

LSX-213 G2 Compatibility: Thermo Fisher Scientific XSeries 2

Introduction

The LSX-213 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 XSeries 2 benchtop quadrupole ICP-MS offers high-sensitivity analysis with low background levels, ideally suited to running trace analysis using the laser ablation system.

Interfacing

Three connections between the instrumentation are required for operation of the LSX-213 G2 with the Thermo Fisher XSeries 2:

1. Sample out from the laser to the XSeries 2 torch
2. Nebulizer out from the XSeries to the laser Carrier Gas inlet
3. Remote Trigger between the laser and the ICP host PC (requires Advantech PCI Remote Trigger Card, provided by Thermo Fisher Scientific)

Additionally, an extra helium tank is required to sweep the laser sample from the ablation cell to the ICP.



(Remote Trigger not shown)

For interfacing the torch, there are two options (see figure below). For old style glass torches, a ground glass S12/5 socket is provided in the completion kit (left); for the newer standard torch, a polymer fitting is provided (right). The ground glass coupling is secured using a clamp (also in the completion kit), whereas the polymer fitting is press-fit into the torch injector.



Enabling Remote Triggering

To enable remote triggering in PlasmaLab, the External Trigger accessory must be installed and configured in the PlasmaLab software.

NB: for Windows® 7 installations, PlasmaLab v2.6.2 must be used to ensure the relevant accessory support files are correctly installed. For older versions of Windows®, any version of PlasmaLab is appropriate.

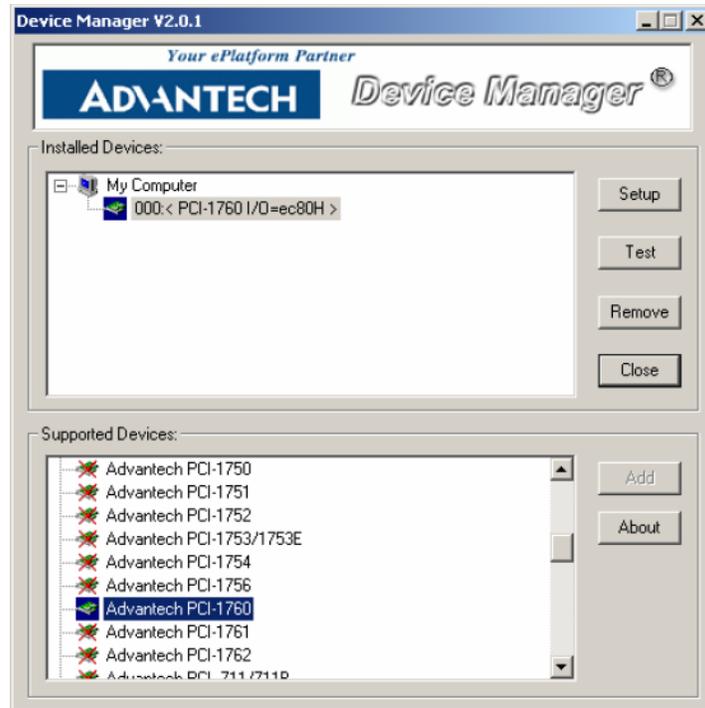
To install the external trigger card:

1. Insert the Thermo Electron External Triggers CD supplied with the trigger card into the CD drive and wait for the program to start.
2. From the 'Welcome' screen, scroll down to the bottom and choose the link to 'Install the Advantech Device Manager'.
3. Once the files have unpacked, follow the wizard to install the device manager, choosing the 'Typical' installation when prompted.
4. After the files have installed, the 'Welcome' screen will display. Scroll down to the bottom and choose the link to 'Install the trigger card device driver'.
5. Follow the wizard, again choosing 'typical' when prompted.
6. Once installed, from the 'Welcome' screen scroll to the bottom and choose to 'Install the accessory support file'. This will install the necessary support files for use with PlasmaLab.
7. Shutdown the PC and install the card in an empty PCI slot.
8. Restart the PC. After logging in, Windows® will automatically detect the device and register the required drivers.



