

LSX-266 G2 Compatibility: Thermo Fisher Scientific iCAP 6500



Introduction

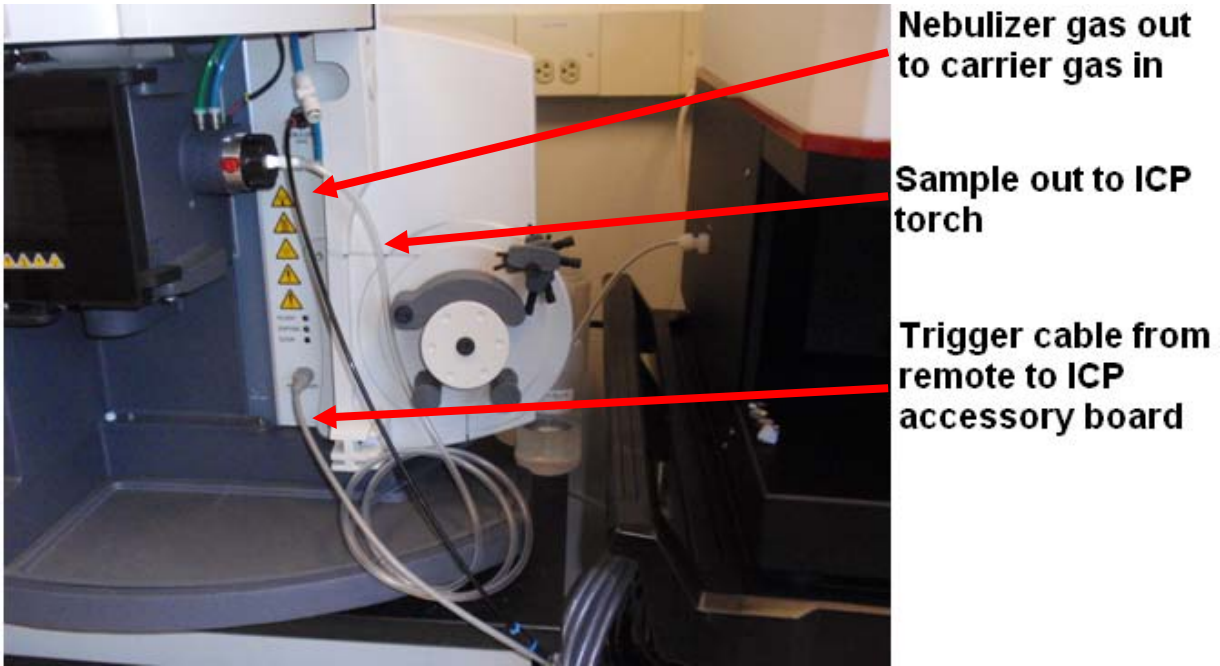
The CETAC LSX-266 G2 laser ablation system features the latest in optical design technology to target areas of interest, while employing the most powerful UV laser in its class to ablate even the most challenging of samples. The intuitive and powerful DigiLaz™ G2 software allows the user to easily create and save methods for all types of analyses. Precise targets can be set for analysis, including single point, multi-point, line scans and depth profiling. Alternatively, simply draw a pattern over the area to be analyzed and ablate.

The iCAP 6500 optical ICP offers sensitive analysis with full wavelength selection and interference handling, ideally suited to running the laser ablation system.

Interfacing

Three connections between the instrumentation are required for operation of the LSX-266 G2 with the Thermo Fisher iCAP 6500:

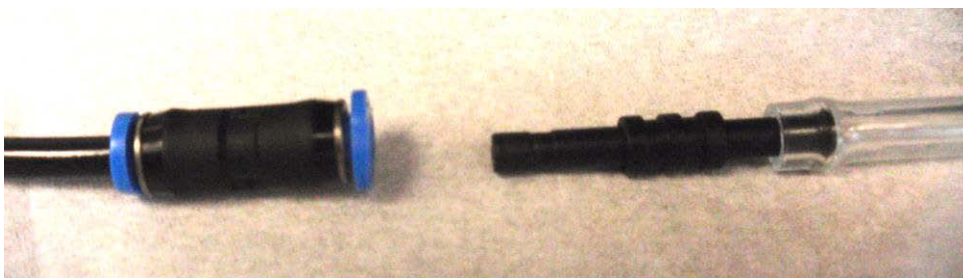
1. Sample out from the laser to the iCAP torch
2. Nebulizer gas out from the iCAP to the laser Carrier Gas inlet
3. Remote Trigger between the laser and the iCAP (requires accessory board provided and installed by Thermo Fisher Scientific)



To interface the torch, attach the adapter to the end of the SAMPLE OUT tube from the laser ablation system. Connect this directly to the base of the iCAP torch. Make sure that the connector is fully inserted to avoid air leaks and plasma stability issues.

