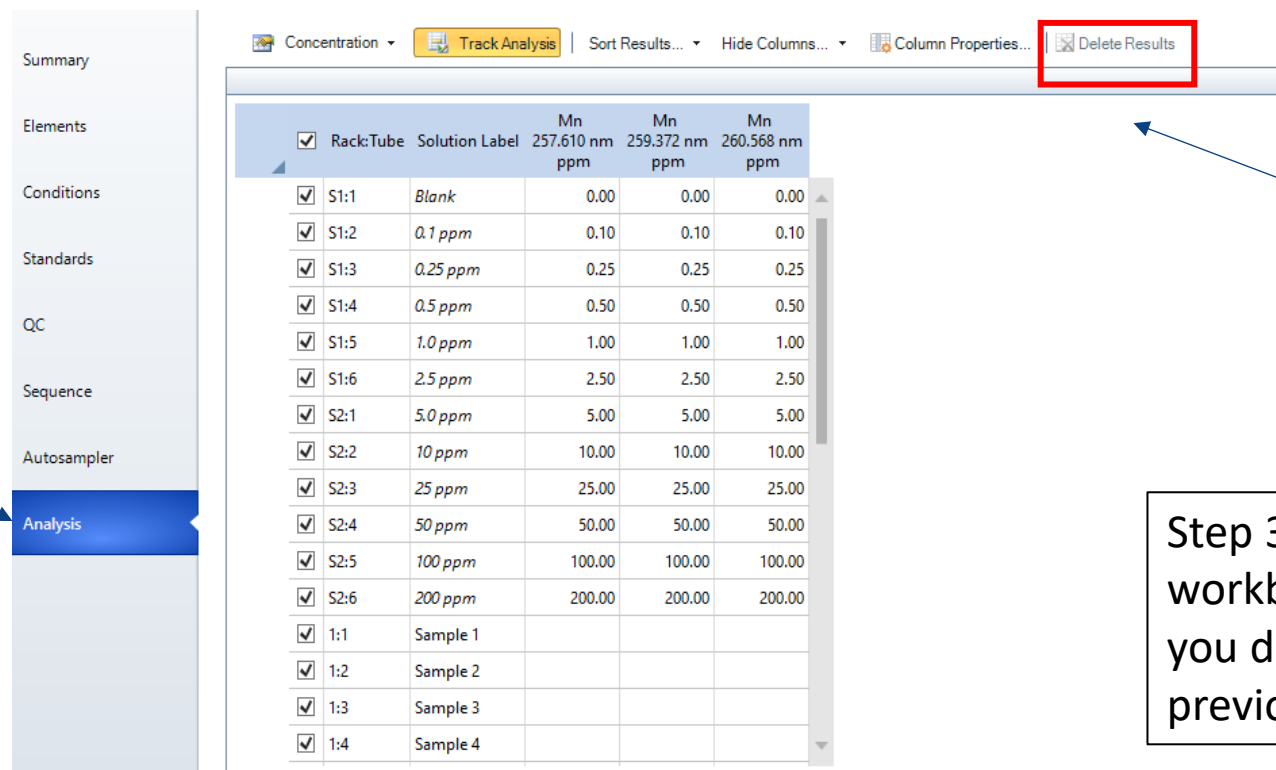
III. Method Setup

Step 1. Create a Method

Step 2 If the worksheet you are using has results attached to it delete the results and click '**Save as**'.

NOTE ensure your workbook has a unique name, a suggested format is [YYMMDD]-[Operator Initial]-[Run]

Step 1: Click on '**Analysis**' tab



☒	Rack:Tube	Solution Label	Mn 257.610 nm ppm	Mn 259.372 nm ppm	Mn 260.568 nm ppm
☒	S1:1	Blank	0.00	0.00	0.00
☒	S1:2	0.1 ppm	0.10	0.10	0.10
☒	S1:3	0.25 ppm	0.25	0.25	0.25
☒	S1:4	0.5 ppm	0.50	0.50	0.50
☒	S1:5	1.0 ppm	1.00	1.00	1.00
☒	S1:6	2.5 ppm	2.50	2.50	2.50
☒	S2:1	5.0 ppm	5.00	5.00	5.00
☒	S2:2	10 ppm	10.00	10.00	10.00
☒	S2:3	25 ppm	25.00	25.00	25.00
☒	S2:4	50 ppm	50.00	50.00	50.00
☒	S2:5	100 ppm	100.00	100.00	100.00
☒	S2:6	200 ppm	200.00	200.00	200.00
☒	1:1	Sample 1			
☒	1:2	Sample 2			
☒	1:3	Sample 3			
☒	1:4	Sample 4			

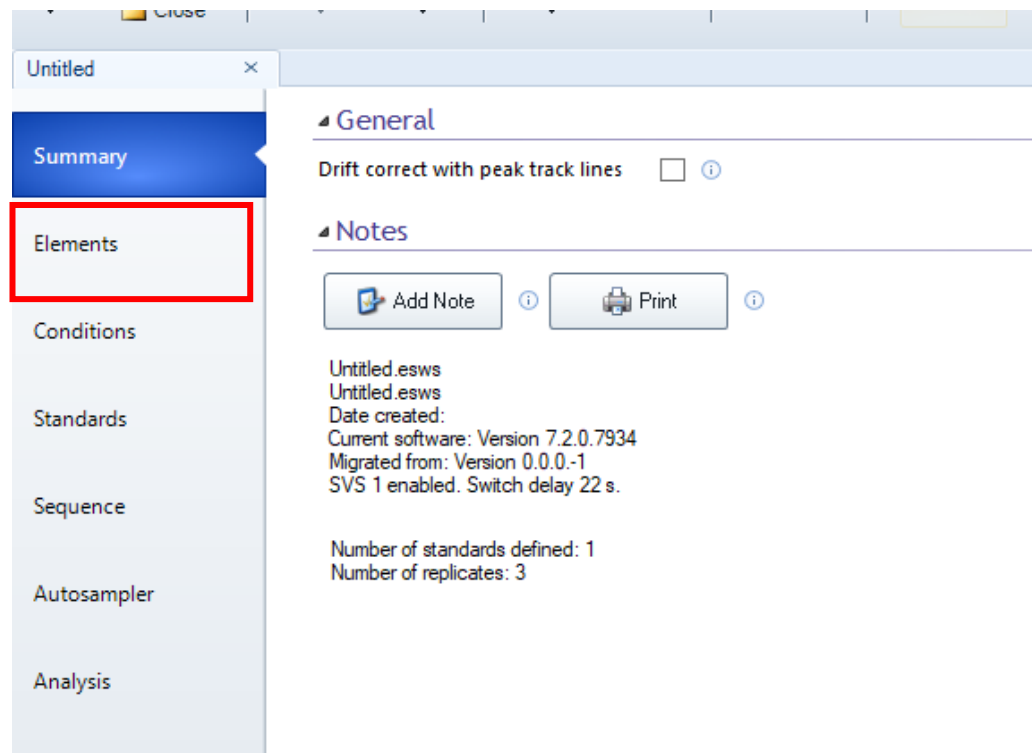
Step 2: Click '**Delete results**'

Step 3: Save as new workbook to ensure you don't lose previous results

Step 2. Setting up Parameters

Step 2.1 Chose the elements and wavelengths for analysis

Click on the '**Elements**' tab on the left toolbar. This will bring up a periodic table



Step 2. Setting up Parameters

Step 2.1 Chose the elements and wavelengths for analysis

Select an element of interest on the periodic table or from the drop down menu.

Step 1: Click on
element of
interest ie Mn

Summary

Elements

Conditions

Standards

Sequence

Autosampler

Analysis

Select element ▼

+

H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	Ac															
			Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	
			Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr	

☐ Enable QC
 ☐ Enable IEC

Element	Wavelength (nm)	Label	Type
---------	-----------------	-------	------

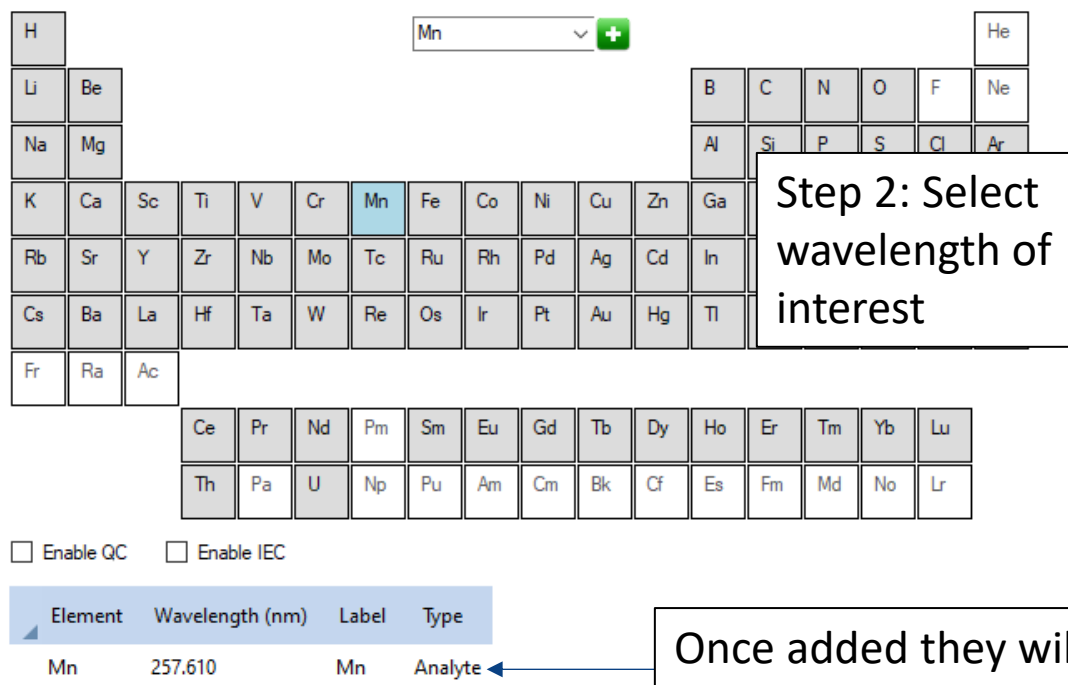
III. Method Setup

Step 2. Setting up Parameters

Step 2.1 Chose the elements and wavelengths for analysis

Select the wavelength of interest on the right hand side then click '**Add**'. Repeat choosing elements and wavelengths until all analytes of interest are added.

