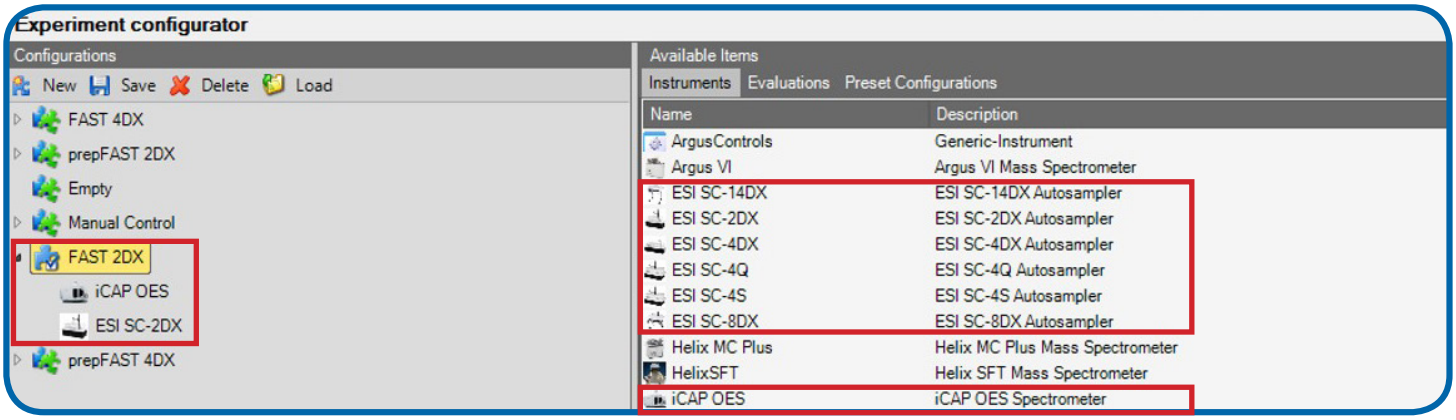
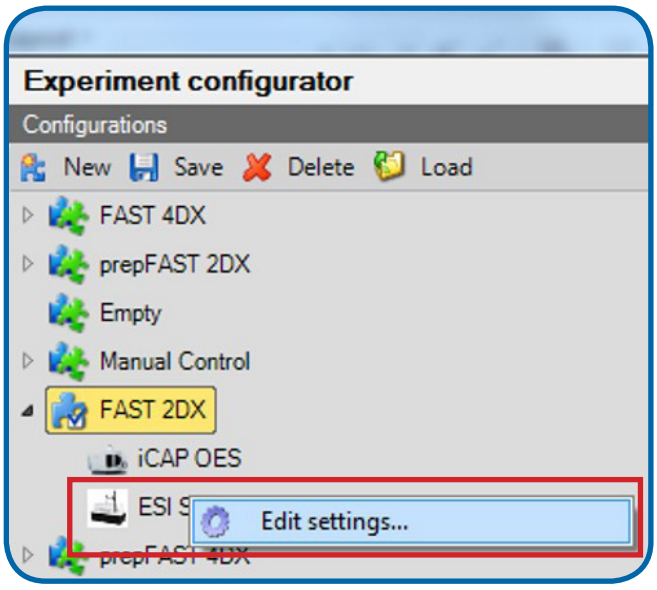
FAST for iCAP
Getting Started/Method Guide

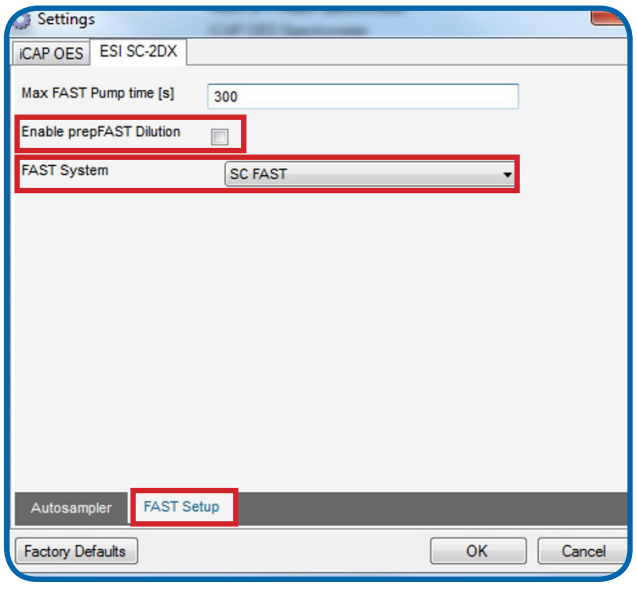
1 Configuration



Step 1. Open the Configurator software.
Step 2: Click the Experiment Configurator tab.
Step 3. Create a new configuration.
Step 4. Drag the iCAP instrument into the configuration.
Step 5. Drag the ESI autosampler being used into the configuration.
Step 6: Save configuration.