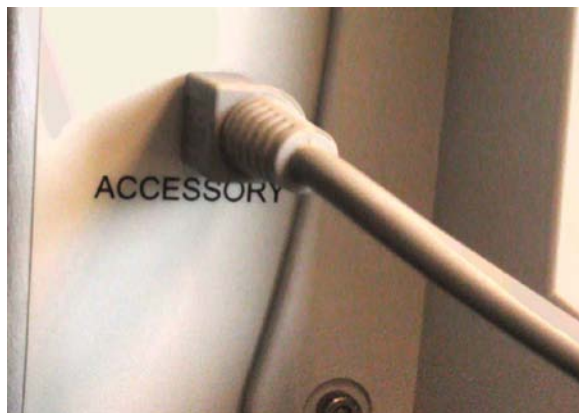


The iCAP nebulizer gas is used as the carrier gas for the laser. Push the adapter into the tubing connected to the carrier gas on the laser and plug into the push fit connector on the nebulizer gas line.

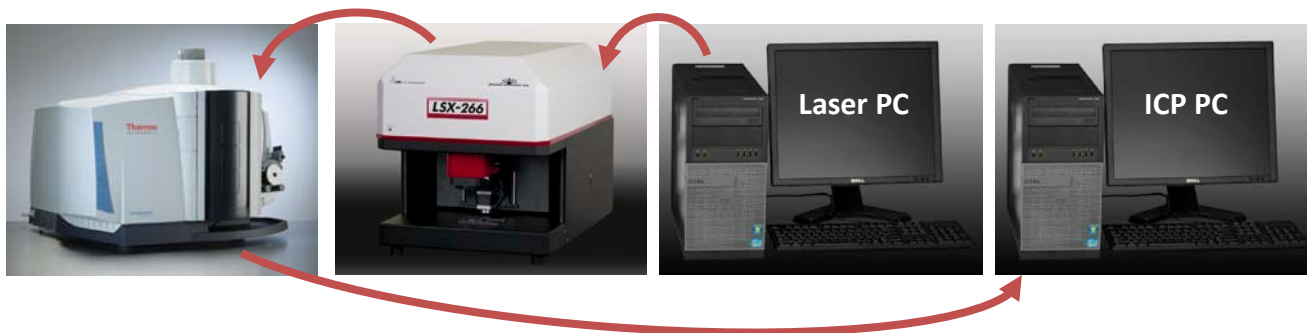


For control of the laser the iCAP must be fitted with an Accessory Interface Board. If this is carried out after initial purchase of the instrument it must be fitted by an approved Thermo Fisher Scientific service representative.

Plug the trigger cable supplied with the laser into the accessory socket located to the left of the sample pump on the iCAP; as shown below.



The following image shows the flow of communication/ trigger.



Note – At the start of a measurement the iCAP will provide a trigger to the laser and a message “waiting for trigger” will be displayed in the software.

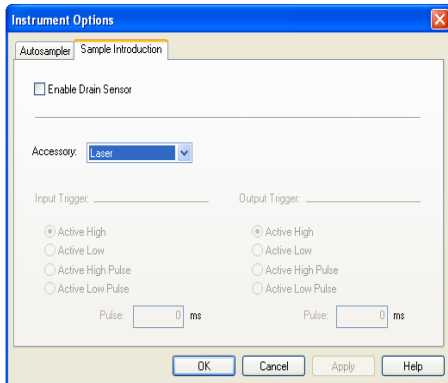
Not all types of laser can use this trigger and the ablation must be started manually. The laser then sends a trigger signal to the iCAP to enable data acquisition to start.