NOTE for new samples it is recommended to add the first three wavelengths for analysis.



Step 2: Select wavelength of interest

Once added they will appear on this list

Element	Wavelength (nm)	Label	Type
Mn	257.610	Mn	Analyte

Selected Element Mn

☒ Recommended ☐ All

Add

Wavelength (nm)	Ion	Intensity	Order
257.610	II	222170.0	1
259.372	II	176940.0	2
260.568	II	68540.4	3
294.921	II	71400.0	4
293.931	II	60615.0	5
293.305	II	30325.0	6
279.482	I	12295.0	7
279.827	I	8945.0	8
280.108	I	5580.2	9
191.446	II	406.4	10
344.199	II	9002.3	11
261.815	II	2212.6	12
262.561	II	2206.1	13
192.061	II	303.7	14
261.020	II	1156.1	15
270.170	II	802.0	16
263.817	II	1084.8	17

Step 3: Click '**Add**'

III. Method Setup

Step 2. Setting up Parameters

Step 2.1a Deleting elements and wavelengths for analysis

If you want to delete an analyte in the '**Elements**' tab right click on the analyte you want to delete and click '**Remove**'

Element ▲	Wavelength (nm)	Label	Type
Mn	257.610	Mn	Analyte
Mn	259.372	Mn	Analyte
Mn	260.568	Mn	Analyte
Mn	294.921	Mn	Analyte

Step 1: Right click on element and wavelength to be deleted

☐ Enable QC ☐ Enable IEC

Element ▲	Wavelength (nm)	Label	Type
Mn	257.610	Mn	Analyte
Mn	259.372	Mn	Analyte
Mn	260.568	Mn	Analyte
Mn	294.921	Mn	Analyte

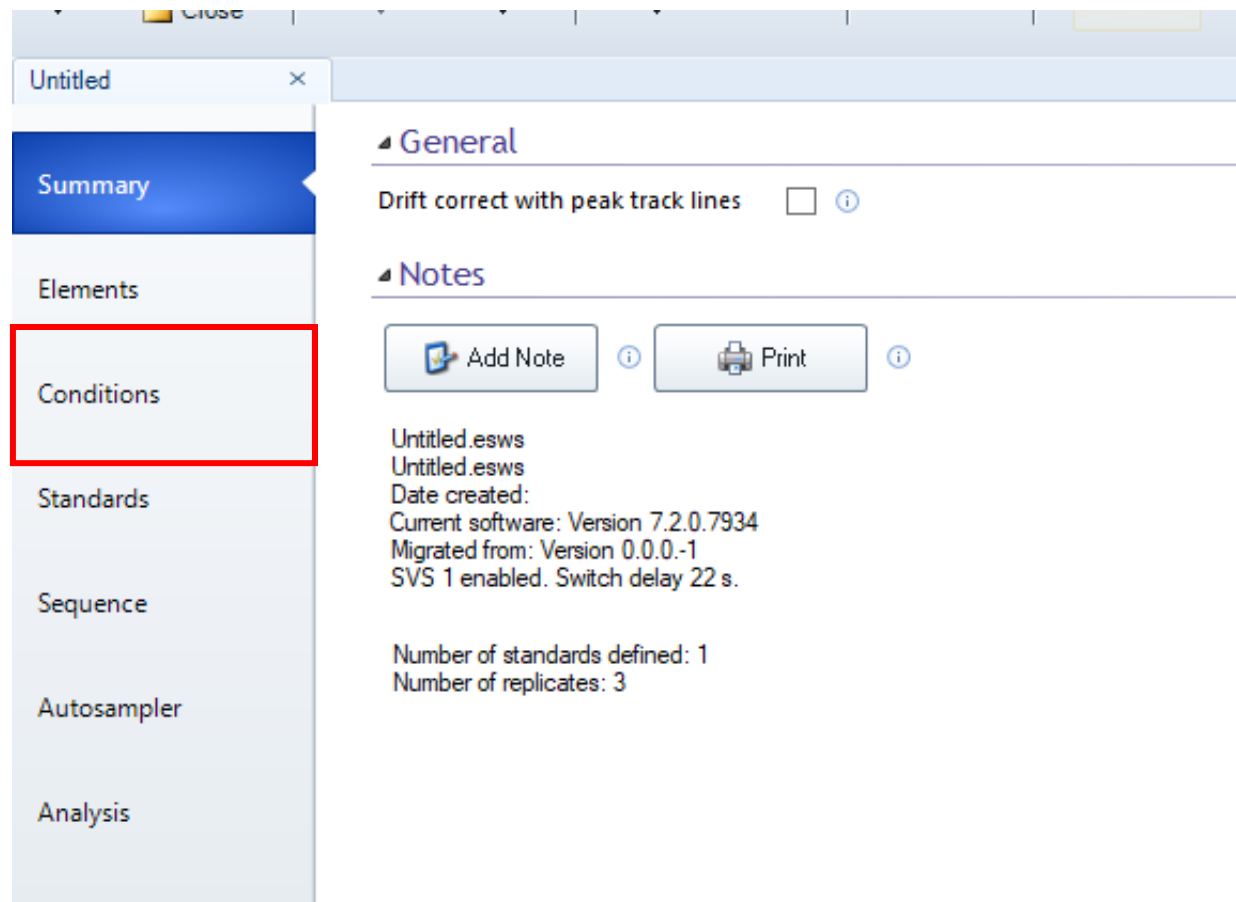
Copy	Ctrl+C
Copy (with headers)	Ctrl+Shift+C
Paste	Ctrl+V
Insert	Insert
Auto fill down	Ctrl+Shift+D
Add Duplicate	
Remove	Delete

Step 2: Click '**Remove**'

Step 2. Setting up Parameters

Step 2.2.1 Setting conditions for analysis

Click on the '**Conditions**' tab on the left toolbar.



Step 2. Setting up Parameters

Step 2.2.1 Setting conditions for analysis

In the section '**Common Conditions**' enter the number of '**Replicates**'.

'**Pump Speed**', '**Sample Introduction**' and '**Uptake Delay**' should stay set to the initial values.

In some cases '**Rinse time**' may need to be increased for "sticky" samples.

Common Conditions

Replicates: ⓘ

Pump speed (rpm): ⓘ

Sample introduction: ☐ Manual
☒ Autosampler

Uptake delay (s): ⓘ ☒ Fast pump

Rinse time (s): ⓘ ☒ Fast pump

Oxygen injection ☐

Step 1 Change
number of
'**Replicates**'

Step 2 Check the
other parameters
haven't been
changed

III. Method Setup

Step 2. Setting up Parameters

Step 2.2.2 Setting conditions for analysis

In the section '**Measurement Conditions**' change '**Viewing mode**' by selecting an option from the dropdown menu

Measurement Conditions

Read time (s):	5		Nebulizer flow (L/min):	0.70	
RF power (kW):	1.20		Plasma flow (L/min):	12.0	
Stabilization time (s):	15		Aux flow (L/min):	1.00	
Viewing mode:	Radial		Make up flow (L/min):	0.00	
Viewing height (mm):	8				

☐ Use multiple conditions

Step 1 Click on the drop down menu

Viewing mode: Radial

Viewing height (mm):

- Radial
- Axial
- Radial
- SVDV

Step 2 Select viewing mode from the menu.
**** describe axial, radial, SVDV??**

Step 2. Setting up Parameters

Step 2.2.3 Setting multiple conditions for analysis

To set up multiple conditions, tick the box next to **'Use multiple conditions'**

Add and navigate to number **'2'** complete Step 2.2.1 to set up new condition parameters

Common Conditions

Replicates: ⓘ

Pump speed (rpm): ⓘ

Sample introduction: ☐ Manual
☒ Autosampler

Uptake delay (s): ⓘ ☒ Fast pump

Rinse time (s): ⓘ ☒ Fast pump

Oxygen injection ☐

Measurement Conditions

Read time (s): ⓘ

RF power (kW): ⓘ

Stabilization time (s): ⓘ

Viewing mode:

Viewing height (mm):

☒ Use multiple conditions

Step 1 Click **'Use multiple conditions'**

☒ Use multiple conditions



Step 2 Click **'+'** to add a new condition

☒ Use multiple conditions



Step 3 Click on **'2'** to navigate to set up parameters of new condition

Step 2.2.3 Setting multiple conditions for analysis

Once you have set up multiple conditions you will need to allocate the condition to the analyte using the '**Condition set**' column.