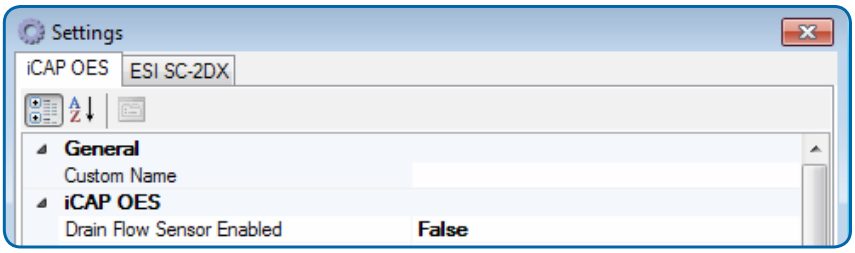


Step 7. Right click the configured ESI autosampler and then select Edit settings.



Step 8. Confirm “Enable prepFAST Dilution” box is **unchecked** and select “SC FAST”. Close the window by selecting OK and save changes in the Configurator.

Step 9. Click the iCAP OES tab. Change “Drain Flow Sensor Enabled” to “False.”



2 Creating the Solutions

Five solutions are needed to operate the prepFAST system. The solutions are:

1. **Carrier:** The carrier solution is used to push the sample from the P7 valve to the instrument.
2. **Internal Standard:** The internal standard is mixed with the sample as it flows into the instrument.
3. **Rinse:** This solution is used to clean the sample probe and loop of the P7 valve.

All solutions should be prepared in order to meet the requirements of your analysis. For initial installation, it is recommended to make all solutions with 2% Nitric Acid.

To prepare the Internal Standard Solution

1. Prepare the FAST Internal Standard solution by filling the appropriately labeled bottle with nitric acid at a concentration that matches the matrix of your samples. Make the solution from acid and ultrapure water, wich are suitable for trace elemental analysis by ICP.
2. Place the tubing labeled FAST Internal Standard in the bottle.
3. Keep in mind that the internal standard is also diluted by a factor of 5, if the FAST methods are used, which were provided with the instrument.

To prepare the Carrier solution

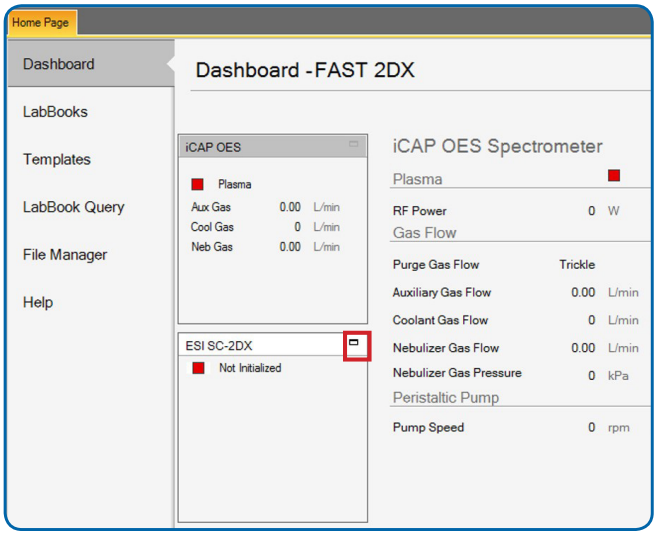
1. Prepare the Carrier solution by filling the appropriately labeled bottle with nitric acid at a concentration that matches the matrix of your samples. Make the solution from acid and ultrapure water, wich are suitable for trace elemental analysis by ICP.
2. Place the bottle with the Carrier solution in the FAST bottle holder.

To prepare the Rinse solution

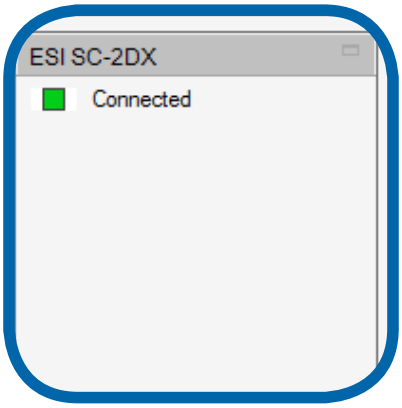
1. Prepare the Rinse solution by filling the appropriately labeled bottle with nitric acid at a concentration that matches the matrix of your samples. Make the solution from acid and ultrapure water, wich are suitable for trace elemental analysis by ICP-OES.
2. Place the tubing labeled RINSE into the R1 bottle. Note the solution can be used to rinse the probe and the sample loop lines.