At the end of the data acquisition the iCAP will check the status of the laser before proceeding to the next measurement. The trigger signal must still be present while the status check is carried out, hence the time of the ablation must be 2-3 seconds longer than the data acquisition time.

iTEVA Configuration

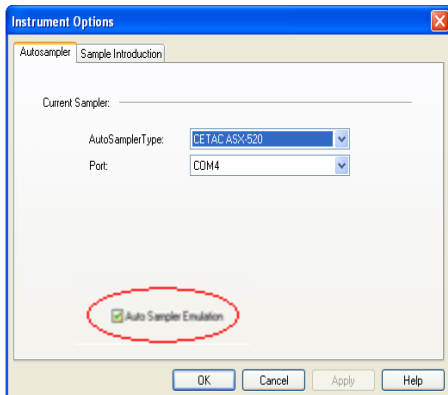
The iCAP must now be configured to use the Laser in place of the normal nebulizer.

1. In the iTEVA Control Center click on the Tools Menu and select Instrument Options.
2. Click on the Sample Introduction tab change the assessor to “Laser”, then un-tick the drain sensor option.



Note – For laser ablation the drain sensor must be disabled, to prevent the plasma from being shut down, the Drain Sensor must be enabled again if solutions are to be analyzed after use with the laser.

3. Return to the autosampler tab and select the “Autosampler Emulation” option. This will allow automated runs to take place without checking if an autosampler is actually present.

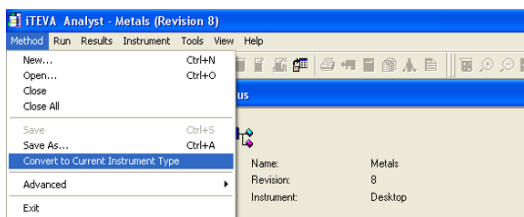


Note – If liquid samples are to be measured with an autosampler after analysis with the laser then the “Autosampler Emulation” option must be deselected otherwise communication with the autosampler will not be possible.

Method Setup

If a new method is to be used then click on “New” in the Method Menu and enter the required elements and wavelengths from the periodic table. Other parameters are described below.

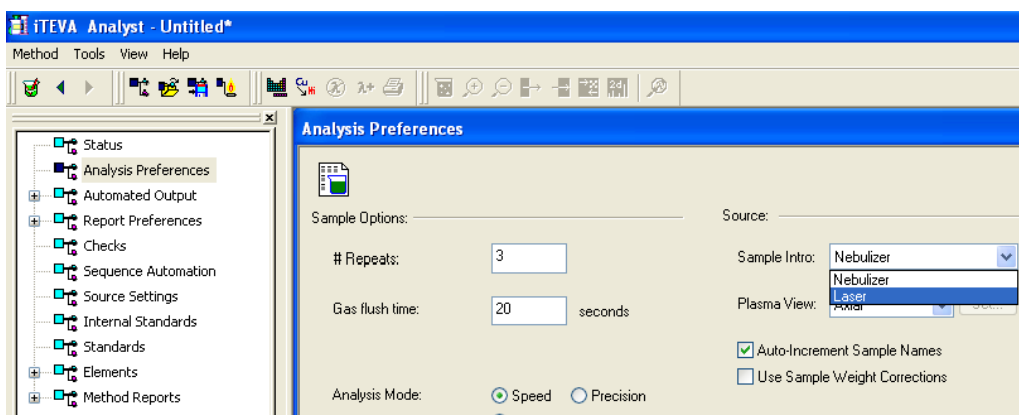
If an existing method is to be used for laser ablation, open the method in the normal way then select the Method Menu and click on “Convert to current Instrument type”. Save the method again with a new name to indicate use with a laser.