Label (Wavelength nm)	Type	Background Correction	Number of Pixels	Condition Set
Mn (257.610 nm)	Analyte Fitted		2	1
Mn (259.372 nm)	Analyte Fitted		2	1
Mn (260.568 nm)	Analyte Fitted		2	1

Replicates: 3

Pump speed (rpm): 12

Sample introduction: ☐ Manual ☒ Autosampler

Uptake delay (s): 25 ☒ Fast pump

Rinse time (s): 30 ☒ Fast pump

Oxygen injection ☐

Measurement Conditions

Read time (s): 5

RF power (kW): 1.20

Stabilization time (s): 15

Viewing mode: Axial

Viewing height (mm): 8

Nebulizer flow (L/min): 0.70

Plasma flow (L/min): 12.0

Aux flow (L/min): 1.00

Make up flow (L/min): 0.00

☒ Use multiple conditions

1 2 3 4 + -

Step 2. Setting up Parameters

Step 2.2.3 Setting multiple conditions for analysis

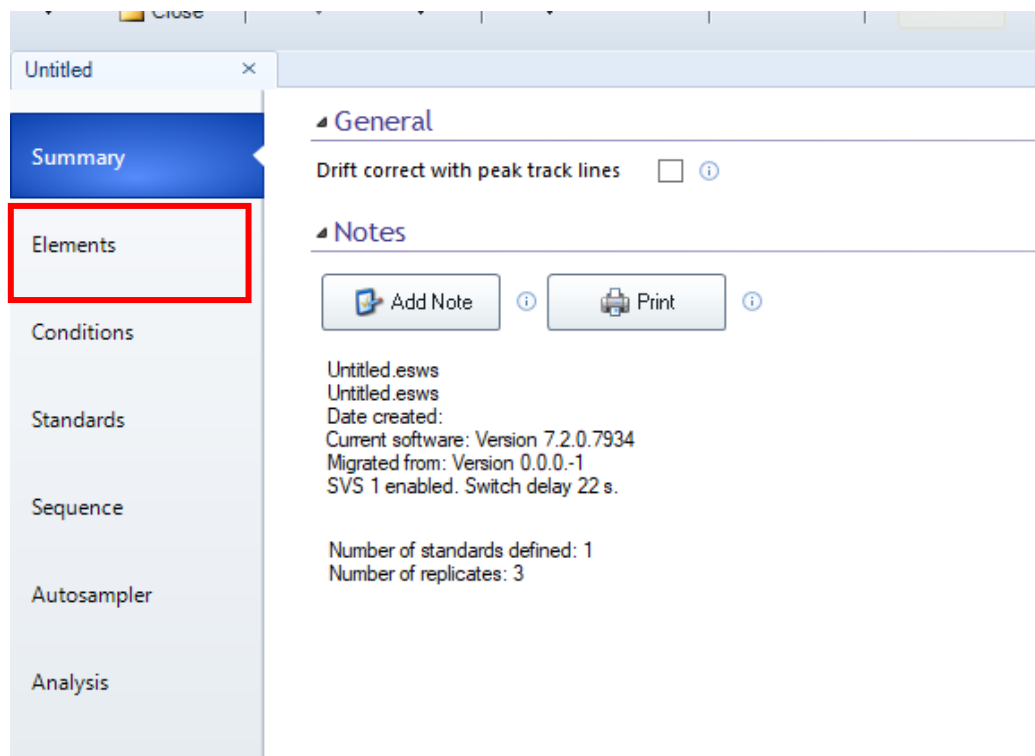
To allocate an analyte to a condition click on the box under '**Condition set**' to bring up a drop down menu and select the condition set number.

Label (Wavelength nm)	Type	Background Correction	Number of Pixels	Condition Set
Mn (257.610 nm)	Analyte Fitted		2	1
Mn (259.372 nm)	Analyte Fitted		2	1
Mn (260.568 nm)	Analyte Fitted		2	1 1 2

Step 2. Setting up Parameters

Step 2.2.4 *Setting multiple conditions for analysis of one analyte*

*To complete multiple conditions on the same set of analytes you will need to duplicate the analytes. First navigate to the '**Elements**' tab on the left hand side.*



III. Method Setup

Step 2. Setting up Parameters

Step 2.2.4 Setting multiple conditions for analysis of one analyte

Repeat **step 2.1** to insert a duplicate of the required analyte.

Periodic table showing element selection. The element **Mn** is selected. A dropdown menu shows **Mn** with a green plus icon.

Step 2: Select wavelength of interest

Enable QC ☐ Enable IEC ☐

Element	Wavelength (nm)	Label	Type
Mn	257.610	Mn	Analyte

Selected Element Mn

☒ Recommended ☐ All

Add

Wavelength (nm)	Ion	Intensity	Order
257.610	II	222170.0	1
259.372	II	176940.0	2
260.568	II	68540.4	3
294.921	II	71400.0	4
293.931	II	60615.0	5
293.305	II	30325.0	6
279.482	I	12295.0	7
279.827	I	8945.0	8
280.108	I	5580.2	9
191.446	II	406.4	10
344.199	II	9002.3	11
261.815	II	2212.6	12
262.561	II	2206.1	13
192.061	II	303.7	14
261.020	II	1156.1	15
270.170	II	802.0	16
263.817	II	1084.8	17

Step 3: Click
'Add'

Once added they will
appear on this list

Step 2. Setting up Parameters

Step 2.2.4 Setting multiple conditions for analysis of one analyte

Once you have duplicated analytes of interest change the label to differentiate the conditions ie. Condition 1 and 2 or radial and axial by clicking on the box and typing the new label

Element ▲	Wavelength (nm)	Label	Type
Mn	257.610	Mn	Analyte
Mn	257.610	Mn Copy 1	Analyte
Mn	259.372	Mn	Analyte
Mn	259.372	Mn Copy 1	Analyte
Mn	260.568	Mn	Analyte
Mn	260.568	Mn Copy 1	Analyte

Element ▲	Wavelength (nm)	Label	Type
Mn	257.610	Mn condition 1	Analyte
Mn	257.610	Mn condition 2	Analyte
Mn	259.372	Mn condition 1	Analyte
Mn	259.372	Mn condition 2	Analyte
Mn	260.568	Mn condition 1	Analyte
Mn	260.568	Mn condition 2	Analyte

Step 2. Setting up Parameters

Step 2.2.4 Setting multiple conditions for analysis of one analyte

In the '**Condition set**' column click on the condition to bring up a drop down box and select the condition set.

Step 1: Click on the condition set column of the analyte

Label (Wavelength nm)	Type	Background Correction	Number of Pixels	Condition Set
Mn condition 1 (259.372 nm)	Analyte	Fitted	2	1
Mn condition 2 (259.372 nm)	Analyte	Fitted	2	1
Mn condition 1 (260.568 nm)	Analyte	Fitted	2	1
Mn condition 2 (260.568 nm)	Analyte	Fitted	2	1
Mn condition 1 (257.610 nm)	Analyte	Fitted	2	1
Mn condition 2 (257.610 nm)	Analyte	Fitted	2	1

Label (Wavelength nm)	Type	Background Correction	Number of Pixels	Condition Set
Mn condition 1 (259.372 nm)	Analyte	Fitted	2	1
Mn condition 2 (259.372 nm)	Analyte	Fitted	2	1
Mn condition 1 (260.568 nm)	Analyte	Fitted	2	1
Mn condition 2 (260.568 nm)	Analyte	Fitted	2	2
Mn condition 1 (257.610 nm)	Analyte	Fitted	2	1
Mn condition 2 (257.610 nm)	Analyte	Fitted	2	1

Step 2: Select the condition from the drop down menu

Step 2.2.4 Setting multiple conditions for analysis of one analyte

Once you have duplicated analytes of interest change the label to differentiate the conditions ie. Condition 1 and 2 or radial and axial by clicking on the box and typing the new label

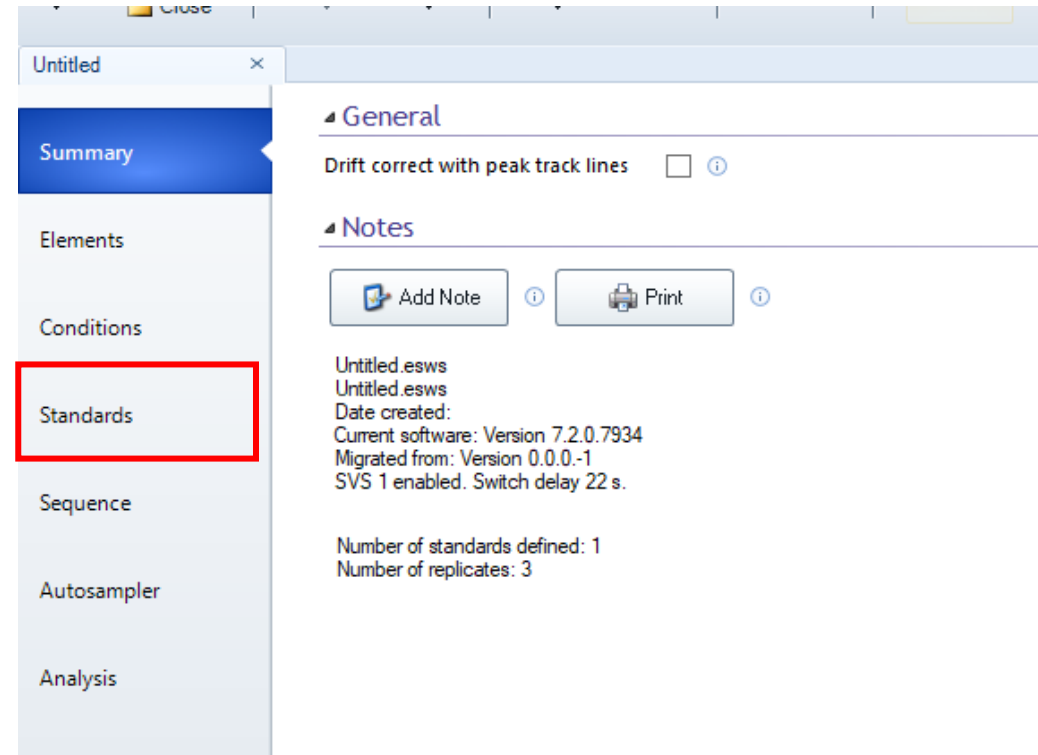
Label (Wavelength nm)	Type	Background Correction	Number of Pixels	Condition Set
Mn condition 1 (257.610 nm)	Analyte	Fitted	2	1
Mn condition 1 (259.372 nm)	Analyte	Fitted	2	1
Mn condition 1 (260.568 nm)	Analyte	Fitted	2	1
Mn condition 2 (259.372 nm)	Analyte	Fitted	2	2
Mn condition 2 (257.610 nm)	Analyte	Fitted	2	2
Mn condition 2 (260.568 nm)	Analyte	Fitted	2	2

You will notice the condition sets will separate out in the list as you change them. This is because the ICP will run one condition and then the next so this will double your analysis time.