3 Initialize *FAST*

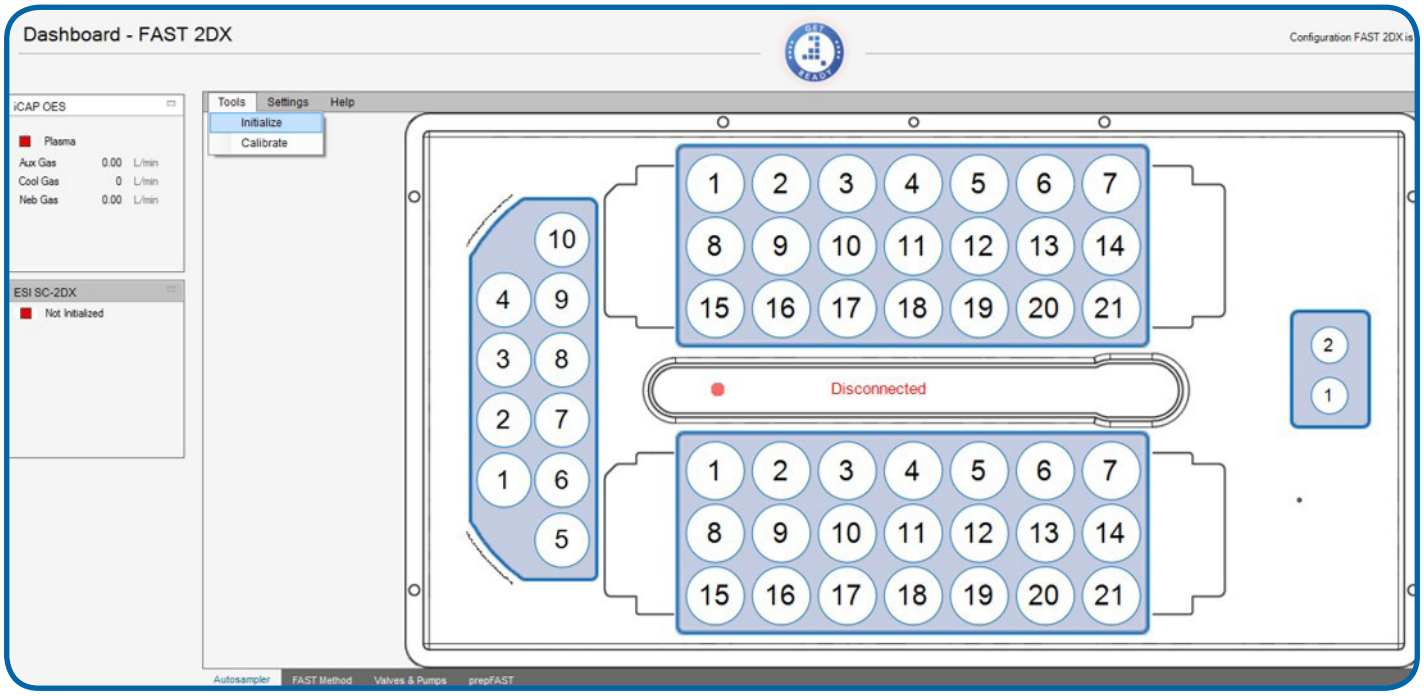
1. Open the Qtegra software.
2. Select the newly created *FAST* configuration and click “OK.”



3. From the Dashboard, open the autosampler display by clicking the headline, seen in the figure below.



4. If the autosampler does not already display Connected and Initialized in the Dashboard, select the Tools > Initialize. If the autosampler is connected properly and all drivers are installed, the state should change after a couple of seconds. Otherwise, please power cycle the complete autosampler and the FAST system, reload the Qtegra configuration and try again.



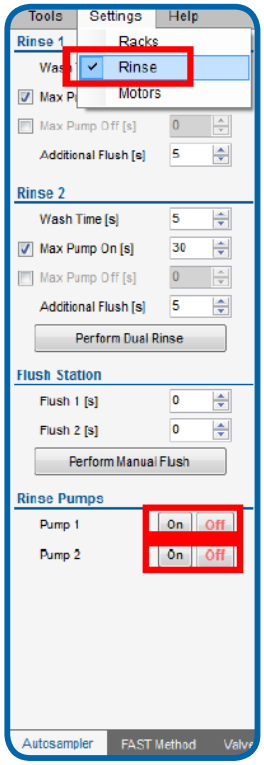
5. The autosampler should be located at the Home Posititon above the first rinse station. If this is not the case, select Tools > Calibrate. A wizard will guide you through the calibration procedure. If the rinse station position was not located correctly upon initialization and calabration was required, it is recommended to check if the vial positions are located correctly. This can be checked using the same calibration procedure. Alternatively, this step can be repeated if the autosampler does not go into the vials properly during the acquisition of a LabBook.

To prime the rinse station

1. In order to initially prime the rinse station with rinse solution, select Settings > Rinse to open the Rinse control window.

2. Turn on pump one by clicking “On” for pump 1. As soon as you see there is solution at the top of the rinse stations, switch the pump “Off” again.

3. When the Rinse solution matrix is changed, this procedure should be repeated in order to flush out the previous Rinse solution.