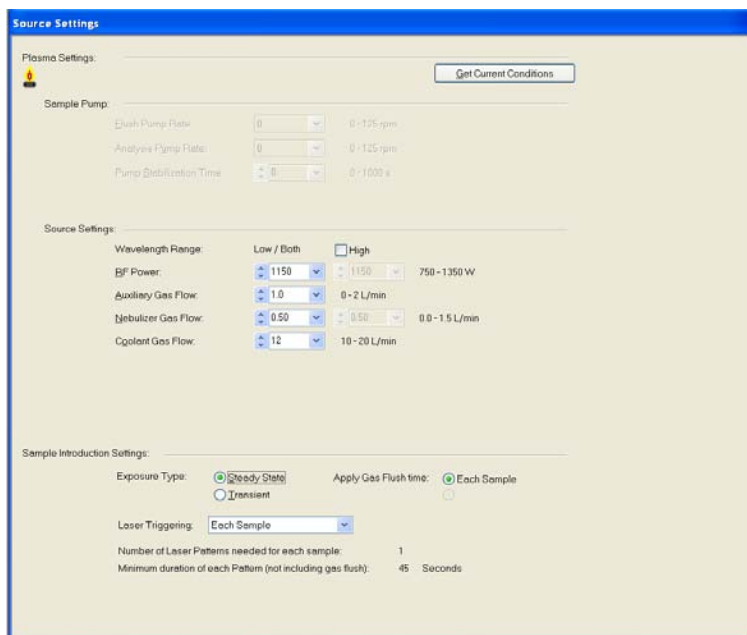
Steady State Analysis

It is possible to measure data in several different ways, for “bulk” analysis the steady state signal approach is most often used. In this mode the laser will continue to ablate material during the entire measurement time of the iCAP. See the note on triggering above.

1. Click on the “Method” tab in Analyst and select the Analysis Preferences option. Set the sample introduction option to “Laser” and set a delay time for the gas flush to allow the ablated material to reach the plasma prior to the start of the measurement, this will be different for each instrument setup and may need to be adjusted later on (see time scan analysis).



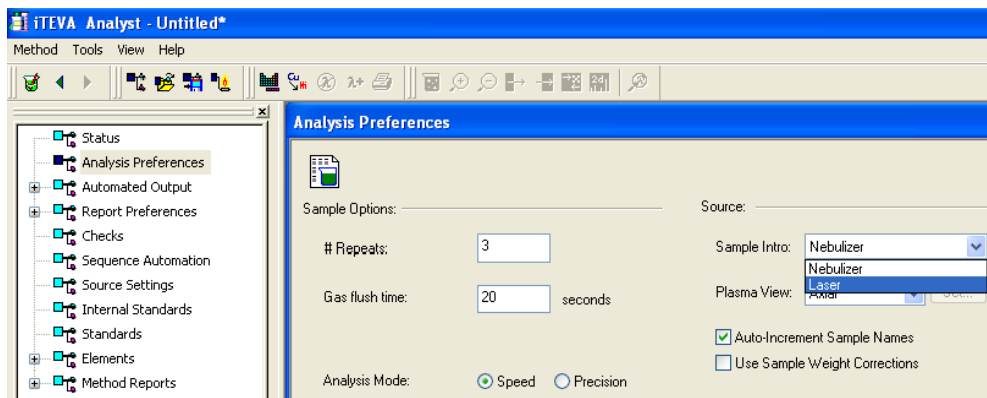
2. Select the Source Settings option, set the exposure type to “Steady State”, laser triggering to “Each Sample” and Apply Gas Flush to “Each Sample”.
The iCAP measures data in a number of sub exposures eg Axial or Radial View, Low (167-230nm) or High (230-847nm) wavelength range. For steady state analysis it is only necessary to wait for the gas flush for each sample.



Transient Analysis

Open an existing method or create a new method as described above.

1. Click on the “Method” tab in Analyst and select the Analysis Preferences option. Set the sample introduction option to “Laser” and set a delay time for the gas flush to allow the ablated material to reach the plasma prior to the start of the measurement, this will be different for each instrument setup and may need to be adjusted later on (see time scan analysis).



2. Select the Source Settings option, set the exposure type to “Transient”, laser triggering to “Each Sub Exposure” and Apply Gas Flush to “Each Sub Exposure”. The iCAP measures data in a number of sub exposures, Axial or Radial View, Low (167-230nm) or High (230-847nm) wavelength range. For transient analysis it is necessary to trigger the laser and wait for the gas flush for each sub exposure.