Step 2. Setting up Parameters

Step 2.3 *Setting up calibration standards for analysis*

*Click on the '**Standards**' tab on the left toolbar.*



Step 2.3 Setting up calibration standards for analysis

Enter '**Number of standards**'

Enter '**Solution label**' and the concentration of analyte(s) of interest for each standard to the table from lowest to highest concentration

Step 1: Select '**Number of standards**'

Step 2: Enter '**Solution label**' for each standard

Step 3: Enter Concentration of analyte(s) in each standard

Concentrations

Correlation coefficient limit: 0.9990 ⓘ

Include blank in calibration ☒

Standard additions

Use reagent blank ☐

Enable reslope: ☐

Enable MultiCal view ☐

MultiCal summary concentration ☐

Number of standards: 1 ⓘ

Solution Label	Rack:Tube	Mn
Blank	S1:1	ppm
	S1:2	0.00

Calibration Fit

Label (Wavelength nm)	Unit	Calibration Fit	Weighted Fit	Through Blank	Minimum Concentration	Maximum Concentration	Calibration Error
Mn (257.610 nm)	ppm	Linear	☒	☐			5 %
Mn (259.372 nm)	ppm	Linear	☒	☐			5 %
Mn (260.568 nm)	ppm	Linear	☒	☐			5 %

III. Method Setup

Step 2. Setting up Parameters

Step 2.3 Setting up calibration standards for analysis

Check the standards have been entered correctly with the correct units.

Number of standards: 11



0.00
0.0

Click to increase number of decimal places

	Solution Label	Rack:Tube	Mn
			ppm
	Blank	S1:1	0.00
	0.1 ppm	S1:2	0.10
	0.25 ppm	S1:3	0.25
	0.5 ppm	S1:4	0.50
	1.0 ppm	S1:5	1.00
	2.5 ppm	S1:6	2.50
	5.0 ppm	S2:1	5.00
	10 ppm	S2:2	10.00

Double click in '**Rack:Tube**' column of each analyte to change location of standard via dropdown boxes if necessary.

Units of analytes may be changed using the drop down menu

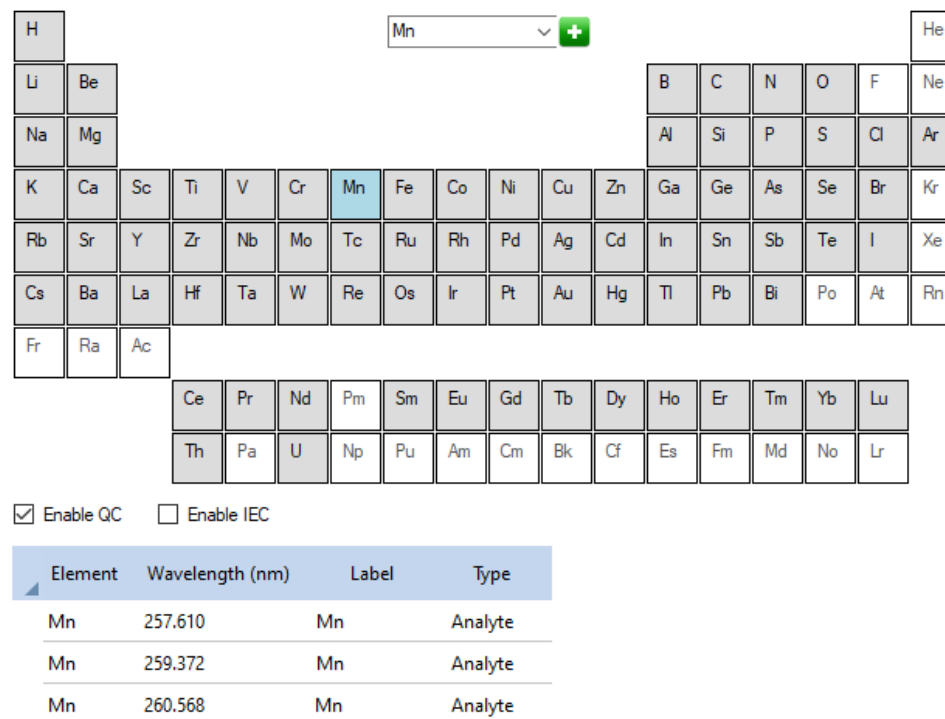
Step 2. Setting up Parameters

Step 2.3 Setting up QC standards

Navigate to the '**Conditions**' tab on the left hand side toolbar.

Check the box labelled '**Enable QC**'

Step 1: Check the box '**Enable QC**'



The screenshot shows the software interface for setting up parameters. At the top, a dropdown menu shows 'Mn' with a green plus button. Below it is a periodic table with 'Mn' highlighted in blue. At the bottom, there are two checkboxes: 'Enable QC' (checked) and 'Enable IEC' (unchecked). Below the checkboxes is a table with the following data:

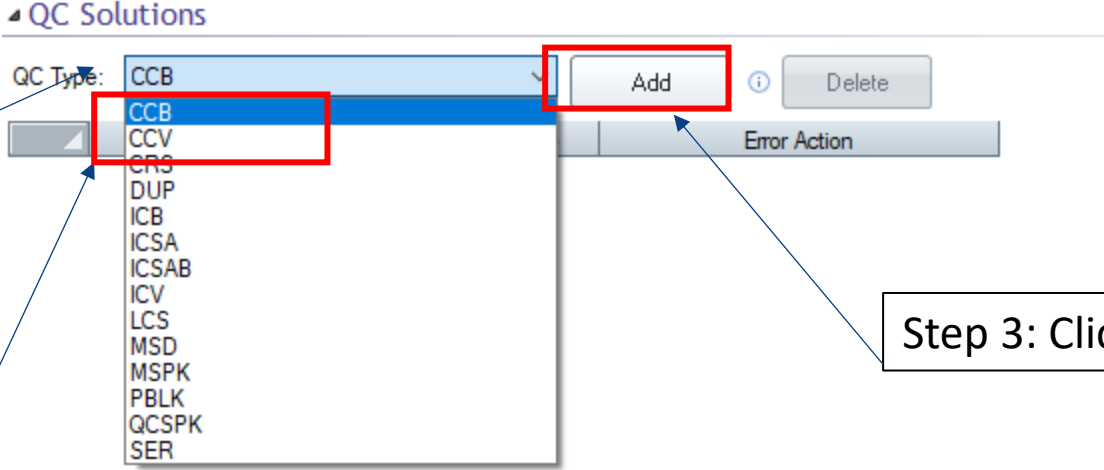
Element	Wavelength (nm)	Label	Type
Mn	257.610	Mn	Analyte
Mn	259.372	Mn	Analyte
Mn	260.568	Mn	Analyte

This will trigger a QC tab to be created in the left hand side panel.

Step 2. Setting up Parameters

Step 2.3 Setting up QC standards

Select QC standards you want to add to your sequence and click add. As a minimum Continuing Calibration Blank ('**CCB**') and Continuing Calibration Verification ('**CCV**') should be used.



The screenshot shows the 'QC Solutions' window. A dropdown menu for 'QC Type:' is open, displaying a list of standard types: CCB, CCV, CRS, DUP, ICB, ICSA, ICSAB, ICV, LCS, MSD, MSPK, PBLK, QCSPK, and SER. The 'Add' button is highlighted with a red box. A red box also highlights the 'CCB' option in the dropdown list. Arrows point from text boxes to these elements.

Step 1: Open the drop box box

Step 2: Select QC standard ie '**CCB**' or '**CCV**'.

Step 3: Click '**Add**'

III. Method Setup

Step 2. Setting up Parameters

Step 2.3 Setting up QC standards

The concentration of the Continuing Calibration Verification needs to be defined by selecting '**Continuing Calibration Verification**' to open up the CCV menu and adding the concentration to the '**Define concentration**' column.

QC Solutions

QC Type:

	QC Name	QC Type	Error Action
▶	Continuing Calibration Blank	CCB	Flag and continue
	Continuing Calibration Verification	CCV	Flag and continue

Step 1: Click on the '**Continuing Calibration Verification**' to open the menu to right.

Step 2: '**Define concentrations**' of analyte(s) of interest

Continuing Calibration Verification

Report value: $\text{MeasuredConc} / \text{DefinedConc} * 100$

Pass test: $\text{ReportValue} \geq \text{LowerLimit}$ and $\text{ReportValue} \leq \text{UpperLimit}$