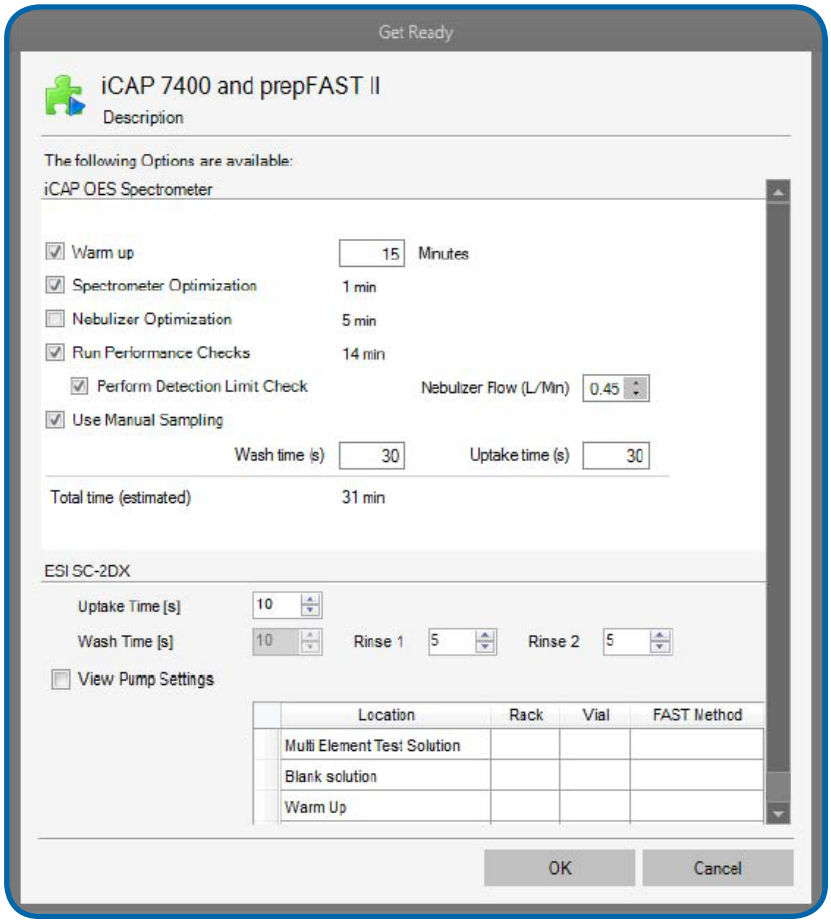
5 Using Get Ready

As the FAST-based system is a discrete sample introduction system that provides steady signals over a fixed time period, the “Get Ready” process for the FAST is different to that for a continuous sample introduction system such as a traditional autosampler.

To use Get Ready

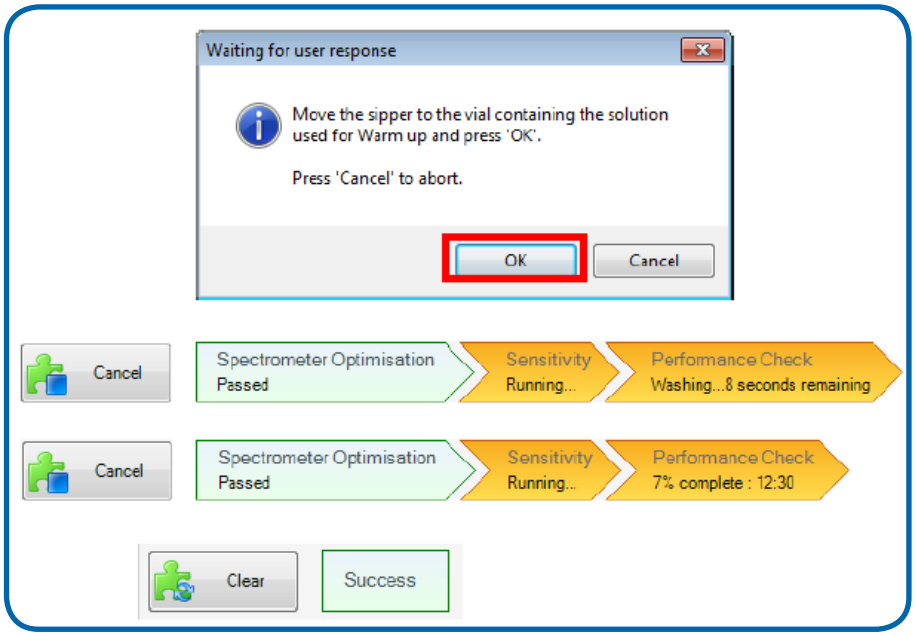
1. From the Qtegra ISDS Dashboard, click the “Get Ready” button, which opens a dialog for the startup of the instrument.

2. Enter the **Warm up time** (if required), the **Wash Time** and the **Uptake time** (for the supplied tubing, 30s should be sufficient) you want to use for the analysis.
3. Select the **Spectrometer Optimization** or **Nebulizer Optimization** if you want them to be performed automatically after the Warm Up.
4. Select the **Use Manual Sampling** checkbox as Permormance Checks conducted by the software require a stable signal, which is longer than the one the FAST system can provide.