Testing the card

Access the Advantech Device Manager from the Start Menu (under Programs or All Programs, depending on the Windows® version). The following window (or similar) should appear, with the Advantech 1760 card already selected:



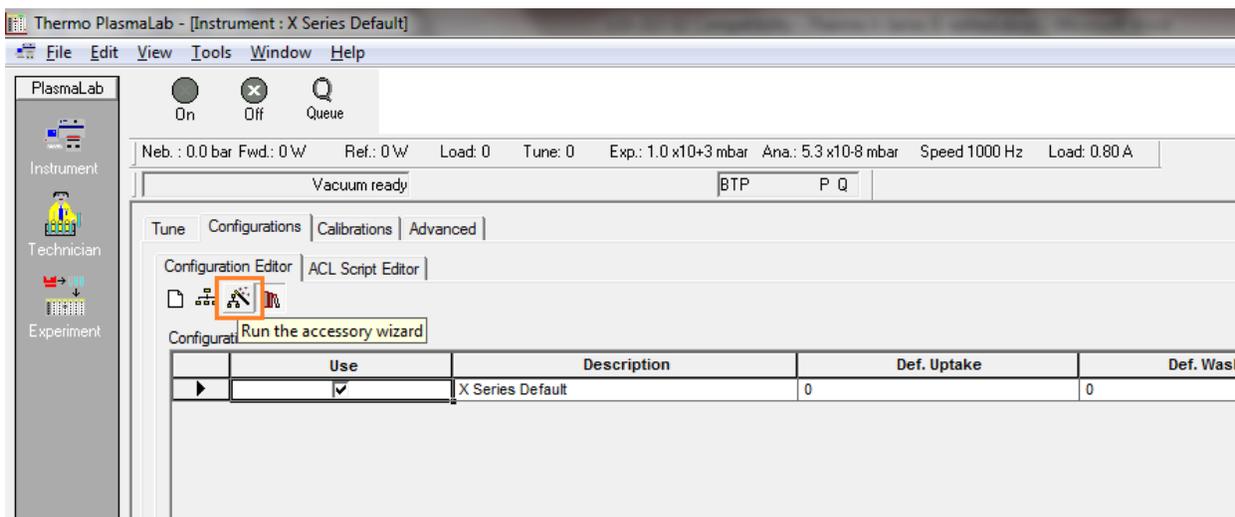
Click on the 'test' button to test the card. Select the 'Digital Input' tab:



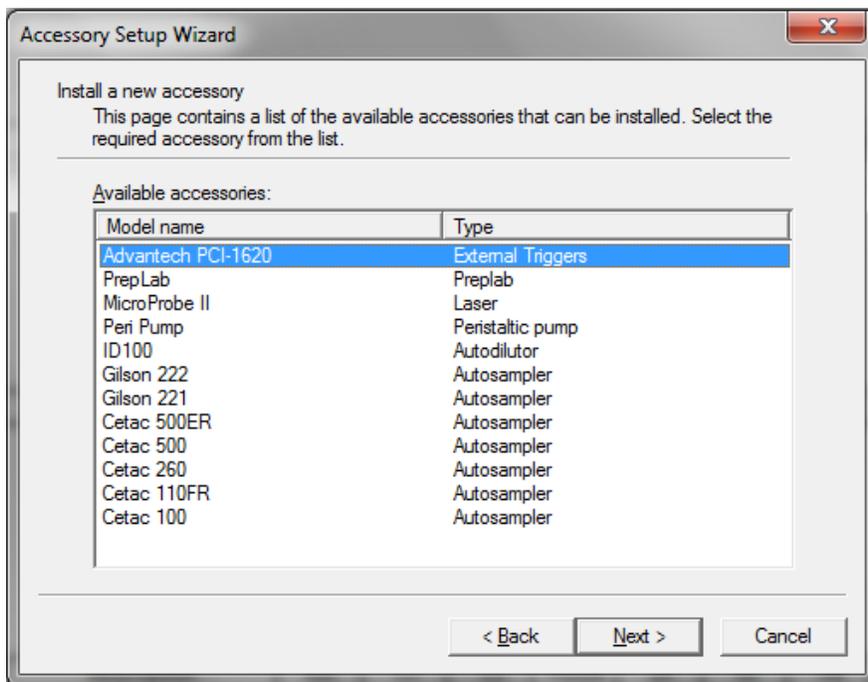
On the laser system, use a Single Point pattern at 5% energy with a Burst Count of 1. Draw a few spots on the sample (e.g. 10), and open the sequence editor. Set the trigger delay to zero seconds and run the sequence. The indicator under '0' in the Advantech Device Test window should turn red whenever the trigger is sent from the laser. NB: This corresponds to Input ID 1 in PlasmaLab.