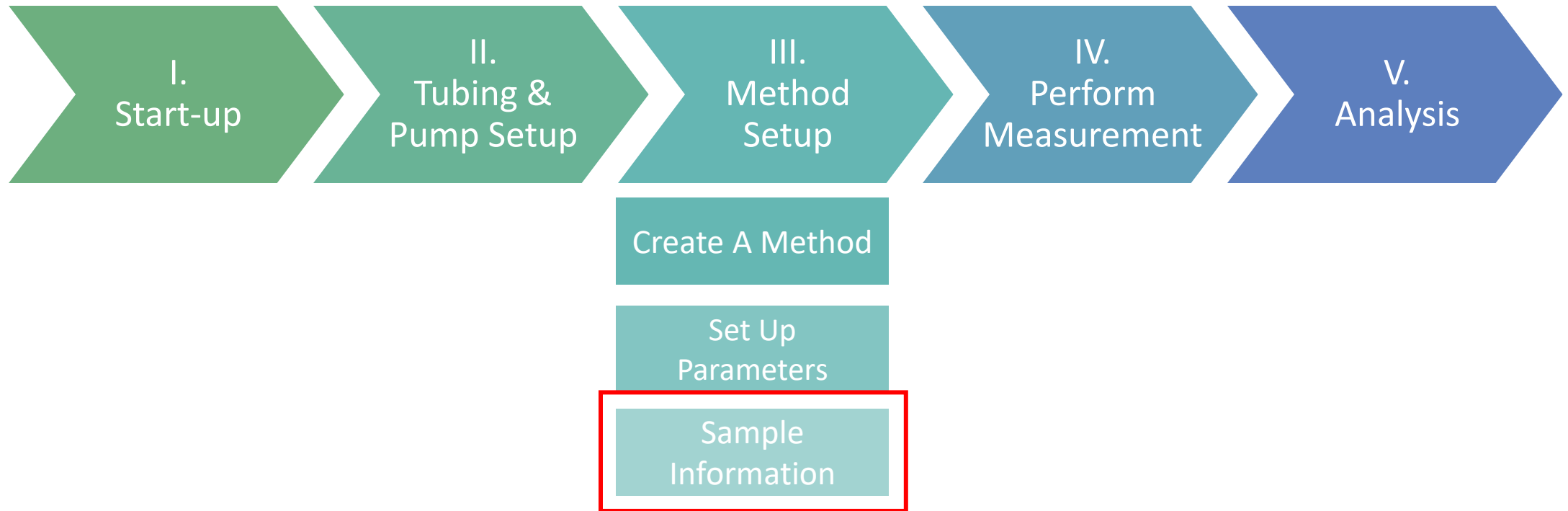
Failure flag: Q

Limits

	☒	Element	Defined Concentration	Units	Lower Limit	Upper Limit
	☒	Mn (257.6...	1.00	ppm	90%	110%
	☒	Mn (259.3...	1.00	ppm	90%	110%
▶	☒	Mn (260.5...	1.00	ppm	90%	110%



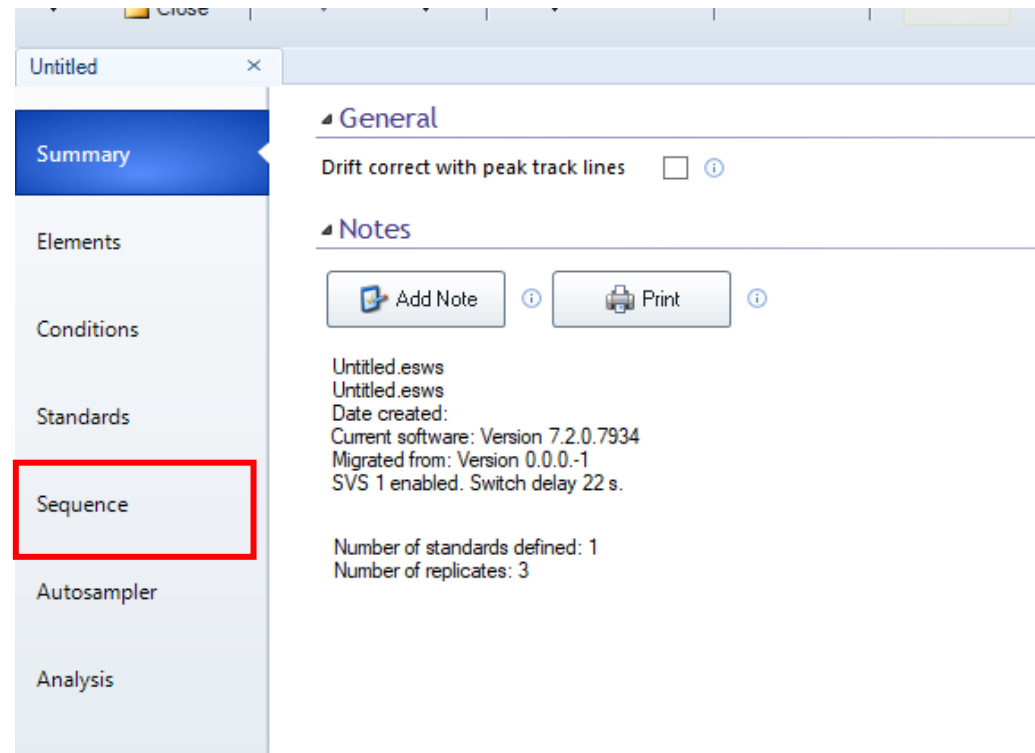
ICP-OES Operation Flow



Step 3. Sample information

Step 3.1 *Setting up sample sequence*

*Navigate to the '**Sequence**' tab on the left hand side toolbar*



Step 3. Sample information

Step 3.1 Setting up sample sequence

Enter '**Number of samples**', '**Sample names**' and locations

Step 1: Enter '**Number of samples**'

▲ Samples and Calibration

Number of samples:

☐ Calibration every (samples):

▲ End of Run Actions

☒ Set pump speed (rpm): ☐ Rinse system (min):

☐ Turn plasma and pump off

☐ Turn plasma off and reduce pump speed

Step 2: Edit '**Sample names**'

Step 3: Change sample location via drop down boxes which appear when you click on the cell under '**Rack:Tube**'

Rack:Tube	Solution Label	Solution Type	Weight (g)	Volume (mL)	Dilution
S1:3	0.25 ppm	Calibration			
S1:4	0.5 ppm	Calibration			
S1:5	1.0 ppm	Calibration			
S1:6	2.5 ppm	Calibration			
S2:1	5.0 ppm	Calibration			
S2:2	10 ppm	Calibration			
S2:3	25 ppm	Calibration			
S2:4	50 ppm	Calibration			
S2:5	100 ppm	Calibration			
S2:6	200 ppm	Calibration			
1:1	Sample 1	Sample	1.0	1.0	1.0
1:2	Sample 2	Sample	1.0	1.0	1.0
1:3	Sample 3	Sample	1.0	1.0	1.0
1:4	Sample 4	Sample	1.0	1.0	1.0
1:5	Sample 5	Sample	1.0	1.0	1.0
1:6	Sample 6	Sample	1.0	1.0	1.0
1:7	Sample 7	Sample	1.0	1.0	1.0
1:8	Sample 8	Sample	1.0	1.0	1.0
1:9	Sample 9	Sample	1.0	1.0	1.0
1:10	Sample 10	Sample	1.0	1.0	1.0
1:11	Sample 11	Sample	1.0	1.0	1.0
1:12	Sample 12	Sample	1.0	1.0	1.0
1:13	Sample 13	Sample	1.0	1.0	1.0
1:14	Sample 14	Sample	1.0	1.0	1.0
1:15	Sample 15	Sample	1.0	1.0	1.0
1:16	Sample 16	Sample	1.0	1.0	1.0
1:17	Sample 17	Sample	1.0	1.0	1.0
1:18	Sample 18	Sample	1.0	1.0	1.0
1:19	Sample 19	Sample	1.0	1.0	1.0
1:20	Sample 20	Sample	1.0	1.0	1.0

Step 4: Enter '**Weight**', '**Volume**' and '**Dilution**' factor for samples (left to right)

III. Method setup

Step 3. Sample information

Step 3.1 Adding quality control samples

QC samples needed to be added manually to the sample sequence every 15 samples to monitor for drift
Right click on the sample you want the QC samples after > **'Insert samples below'** > **'Continued Calibration Blank'** (CCB) or **'Continued Calibration Verification'** (CCV)

Step 1: Right click
on sample

Step 2: Hover
mouse over
**'Insert solution
below'**

1:11	Sample 11	Sample	1.0	1.0	1.0
1:12	Sample 12	Sample	1.0	1.0	1.0
1:13	Sample 13	Sample	1.0	1.0	1.0
1:14	Sample 14	Sample	1.0	1.0	1.0
1:15	Sample 15	Sample	1.0	1.0	1.0
1:16	Sample 16	Sample	1.0	1.0	1.0
1:17	Sample 17	Sample	1.0	1.0	1.0
1:18	Sample 18	Sample	1.0	1.0	1.0
1:19	Sample 19	Sample	1.0	1.0	1.0
1:20	Sample 20	Sample	1.0	1.0	1.0

Copy Ctrl+C

Copy (with headers) Ctrl+Shift+C

Paste Ctrl+V

Auto increment down

Auto fill down Ctrl+Shift+D

Insert solution below

Delete Selected Solutions

Rate QC preferences...

Expand all summary rows

Collapse all summary rows

Sample

Continuing Calibration Blank

Continuing Calibration Verification

QC Spike

Step 3: Select
**'Continuing
Calibration Blank'**

Step 4: Repeat step
1-2 and Select
**'Continuing
Calibration
Verification'**

III. Method setup

Step 3. Sample information

Step 3.1 Adding quality control samples

Repeat addition of CCB and CCV every 15 samples and sample locations need to be specified.