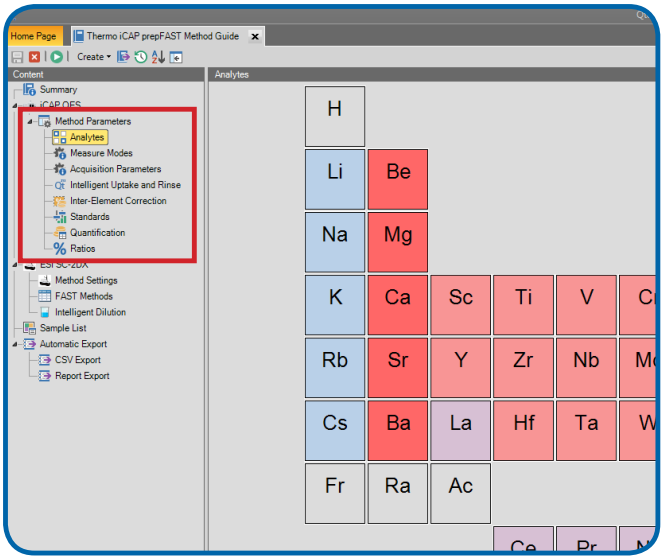


To run the Get Ready process

1. Make sure the FAST sample out line is connected to the nebulizer and that all waste tubings are fitted to an appropriate waste bottle.
2. Make sure that the carrier and the spray chamber drain pump tubing are aligned correctly on the peristaltic pump.
3. Click “OK.”
4. The plasma will be started, and after any defined warm up time and optimization procedures, the process performacnce checks will be acquired. During these actions, you will be asked to provide the corresponding solutions for each step. Use the carrier tubing, which goes from the peristaltic pump through the FAST valve into the iCAP ICP-OES to supply these solutions.
5. Once the performance checks are finished, the state of the instrument should be ready and the analysis can be started.



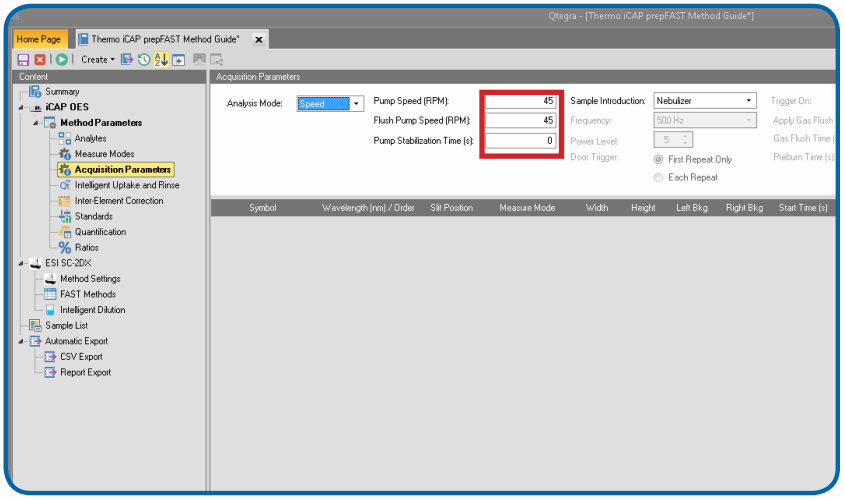
6 Setup LabBook - Method



Step 1. Open Qtegra software and create a LabBook with the desired analytes. For further Information regarding the setup of a method, please refer to “Qtegra for iCAP 7000 series ICP-OES software manual.”

Step 2. Select all method parameters: analytes, dwell time, sweeps channels, measurement mode, standards, etc.

Step 3. For iCAP 6000 series and older, use a pump setting of 15 RPM. For iCAP 7000 series use a pump setting of 50 RPM.



Carrier Peripump Tubing		iCAP 6000 series (rpm)	iCAP 7000 series/DXI Pump Setting (rpm)
orange	green	60	
green	yellow	45	
orange	yellow	35	
white	yellow	25	70
orange	white	20	60
black	black	15	50
orange	orange	11	40

FAST Method Length Calculation

Calculate the estimated measurement time using the formula:

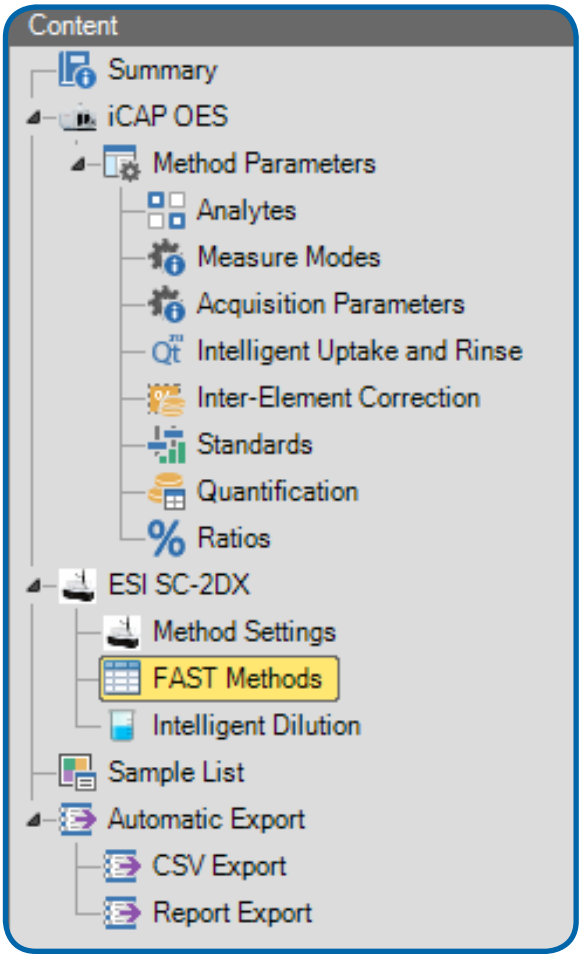
Measurement time=
(Total Low and High WL Maximum integration time) x Repeats x 2