Installing the External Triggers Accessory

1. In PlasmaLab, go to the 'Configurations' tab under 'Instrument' and run the accessory wizard:

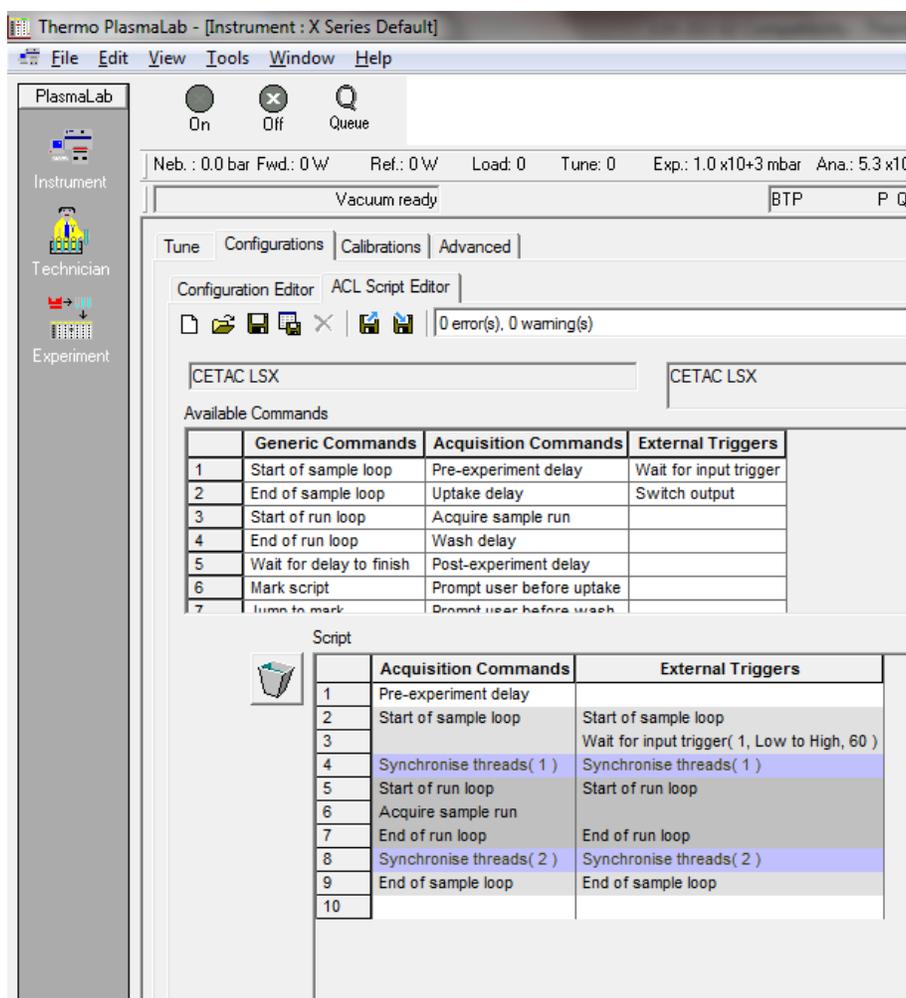


The external trigger card will be listed as the Advantech PCI-1620:

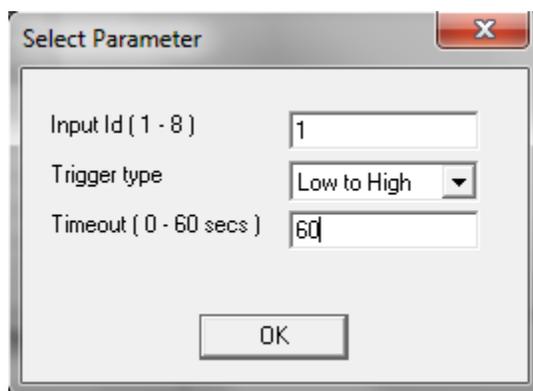


NB: if this is not listed, the triggers.acc file will have to be transferred to the 'bin' folder in the PlasmaLab installation directory. This can be obtained from CETAC on request.

2. Follow the wizard to completion. The Advantech PCI-1620 will now be listed under 'Available Accessories and Devices'.
3. Create a new configuration and name it 'Laser'; then, check the box next to the trigger card.
4. Select the 'ACL Script Editor' tab. Edit the script to match the following:



To use the script editor, commands are dragged from the 'Available Commands' section into the 'Script' section. Unwanted commands are deleted by dragging the item into the trash can on the left. For the 'Wait for Input Trigger' command, an additional window will pop up. Enter the values as shown:



5. Save the script as 'CETAC LSX'.
6. Back in the 'Configurations' tab, select the 'CETAC LSX' script for the 'Laser' Configuration:

Tune | Configurations | Calibrations | Advanced |

Configuration Editor | ACL Script Editor |

Configurations:

| | Use | Description | Def. Uptake | Def. Wash | ACL Script |
|---|-------------------------------------|------------------|-------------|-----------|----------------|
| | <input type="checkbox"/> | X Series Default | 0 | 0 | No accessories |
| ▶ | <input checked="" type="checkbox"/> | Laser | 0 | 0 | BETAC LSX |

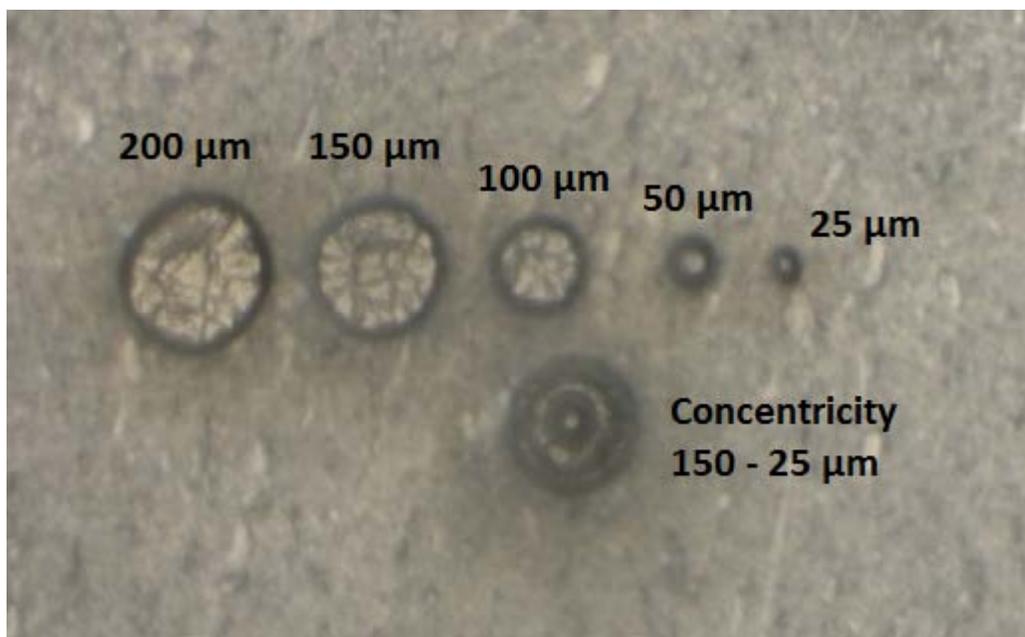
Available Accessories and Devices:

| | Use | Model | Device Type | Description |
|---|-------------------------------------|--------------------|-------------|--------------------------|
| ▶ | <input checked="" type="checkbox"/> | Advantech PCI-1620 | Trigger | Using un-filtered inputs |