Samples and Calibration

Number of samples:

☐ Calibration every (samples):

QC Tests

QC Name	QC Type	Count as sample when manually inserted
Continuing Calibration Blank	CCB	☐
Continuing Calibration Verification	CCV	☐

End of Run Actions

☒ Set pump speed (rpm):
☐ Rinse system (min):

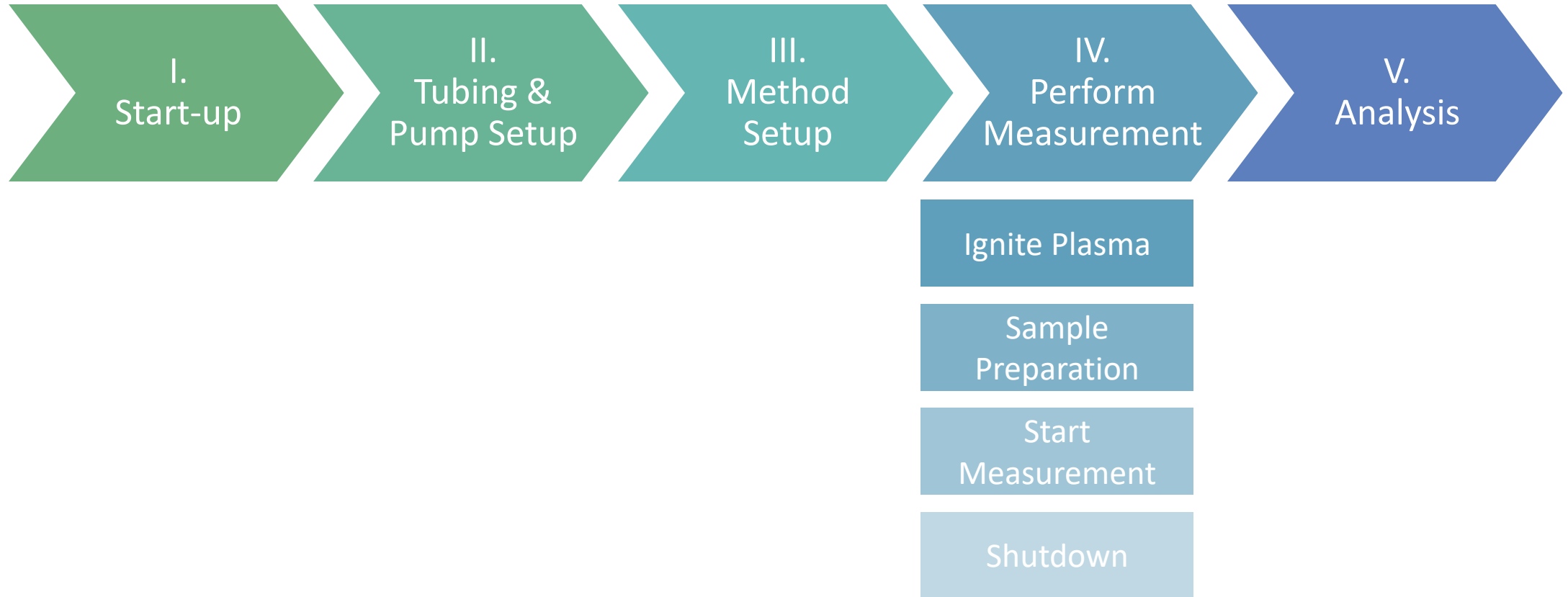
☐ Turn plasma and pump off
 ☐ Turn plasma off and reduce pump speed

Rack:Tube	Solution Label	Solution Type	Weight (g)	Volume (mL)	Dilution
S1:5	1.0 ppm	Calibration			
S1:6	2.5 ppm	Calibration			
S2:1	5.0 ppm	Calibration			
S2:2	10 ppm	Calibration			
S2:3	25 ppm	Calibration			
S2:4	50 ppm	Calibration			
S2:5	100 ppm	Calibration			
S2:6	200 ppm	Calibration			
1:1	Sample 1	Sample	1.0	1.0	1.0
1:2	Sample 2	Sample	1.0	1.0	1.0
1:3	Sample 3	Sample	1.0	1.0	1.0
1:4	Sample 4	Sample	1.0	1.0	1.0
1:5	Sample 5	Sample	1.0	1.0	1.0
1:6	Sample 6	Sample	1.0	1.0	1.0
1:7	Sample 7	Sample	1.0	1.0	1.0
1:8	Sample 8	Sample	1.0	1.0	1.0
1:9	Sample 9	Sample	1.0	1.0	1.0
1:10	Sample 10	Sample	1.0	1.0	1.0
1:11	Sample 11	Sample	1.0	1.0	1.0
1:12	Sample 12	Sample	1.0	1.0	1.0
1:13	Sample 13	Sample	1.0	1.0	1.0
1:14	Sample 14	Sample	1.0	1.0	1.0
1:15	Sample 15	Sample	1.0	1.0	1.0
	Continuing Calibration Blank	Continuing Calibration Blank			
	Continuing Calibration Verification	Continuing Calibration Verification			
1:16	Sample 16	Sample	1.0	1.0	1.0
1:17	Sample 17	Sample	1.0	1.0	1.0
1:18	Sample 18	Sample	1.0	1.0	1.0
1:19	Sample 19	Sample	1.0	1.0	1.0
1:20	Sample 20	Sample	1.0	1.0	1.0

Sample locations will need to be specified by clicking and entering via the two drop down boxes



ICP-OES Operation Flow



IV. Perform Measurement

Step 1. Check the system is operational

Step 1.1 Click the '**Instrument**' button on the icon toolbars

NOTE: If the instrument is greyed out you will need to connect to the instrument (**Step 1.1a**)

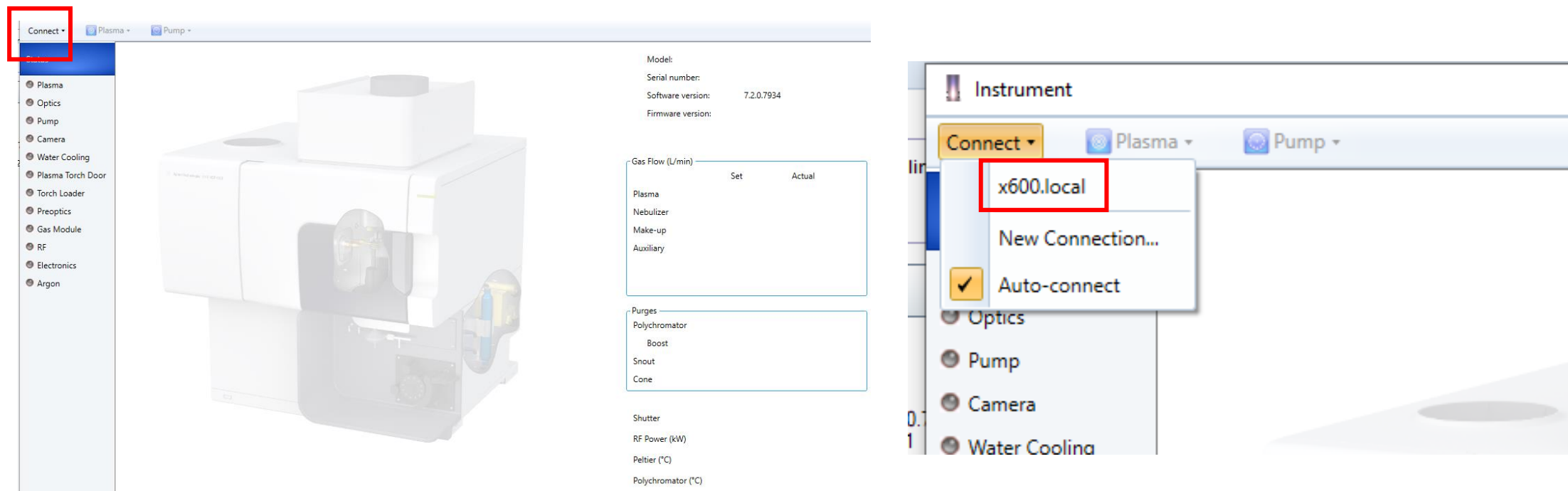


IV. Perform Measurement

Step 1. Check the system is operational

Step 1.1a Connecting the instrument to the computer

Click '**Connect**' on the top toolbar > '**x600.local**' and wait for the computer to connect



IV. Perform Measurement

Step 1. Check the system is operational

Step 1.2 Ensure that all systems are operational as indicated by a green light on the left hand panel

All should have green lights except '**Camera**' and '**Plasma**' which will only be green once the plasma is turned on



The screenshot displays the software interface for the Agilent 5100 SVDV ICP-OES system. On the left, a sidebar menu lists various system components with status indicators (green circles). A red box highlights the 'Plasma' and 'Camera' components, which are currently not green. The main area shows a 3D model of the instrument. On the right, hardware information and operational data are displayed.

Hardware Information:

- Model: Agilent 5100 SVDV ICP-OES
- Serial number: MY15130002
- Software version: 7.2.0.7934
- Firmware version: 3121
- [More hardware information...](#)

Gas Flow (L/min):

	Set	Actual
Plasma	8.00	0.00
Nebulizer	0.00	0.00
Make-up	0.00	0.01
Auxiliary	0.00	0.00
O ₂ injection		

Purges:

	Status
Polychromator	Green
Boost	Grey
Snout	Grey
Cone	Grey

Other Parameters:

	Status
Shutter	Grey
RF Power (kW)	0.00
Peltier (°C)	-1.8
Polychromator (°C)	35.0

IV. Perform Measurement

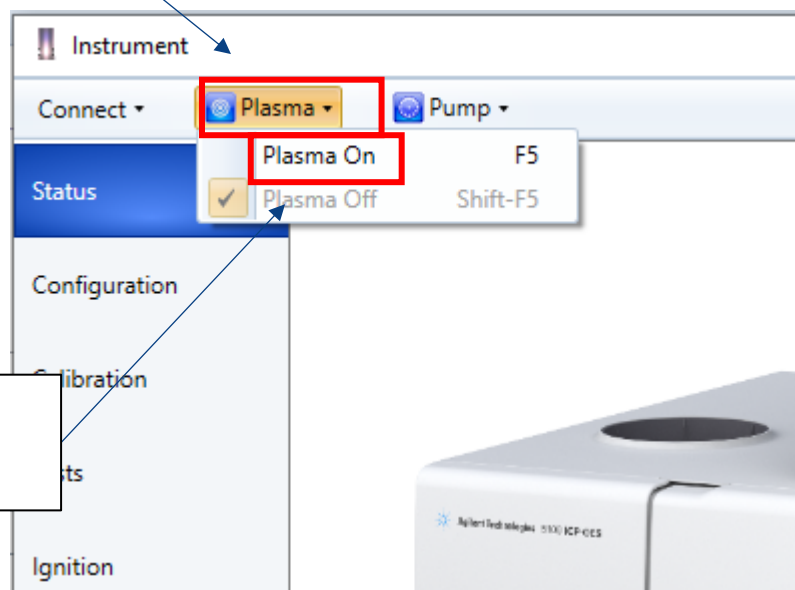
Step 2. Ignite Plasma

Step 2.1 Click '**Plasma**' button on the icon toolbars

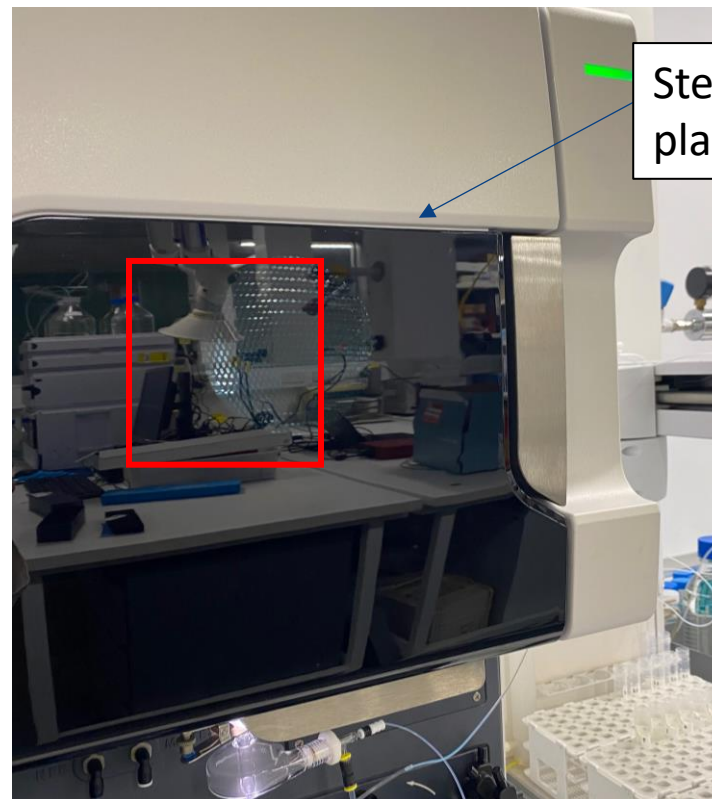
Step 2.2 Click '**Ignite plasma**' and this will take up to 60 seconds to complete.

Step 2.3 Check that the plasma has ignited through the view window

Step 1: Click on
'**Plasma**'



Step 2: Click
'**Plasma on**'



Step 3 Check the
plasma has ignited

IV. Perform Measurement

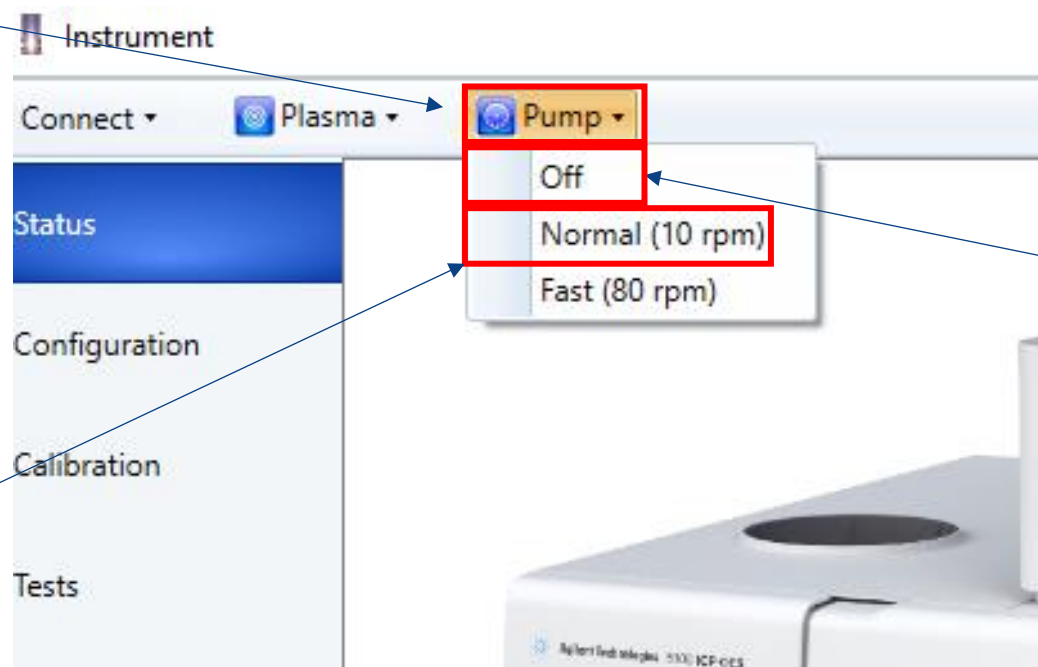
Step 2. Ignite Plasma

Step 2.4 Click on '**Pump**' in the top toolbar and turn on pump to '**Normal (10 rpm)**'. Ensure pump is flowing and turn '**Off**'

Step 1: Click on '**Pump**'

Step 2: Click '**Normal (10 rpm)**'

Step 3: Click on '**Off**'



IV. Perform Measurement

Step 2. Ignite Plasma

Step 2.5 Open the autosampler menu by clicking on '**Autosampler**' in the top toolbar.



IV. Perform Measurement

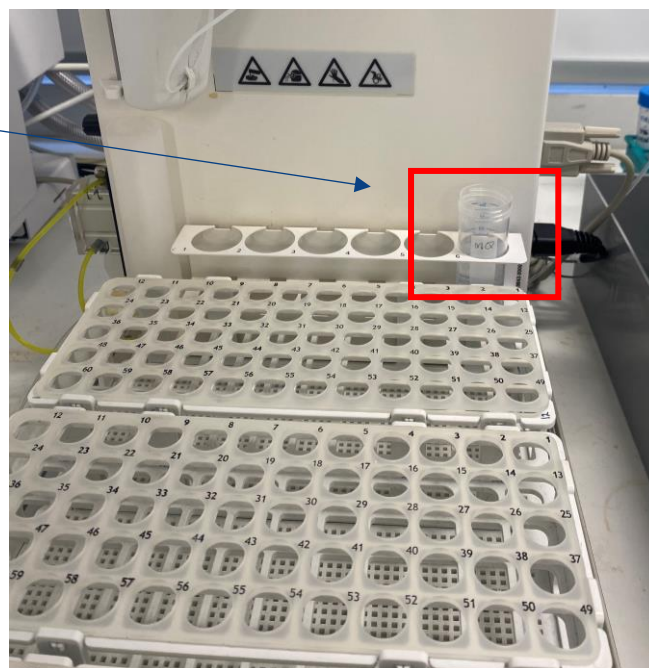
Step 2. Stabilise Plasma

Step 2.6 Put a fresh MilliQ water blank into the autosampler tray

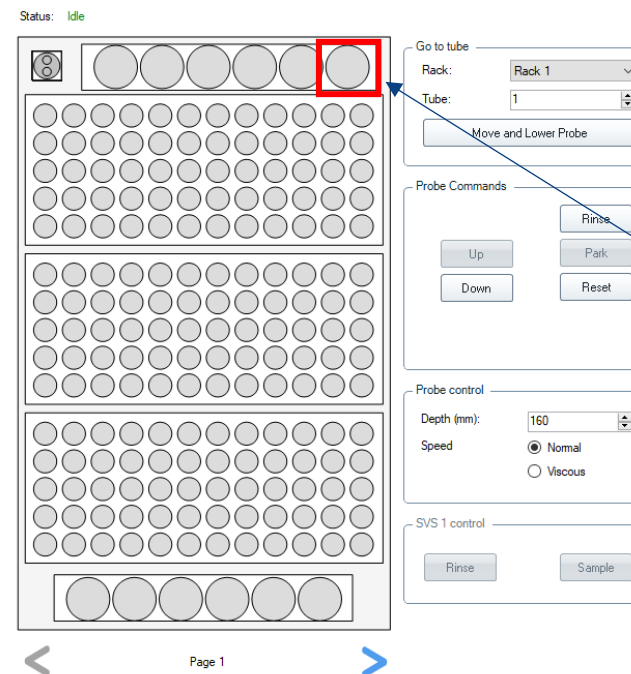
Step 2.7 Double click on the position which holds the milliQ water blank to move the autosampler into the MilliQ water.

NOTE: Plasma now needs to stabilise for **30 minutes** prior to analysis.

Step 1: Put in a fresh MilliQ blank



Autosampler



Step 2: Double click on the location of the blank

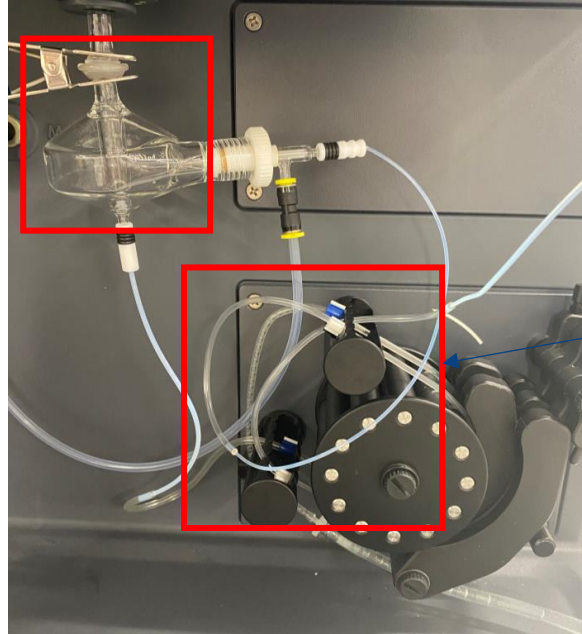
IV. Perform Measurement

Step 2. Stabilise Plasma

Step 2.8 Check that the nebuliser is producing a consistent and steady stream of the sample solution by looking at the glass bulb and the draining tubing.

NOTE if the nebuliser does not appear to be producing a steady flow contact **Yukie** or **Alex** for further assistance.