Source Settings

Plasma Settings: Get Current Conditions

Sample Pump:

Flush Pump Rate: 0 0 - 125 rpm

Analysis Pump Rate: 0 0 - 125 rpm

Pump Stabilization Time: 0 0 - 1000 s

Source Settings:

Wavelength Range: Low / Both High

RF Power: 1150 1150 750 - 1350 W

Auxiliary Gas Flow: 1.0 0 - 2 L/min

Nebulizer Gas Flow: 0.50 0.50 0.0 - 1.5 L/min

Chillat Gas Flow: 12 10 - 20 L/min

Sample Introduction Settings:

Exposure Type: Steady State Transient

Apply Gas Flush time: First Sub-Exposure Only Each Sub-Exposure

Laser Triggering: Each Sub Exposure

Number of Laser Patterns needed for each sample: 3

Minimum duration of each Pattern (not including gas flush): 15 Seconds

- In order to integrate the correct part of the peak it is possible to set gate times for each wavelength. Select the Elements option in the Method and click on the "General" tab. Select Gate 1 (start) and Gate 2 (stop) times for each wavelength.

Element Parameters for Fe 259.940 {130}

General Standards Fit IECs Subarray

Element Name: Fe2599

Timing:

Gate 1: 0

Gate 2: 0

Intensity Factor: 1

Analytical Report:

Print Units: ppm

Significant Digits: 4

Conc. Factor: 1

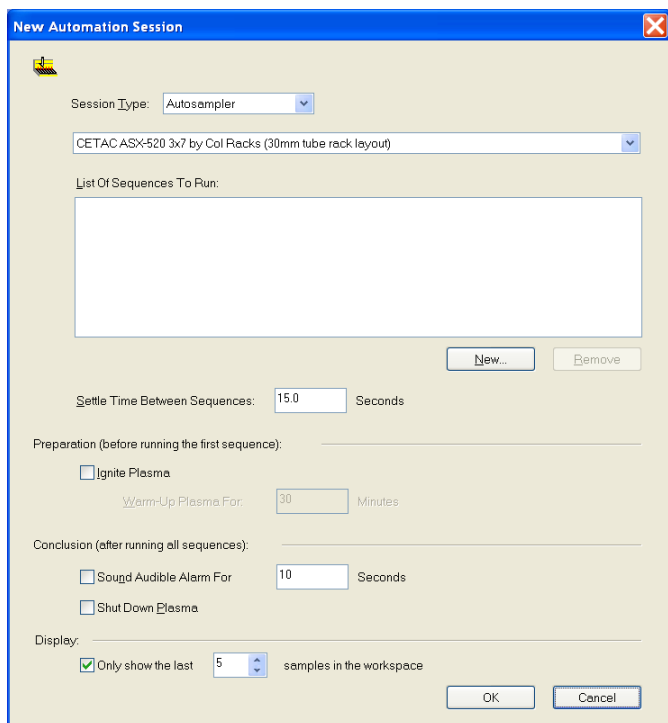
Display in Analytical Report

Note – Gate times are best selected by using a Time Scan to carry out the method development, see section below.

Automated Analysis Using Steady State or Transient Measurements

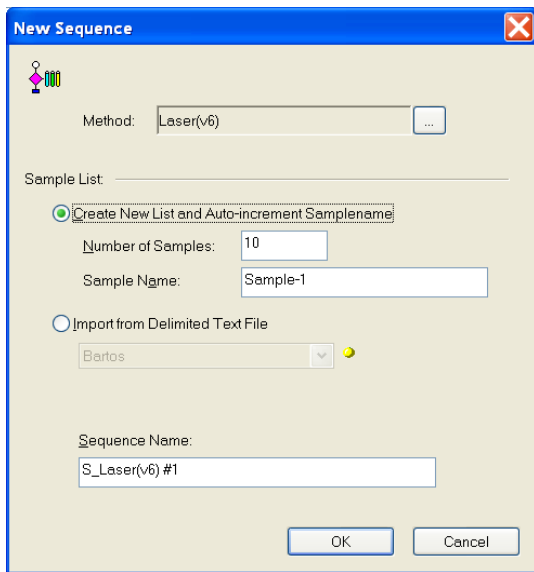
A sequence of standards and samples can be measured automatically by creating a simulated Autosampler Autosession.

1. Click on the “Sequence” tab in Analyst and select ‘Auto Session’ and ‘New Autosampler’. It doesn’t matter which autosampler layout is selected as the autosampler will not actually be used for the analysis.