For example, suppose a method uses the follow settings: 15 second UV axial, 5 second UV radial, 10 second Visible axial, and 5 second visible radial. To estimate the analysis time the calculation is as follow:

(15+5+10+5) x 3 x 2= 210s

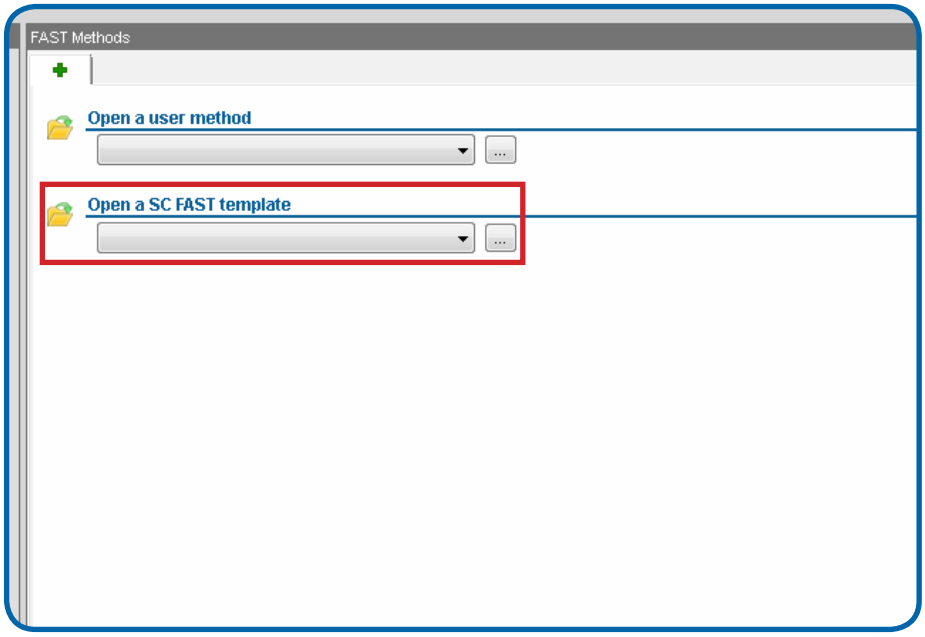
Table 1			
Estimated Analysis Time (s)	**Loop Size (mL)**	**iCAP Uptake Time (s)**	**iCAP wash Time (s)**
10-25	0.5	15	10
25-60	1.0	17	12
60-80	1.5	18	13
80-120	2.0	20	15
120-160	2.5	22	17
160-210	3.0	25	17
210-300	4.0	29	17

Notice: All timings are based off using a Black-Black peripump at a flow rate of 50 rpm (Micro Peristaltic Pump) or 15 RPM (Large Peristaltic pump).
If a longer analysis time is needed that the times shown in the table, the peristaltic pump flow rate should be slowed accordingly. For example if 340 seconds of analysis time is necessary, the 3.0 mL loop should be used with a pump speed of 25 RPM (micro pump) or 7 RPM (large pump).

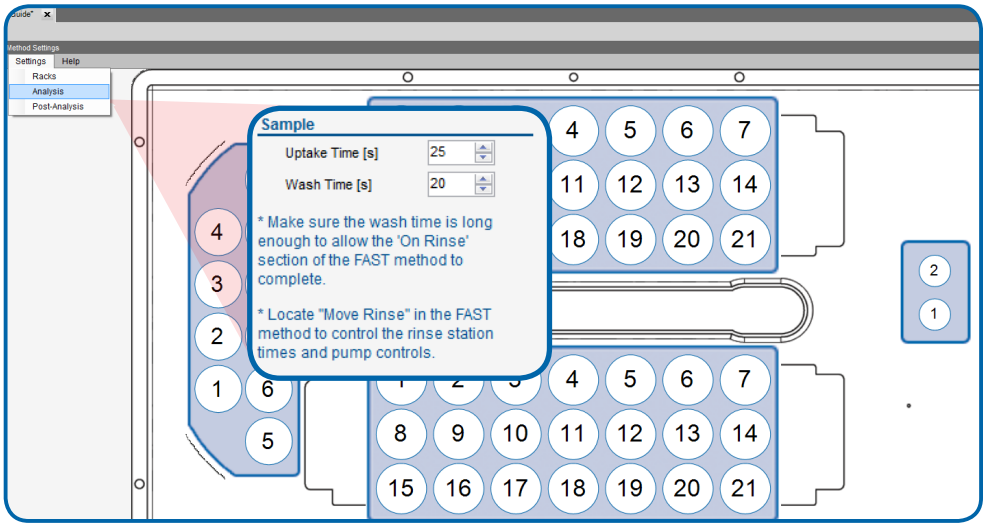


Step 4. Set the desired FAST method parameters in the Content menu.