Step 1: Observe flow from the nebulizer and ensure it is free from pulsating



Step 2: Look at the draining tube with the blue clips and ensure the bubbles are steady and consistent

Step 3. Sample preparation

Step 3. Important sample preparation

- *Samples must be completely dissolved in an aqueous solution (either acid or base) which can be achieved via digestion using thermal or microwave method.*
- *All glassware used in ICP-OES preparation must be washed in an acid bath prior to use to avoid contamination. (The acid bath is located under the ICP-OES)*
- *All samples need to be filtered using 0.45 μm syringe filters.*

Many methods can be applied depending on samples. Details for specific sample preparation are not included in this material.

IV. Perform Measurement

Step 4. Starting the measurement

Step 4.1 Check wash solution to make sure it is full. Usually 2% nitric acid is used however if you need to change the wash solution due to factors like solubility rinse the tubing into a beaker and then transfer to a new wash solution

Step 1: Check
volume of the
washing solution



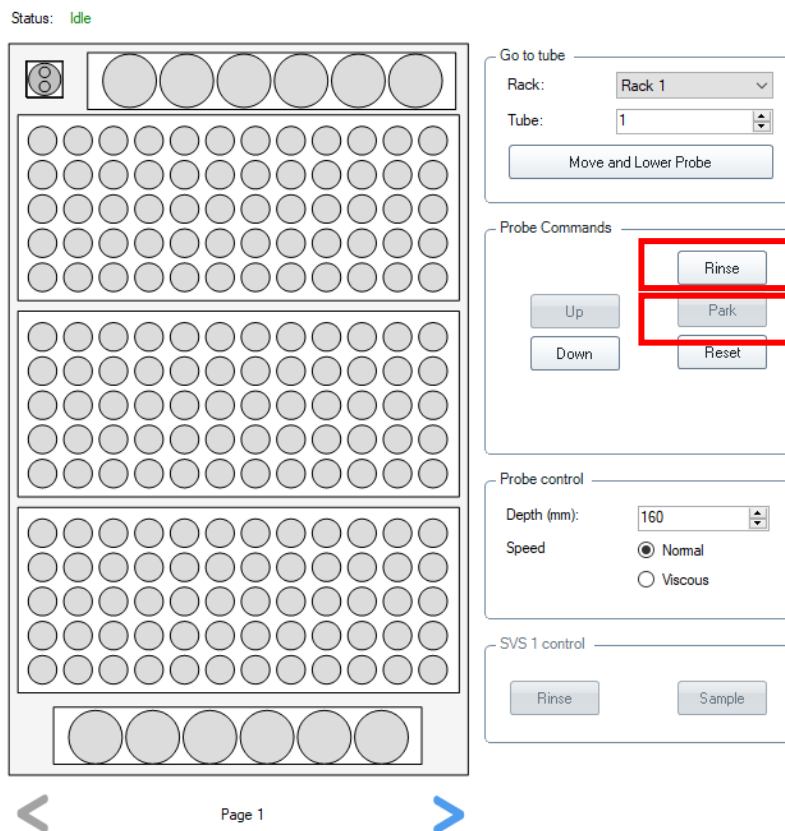
Step 2: Refill from
bottles labelled 2%
w/v HNO_3 if
necessary

IV. Perform Measurement

Step 4. Starting the measurement

Step 4.2 Move the autosampler to '**Rinse**' until the drain tubing starts to flow then return autosampler to '**Park**' in the '**Autosampler**' menu.

Autosampler



Step 1: Click on '**Rinse**'

Step 2: wait until the drain tubing starts to flow

Step 3: Click on '**Park**'

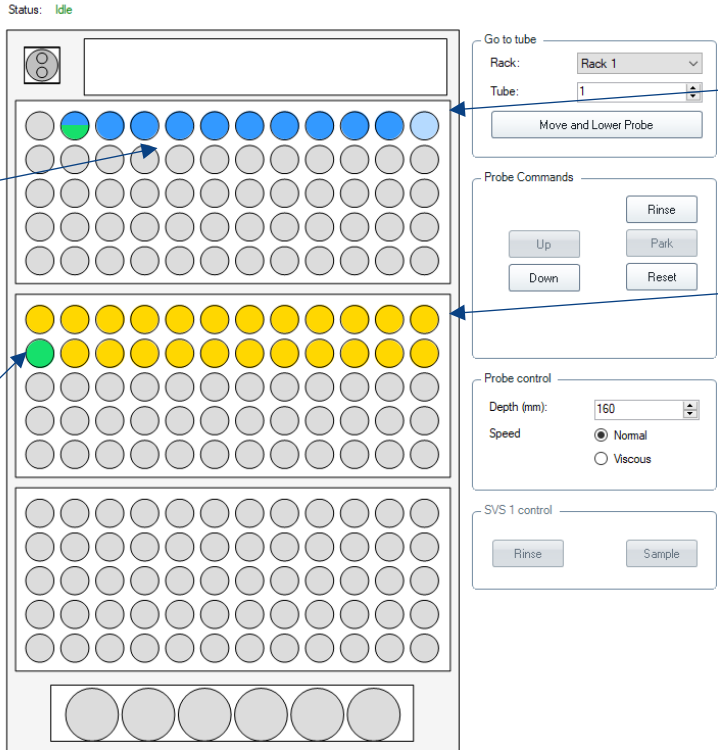


IV. Perform Measurement

Step 4. Starting the measurement

Step 4.3 Check sample locations match the sequence table, you can look at the '**Autosampler**' tab to compare position

Step 4.4 Ensure samples have enough volume for the number of runs, important to add up number of '**QQB**' and '**QQV**' in sequence table ie. If you have 4 QQB's you will need at least 8 ml. Account for 1.5-2 mL per analysis.



Dark blue indicates standards

Green indicates a quality control sample/standard ie a QQV, QQB or spike

Light blue indicates blanks

Yellow indicates sample

Go to tube
Rack: Rack 1
Tube: 1
Move and Lower Probe

Probe Commands
Rinse
Up
Down
Park
Reset

Probe control
Depth (mm): 160
Speed: ☒ Normal ☐ Viscous

SVS 1 control
Rinse
Sample

Legend:
Blank (light blue)
Standards (dark blue)
Sample (yellow)
QC (green)

Page 1

IV. Perform Measurement

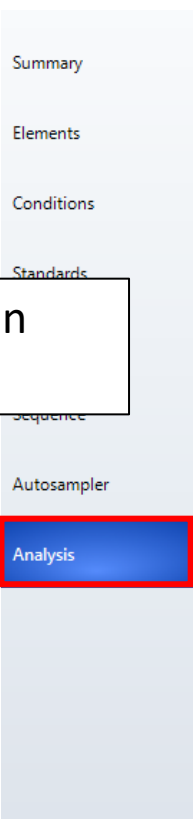
Step 4. Starting the measurement

Step 4.5 Check box to chose all samples in the sequence table for analysis

Step 4.6 Click '**Run**' and watch first sample to ensure the run has begun and the autosampler is operational.

Step 4.7 Relax while the machine takes over

Step 1: Click on
'**Analysis**' tab

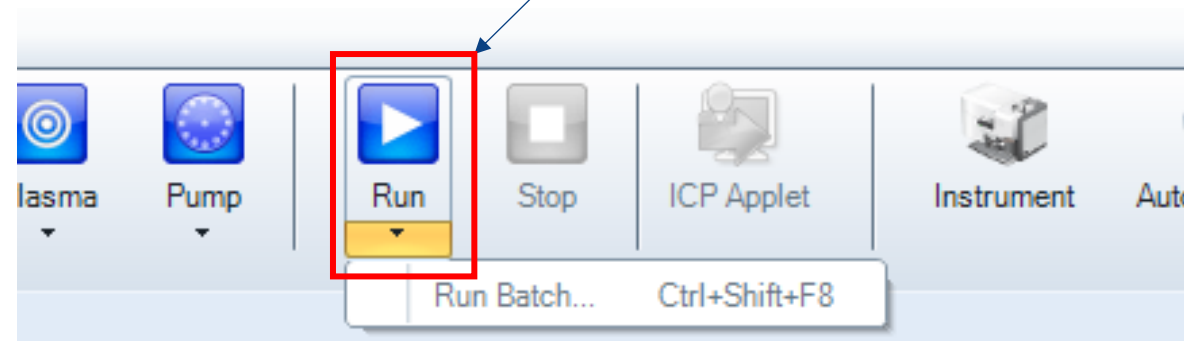


Step 2: Tick the
box to select all
samples for
analysis

Concentration Track Analysis Sort Results... Hide Column

	Rack:Tube	Solution Label	Mn 257.610 nm ppm	Mn 259.372 nm ppm	Mn 260.568 nm ppm
☒	S1:1	Blank	0.00	0.00	0.00
☒	S1:2	0.1 ppm	0.10	0.10	0.10
☒	S1:3	0.25 ppm	0.25	0.25	0.25
☒	S1:4	0.5 ppm	0.50	0.50	0.50
				1.00	1.00
				2.50	2.50
				5.00	5.00
				10.00	10.00
				25.00	25.00
				50.00	50.00
				100.00	100.00
☒	S2:6	200 ppm	200.00	200.00	200.00
☒	1:1	Sample 1			
☒	1:2	Sample 2			
☒	1:3	Sample 3			
☒	1:4	Sample 4			

Step 3: Click on
'**Run**'



IV. Perform Measurement

Step 5. SHUTDOWN after measurement is complete

Once the measurement is completed:

*Step 1. Move autosampler to park via the '**autosampler**' menu*

*Step 2. Click '**Plasma**' button on the toolbars > Click '**Plasma Off**' > Exit the software*

Step 3. Unlock arms and release tension sample and drain tubing;

Step 4. Unlock arms and release tension an autosampler tubing;

Step 5. Ensure plasma has been turned off for a few minutes and turn off the water chiller;

Step 6. Ensure all samples have been removed from the autosampler

DO NOT switch off any button on the instrument, nitrogen or argon gas and the exhaust fan