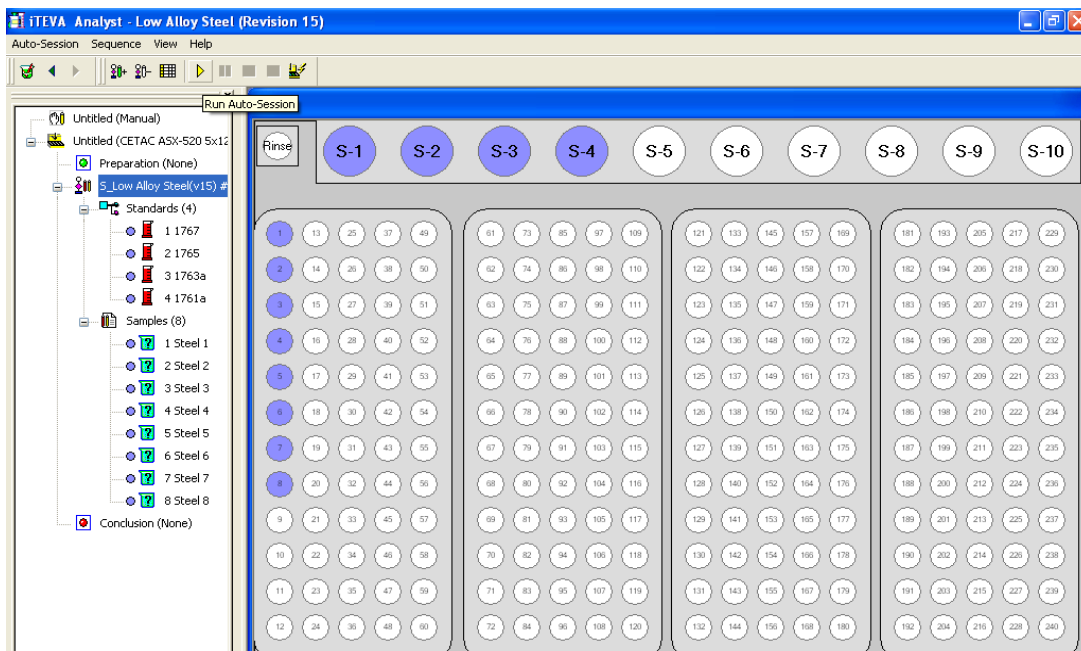


The screenshot shows the 'New Automation Session' dialog box. The 'Session Type' is set to 'Autosampler'. The selected autosampler is 'CETAC ASX-520 3x7 by Col Racks (30mm tube rack layout)'. The 'List Of Sequences To Run' is currently empty, with 'New...' and 'Remove' buttons below it. The 'Settle Time Between Sequences' is set to 15.0 Seconds. Under 'Preparation (before running the first sequence)', the 'Ignite Plasma' checkbox is unchecked, and 'Warm-Up Plasma For' is set to 30 Minutes. Under 'Conclusion (after running all sequences)', the 'Sound Audible Alarm For' checkbox is unchecked and set to 10 Seconds, and the 'Shut Down Plasma' checkbox is also unchecked. The 'Display' section has the 'Only show the last' checkbox checked and set to 5 samples in the workspace. 'OK' and 'Cancel' buttons are at the bottom.

2. Now click on the “New” button and select the method name and number of samples to be measured, you may also name the sequence if you wish.