Step 5. Click on the “New user method from a FAST template” drop down menu. Open a FAST method in the Template Methods drop down. Select the appropriate FAST method according to the loop size being used (e.g. FAST_1.5mL loop).

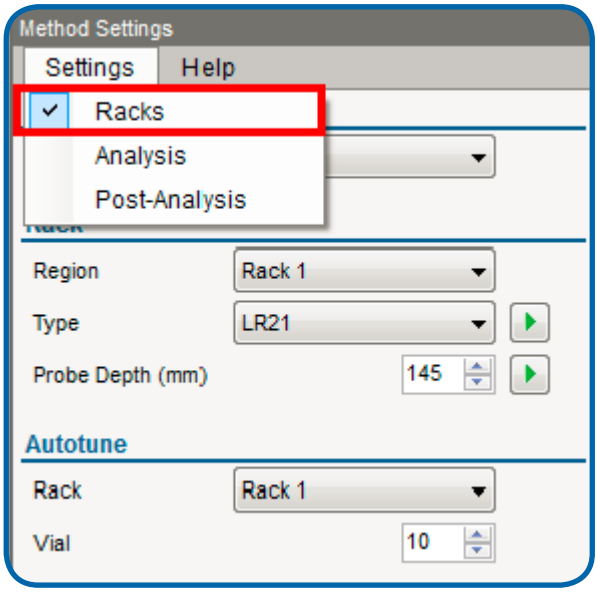


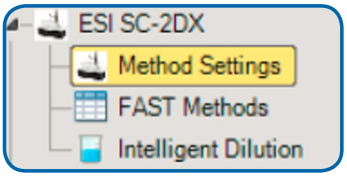
Step 6. Open the method settings window in the top left corner select “Settings → Analysis” and enter the uptake and wash time from the table.



Rack setup

Step 7. In the Method Settings Window, go to settings and select “Racks” and set the correct down height and rack size for what is being used on the autosampler.





9 *FAST* Troubleshooting Table

Problem:	
The Autosampler does not respond when the ICPMS sends it to a vial location.	
Possible Causes	**Possible Solution**
The autosampler is not powered on.	Make sure the autosampler is plugged in and turned on. Initialize the Autosampler.
The autosampler communication is not configured properly.	Restart the computer. Start the ICPMS software, and initialize the autosampler.
Problem:	
The probe moves to the correct vial but has no further movement. No signal is observed.	
Possible Causes	**Possible Solution**
No FAST method is loaded.	Open the FAST tab in the ICP MS software. Locate the proper FAST method. Make sure the correct FAST method is labeled "Host Active."
FAST method is "Inactive."	Make sure the correct FAST method is labeled “Host Active.”
Problem:	
The probe goes to the proper vial and then to the rinse station, but the rise in signal is not as expected.	
Possible Causes	**Possible Solution**
The vial does not contain the proper standard or concentration of standard.	Ensure that a standard with appropriate levels of analytes is in the vial location.
The peristaltic pump tubing or rate in the ICPMS software is set incorrectly.	Double check the peristaltic pump to tubing and speed in the ICPMS software.
The peristaltic pump tubing is worn out, disconnected, or is not connected properly.	Check all tubing quality and connections, especially the carrier line (black/black). Refer to the FAST diagram to verify the correct configuration.
The timers are not set according to the installed loop size.	Check what loop is installed on the valve. Varify the timers in the FAST method are appropriate for the loop size being used.
The sample introduction system contains non-genuine or non-standard ESI parts.	Remove non-genuine or non-standard parts and replace them with genuine ESI parts.
No liquid is being removed from the vial.	Use the FAST diagram to check the vacuum plumbing. Make sure that the FAST method contains the line: “On Probe Down | FAST Vacuum 1 - On.” Navigate to the FAST menu, In the autosampler settings of the ICP MS software. While feeling the vacuum port on the exterior of the autosampler, turn Vacuum 1 on and off. If no virabtion is felt from the pump, contact customer support. Check for clogged of kinked capillaries. If the pump turns on, verify the pump rate is at least 30 mL/min. Replace the Vacuum line.
The valve is not switching properly.	Make sure the valve cable is secure both at the back of the autosampler and at the back of the valve module. Verify that it is plugged into the V1 port on the back of the autosampler. Navigate to the FAST menu, In the autosampler settings of the ICP MS software.Toggle valve 1 between Load and Inject. Listen for the sound of the valve toggling, and verify that the Load and Inject lights are switching. If you do not hear the valve toggling, contact Technical Support. If you hear a valve toggle but one or both of the Load and Inject lights near the valve are not turning green as you toggle between Load and Inject, unplug the valve cable from the autosampler, toggle between Load and Inject 5 times, plug the cable back in to the autosampler, and toggle between Load and Inject 10 times. If both the Load and Inject lights are not turning on after this procedure, contact Technical Support.