Representative Data

Performance testing was carried out using NIST CRM-612 glass, which contains between 30 and 40 ppm for trace elements such as La, Th and U. To set the initial power conditions for the laser, the X-Series 2 was optimized using 55% laser power with a helium sweep flow of 0.6 l min^{-1} . Using these approximate tune conditions, the optimum power level can be found for the laser interaction with the sample.

Craters produced in NIST were well defined and concentric, indicating good coupling of the laser energy with the sample surface and good optical alignment.



Power tuning was performed by ablating ten 60 sec lines ranging in power from 5 % energy to 50 % energy. The stabilized signal was averaged and the RSD calculated. Plotting intensity and RSD against laser power yields a positive correlation comprising two separate gradients, with a defined 'break-point' in the correlation. This break-point indicates an increased probability of laser induced fractionation, so the optimum laser power is below the break-point. Additionally, the optimum conditions will have the lowest RSD achievable since this indicates that a consistent aerosol is generated at the sample surface. From Figure 1 below, the optimum laser power for NIST-612 using this particular laser system is 20 % energy.

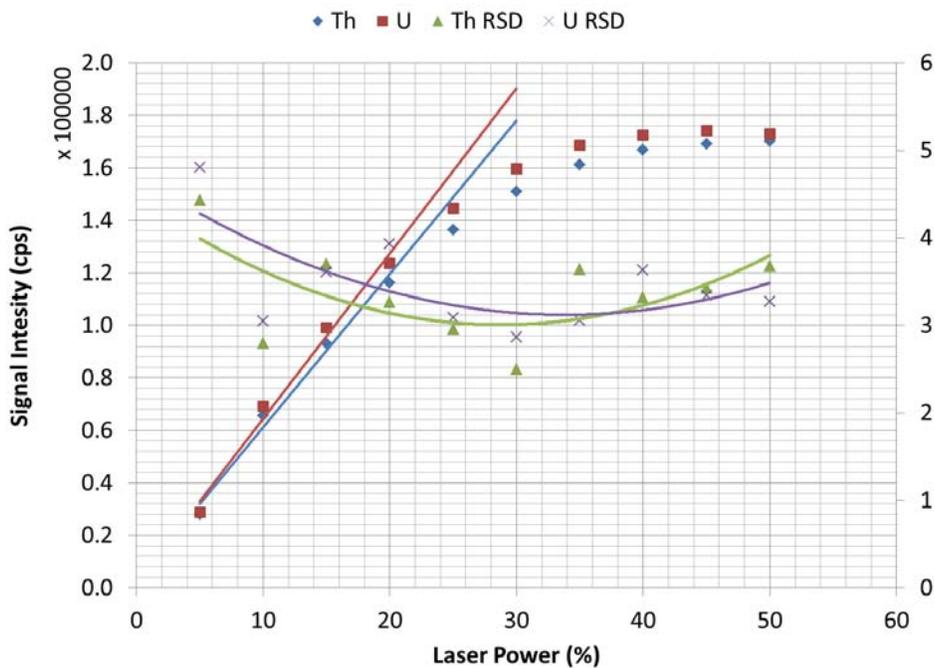


Figure 1. Power tune for NIST 612 using a 100 μm spot tracked across the sample surface for 1 min at $10 \mu\text{m s}^{-1}$

Once the optimum laser power was established, the XSeries 2 was fine tuned. Optimum conditions for both the laser and the XSeries 2 are summarized in the table below:

Table 1. Optimum instrumental conditions for the LSX-213 G2 and the XSeries 2.

| Thermo Fisher Scientific XSeries 2 | | | | |
|---|--------------|---------------------|----------------|--------------------------|
| | <i>Major</i> | | <i>Minor</i> | |
| Extraction | -216 | V | Lens 3 | -195.3 V |
| Lens 1 | - | V | Forward Power | 1400 W |
| | 1110 | | | |
| Lens 2 | -71.4 | V | Horizontal | 61 |
| Focus | 8.4 | V | Vertical | 521 |
| D1 | -49.4 | V | DA | -24.3 |
| D2 | -140 | V | Cool | 13.0 l min ⁻¹ |
| Pole Bias | 0 | V | Auxiliary | 0.90 l min ⁻¹ |
| Hex Bias | -30 | V | Sampling Depth | 100 |
| Nebulizer (Carrier) Gas | 0.58 | l min ⁻¹ | Interface type | Xt |
| LSX-213 G2 | | | | |
| Laser Power | 20 | % | | |
| Frequency | 20 | Hz | | |
| Spot size | 100 | µm | | |
| Scan rate | 10 | µm s ⁻¹ | | |
| He sweep gas | 0.6 | l min ⁻¹ | | |
| Gas Blank | 10 | s | | |
| Shutter Delay | 10 | s | | |

Sensitivity and Stability

Short (1 min), medium (5 min) and long term (10 min) stability was tested by ablating an appropriate line and continuously acquiring data. The long term stability trace is shown in Figure 2 below.

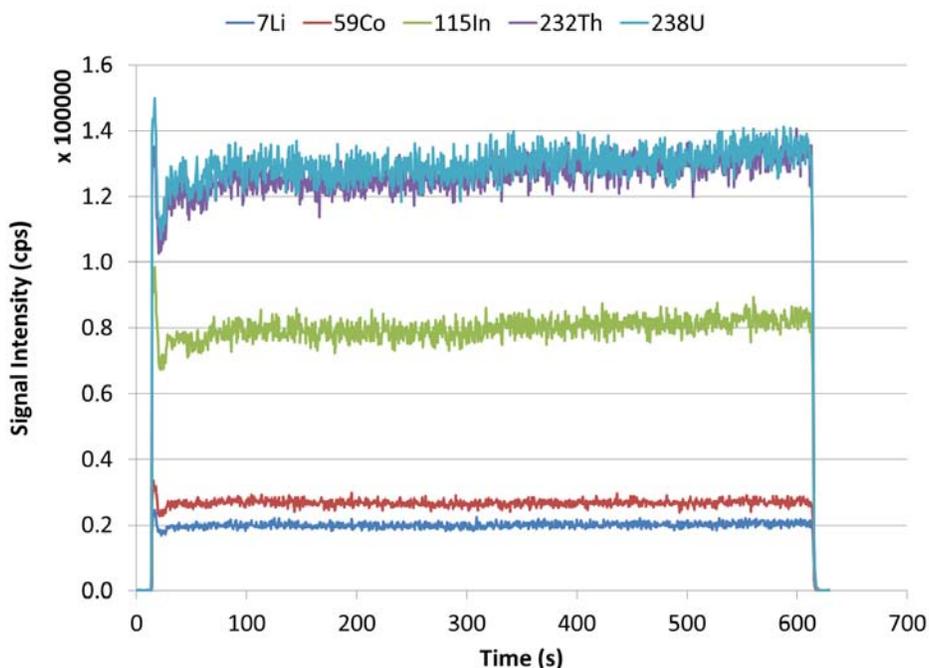


Figure 2. Stability plot for a 100 μm spot tracked across the sample surface at 10 $\mu\text{m s}^{-1}$

The initial spike seen is due to the initial laser interaction with the sample surface. The initial interaction is a full 100 μm spot in focus on the surface, whereas as the laser line tracks at 10 $\mu\text{m s}^{-1}$ only approximately 50% of the sample surface is fully in focus. Additionally, washout of the cell results in peak broadening, which results in the slight signal rise after the initial drop off.