3. Right click on the title of the sequence and select “Autolocate” to create the autosampler layout.

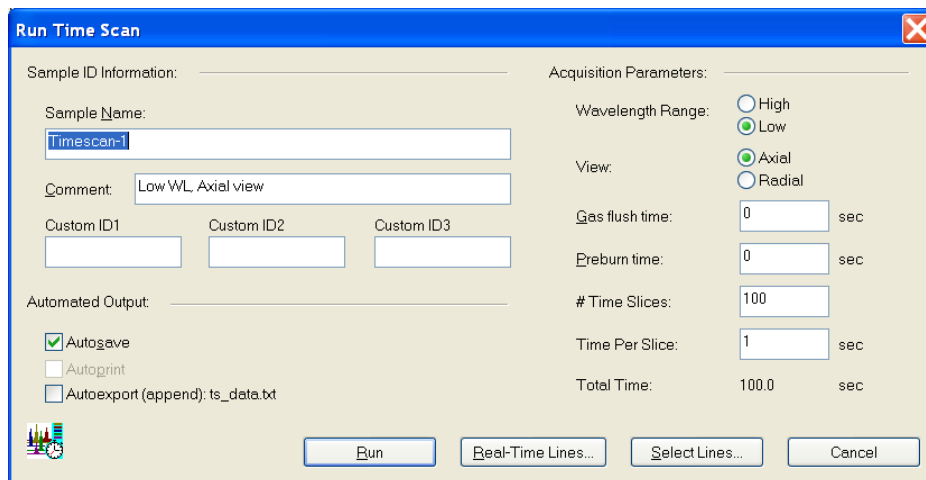


4. To start the analysis sequence click on the Start button on the toolbar. The analysis will start and iTEVA will display a message “Waiting for trigger”. When the laser is started it will provide the trigger signal and the analysis will start.