There is an obstruction in the valve.	Perform the valve cleaning procedure found in the Maintenance section of the ESI Help file. Navigate to the Autosampler window and open the Help tab (Help→Manual).
The valve is damaged or beyond its useful life.	Determine if the rotor and/or stator are damaged, and replace as needed.

Problem:	
Washout is not fast enough.	
Possible Causes	**Possible Solution**
The peristaltic pump tubing or rate in the ICPMS software is set incorrectly.	Double check the peristaltic pump tubing and speeds in the ICPMS software.
The peristaltic pump tubing is worn out, disconnected, or is not connected properly.	Check all tubing quality and connections, especially the carrier line (black/black). Refer to the FAST diagram to verify the correct configuration.
The element that is showing carry-over is more prone to carryover than typical elements, e.g., B, Sb, Au, Hg	Increase wash time or add a suitable chemical modifier to the carrier solution to increase washout for that element.
There is an obstruction in the valve.	Perform the valve cleaning procedure found in the Maintenance section of the ESI Help file. Navigate to the Autosampler window and open the Help tab (Help→Manual).
The valve is damaged or beyond its useful life.	Determine if the rotor and/or stator are damaged, and replace as needed.

Problem:	
When running samples or standards, RSDs are always too high.	
Possible Causes	**Possible Solution**
The peristaltic pump tubing or rate in the ICPMS software is set incorrectly.	Double check the peristaltic pump tubing and speeds in the ICPMS software.
The peristaltic pump tubing is worn out, disconnected, or is not connected properly.	Check all tubing quality and connections. Refer to the FAST diagram to verify the correct configuration.
The uptake time is too short.	Verify all the settings for the loop size chosen for the method. View the replicate data; if the first replicate is consistently lower than other replicates, the total flush time/read delay/uptake time is too short. If necessary, increase this time in the ICPMS method appropriately.
The loop is too small for the method.	Look at the replicate data. If the last replicate is consistently lower than the previous replicates, the sample loop is running out of sample before the measurement is completed. Verify that the correct loop size is installed, and verify all other parameters. If necessary, increase the loop size and update other settings accordingly.
The dwell/integration time is too short for the desired RSDs.	Increase the dwell/integration time to improve statistics. Install the proper loop and update all settings accordingly.

Problem:	
RSD's are usually good but occasionally very bad.	
Possible Causes	**Possible Solution**
The "Max Vacuum Time" is too short.	Navigate to the FAST menu, In the autosampler settings of the ICP MS software. Set the "Max Vacuum Time" to 300 s.
The wash time in the ICPMS method is set to 0 s.	Set the wash time to at least 15 s or add 5s to your correct time.
The sample read time is more than 300 s	Set the "Max Vacuum Time" to be 30 s longer than the total sample-to-sample time.
A "Vacuum1 Off" command has been added to the FAST method.	Remove the line with "Vacuum1 Off" from the FAST method.

The autosampler was sent to a location without a sample, or the sample ran out during the sample loading step.	Ensure all vials have an even and adequate amount of sample.
The autosampler is missing vials.	Verify that the correct racks are configured in the autosampler software. Verify the autosampler calibration and rack calibrations.

Problem:	
Carryover is always too high.	
Possible Causes	**Possible Solution**
There is an obstruction in the valve.	Perform the valve cleaning procedure found in the Maintenance section of the ESI Help file. Navigate to the Autosampler window and open the Help tab (Help→Manual).
The valve is damaged or beyond its useful life.	Determine if the rotor and/or stator are damaged and replace as needed.
The wash time is too short.	Verify that the washout time is at least 15 s. The FAST system provides >2000x washout under standard conditions. If this level of washout is insufficient or if memory-prone elements are present, increase wash time in the ICPMS method.
The samples or standards are very concentrated and there is carryover in the loop or valve.	Implement the LoopRinseExample.txt FAST method as described in the Contact ESI Support for customer prep*FAST* methods.

Problem:	
The internal standard signal is not as high as expected or is unstable.	
Possible Causes	**Possible Solution**
The ICPMS torch is not aligned.	Place the carrier probe into tuning solution, and perform autotune.
The peristaltic pump tubing or rate in the ICPMS software is set incorrectly.	Double check the peristaltic pump tubing and speed in the ICPMS software.
The peristaltic pump tubing is worn out, disconnected, or is not connected properly.	Check all tubing quality and connections, especially the internal standard line (orange/green). Refer to the FAST diagram to verify the correct configuration.
The ICPMS is not tuned properly.	Tune the ICPMS using the manufacturer's recommended procedure.
The sample gas is not flowing or is not connected properly.	Verify that the sample gas is flowing through the nebulizer. Check for leaks at the nebulizer and at the ICPMS gas outlet.
There is an obstruction in the nebulizer.	Turn off the ICPMS. Remove the nebulizer line from the back of the nebulizer. Turn on the sample gas flow. Loosen the nut that holds the nebulizer in place, and gently remove the nebulizer. Place a gloved finger firmly over the tip of the nebulizer for a few seconds. Release and repeat. Replace the nebulizer, gently tighten the nut (do not over tighten), and replace the nebulizer line. Restart the plasma and check for stability and sensitivity.