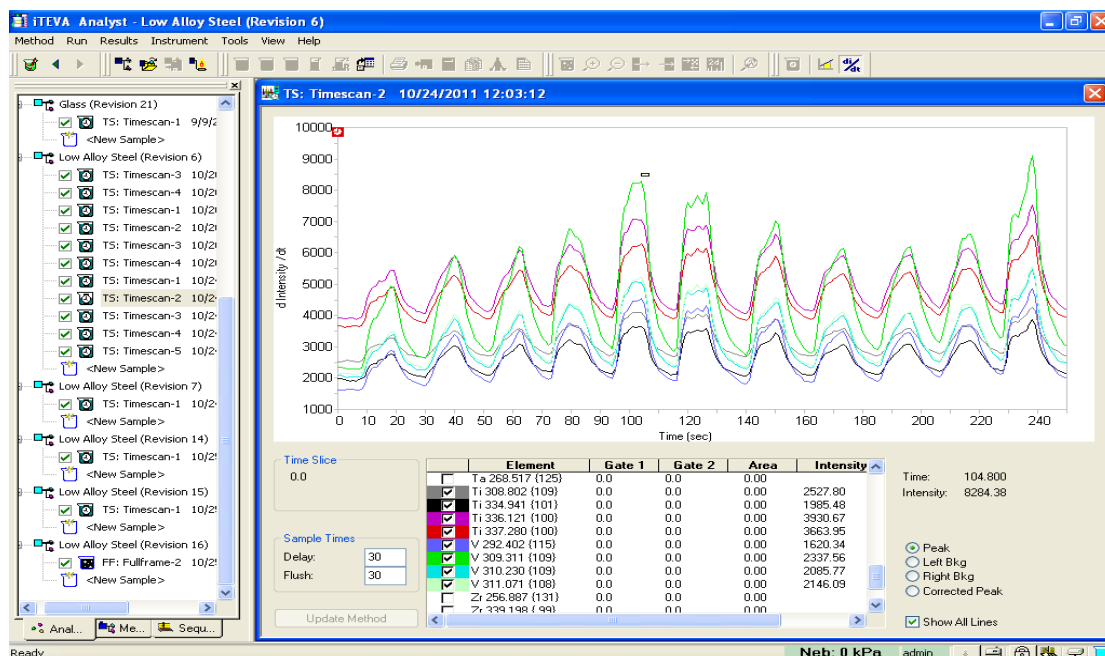
Time Scan Analysis

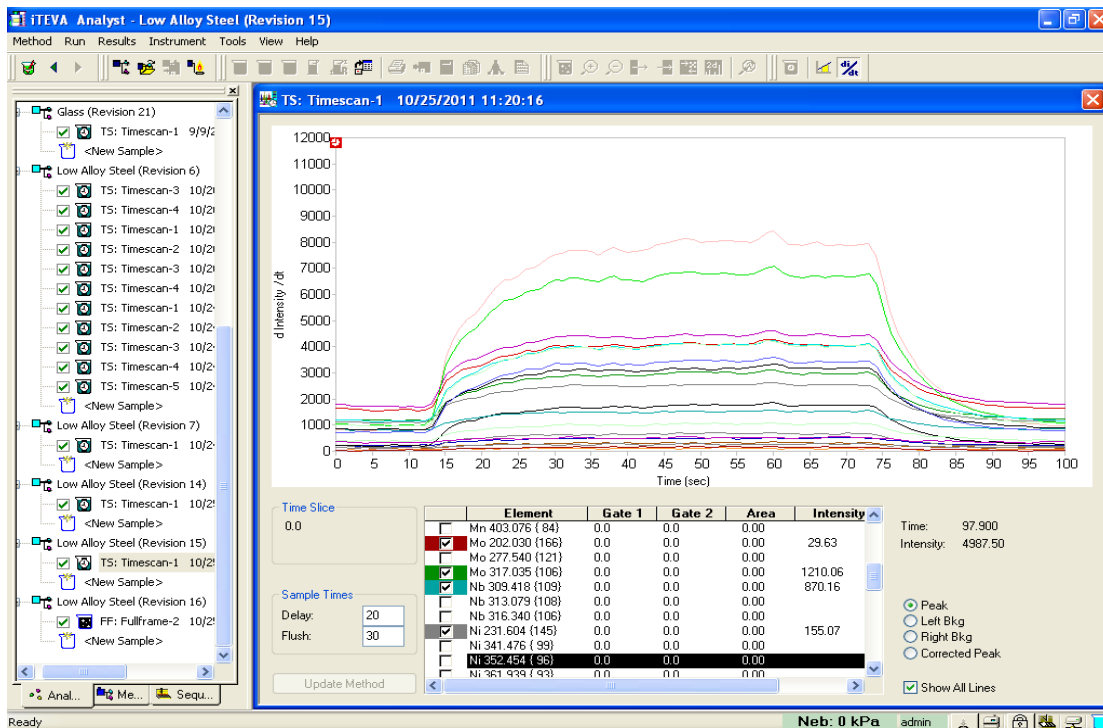
A time scan can be used to assist in determining appropriate gas flush times and sample stability as well as to optimize laser running parameters.

Click on the Run menu in Analyst and select “Time Scan”, select the appropriate sub exposure (axial, radial, low or high wavelength) and enter values for the number of time slices and time per slice to give the required resolution. Then click the Run button to start the measurement. The total time of the measurement including flush time must be 3 seconds less that the total time for the ablation. See note on triggering above.



In the figure below, spots were ablated quickly with varying laser parameters to find the optimized laser parameters. In the second figure below, patterns were ablated to optimize RSD, gas flush time, and washout time.





Automated Analysis of Multiple Time Scans

Multiple time scans cannot be run using the simulated autosampler sequence approach described above. For this kind of application it is better to use the “Start Continuous Run” approach.

1. Click on the “Run” menu in Analyst and select the “Start Continuous Run” option. Select “Timescan” as the Sample Type, untick the “Run Forever” option and select the number of samples to be measured.

Make sure to tick the “Single Sample” option, this will then only display the last timescan that was run although all timescans will be saved in the database and can be recalled at a later date. If large numbers of timescans accumulate in the workspace Windows can begin to slow down which may cause communication issues.

Continuous Run Options

Sample Type: Time Scan

Run Forever

Number of Samples: 10

Pause time between samples: 1 seconds

Single sample run mode
(Samples will not accumulate in the workspace)

Start... Cancel

2. Click on the “Start” button to begin the analysis; the timescan window will then be displayed. Select the appropriate sub exposure (axial, radial, low or high wavelength) and enter values for the number of time slices and time per slice to give the required resolution. These parameters will be used for each individual timescan. Then click the Run button to start the measurement sequence.

Run Time Scan

Sample ID Information:

Sample Name: Timescan-1

Comment: Low WL, Axial view

Custom ID1 Custom ID2 Custom ID3

Automated Output:

Autogave

Autoprint

Autoexport (append): ts_data.txt

Acquisition Parameters:

Wavelength Range: High Low

View: Axial Radial

Gas flush time: 0 sec

Preburn time: 0 sec

Time Slices: 100

Time Per Slice: 1 sec

Total Time: 100.0 sec

Run Real-Time Lines... Select Lines... Cancel

The total time for each timescan measurement including flush time must be 3 seconds less than the total time for the each ablation. See note on triggering above.