Stable signals were achieved after 15 seconds of ablation (25 s including the gas blank), so stability was calculated from 25 seconds to the end of the ablation line. Stability and background corrected average signal levels are shown in Table 2 below. The stable region shows very little drift throughout the scan.

Table 2. Signal levels and RSD's for stability lines

| | 7Li | 59Co | 115In | 232Th | 238U |
|----------------------|-------|-------|-------|--------|--------|
| 1 min Counts | 20848 | 29489 | 81531 | 124657 | 131115 |
| RSD | 3.72% | 3.46% | 3.36% | 3.66% | 3.73% |
| 5 min Counts | 20587 | 28334 | 82394 | 128292 | 133256 |
| RSD | 3.92% | 3.35% | 4.10% | 3.88% | 4.12% |
| 10 min Counts | 19853 | 26765 | 79865 | 126820 | 129845 |
| RSD | 3.74% | 3.38% | 3.62% | 3.68% | 3.37% |

The table shows consistent stability achieved over the whole mass range over short, medium and long term runs. Signal levels are also shown to be consistent over the three time ranges, indicating stable signals can be achieved over longer runs.

Sensitivity (averaged over the three stability runs) of the scanned elements is shown in Table 3 below. The trend in sensitivity follows the mass bias of the XSeries 2.

Table 3. Sensitivity over the mass range

| | 7Li | 59Co | 115In | 232Th | 238U |
|------------------------------|------------|-------------|--------------|--------------|-------------|
| Counts (cps) | 20429.90 | 28196.28 | 81263.71 | 126590.05 | 131405.73 |
| Concentration (ppm) | 40.2 | 35.5 | 38.9 | 37.79 | 37.38 |
| Sensitivity (cps/ppm) | 508.21 | 794.26 | 2089.04 | 3349.83 | 3515.40 |

Background and Limits of Detection

Background levels were taken over two days by measuring ten gas blanks for 60 seconds on each day. Blank Equivalent Concentrations (BECs) were calculated from the average of the two blanks, referenced to the sensitivity values in Table 3.

The standard deviations were pooled using Equation (1) below. Limits of Detection (LOD's) were then calculated using Equation (2).

$$\text{SD} = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}} \tag{1}$$

$$\text{LOD} = 3 \times \text{BEC} \tag{2}$$

Results are summarized in Table 4 below. Blank levels are relatively low, yielding sub-ppm BECs and LODs, sub-ppb levels for the higher mass elements due to the mass bias of the XSeries 2.

Table 4. Blank values, BECs and LODs across the mass range

| | 7Li | 59Co | 115In | 232Th | 238U |
|----------------------------|------------|-------------|--------------|--------------|-------------|
| Day 1 Average Blank | 143.30 | 6.12 | 2.83 | 0.42 | 0.48 |
| Standard Deviation | 10.66 | 0.94 | 0.30 | 0.18 | 0.21 |
| Day 2 Average Blank | 162.49 | 8.35 | 4.35 | 0.51 | 0.43 |
| Standard Deviation | 28.00 | 2.74 | 1.62 | 0.16 | 0.21 |
| BEC (ppb) | 300.85 | 9.11 | 1.72 | 0.14 | 0.13 |
| LOD (ppb) | 425.90 | 16.85 | 3.39 | 0.29 | 0.31 |

Washout Characteristics

An important characteristic of laser ablation ICP-MS is washout of the analyte from the system. This is the time taken for the aerosol to be swept through the system, which affects both sensitivity and sample throughput.

Three washout figures were calculated as shown in Figure 3 below, each one appropriate for different applications. 90 % washout is mostly appropriate for high throughput bulk analysis, where a small amount of sample blurring can be allowed. 99 % washout is appropriate for the majority of laser sampling applications. Washout to baseline is appropriate for mapping applications, as each pixel will need to be washed out of the cell to prevent blurring in the final processed image.

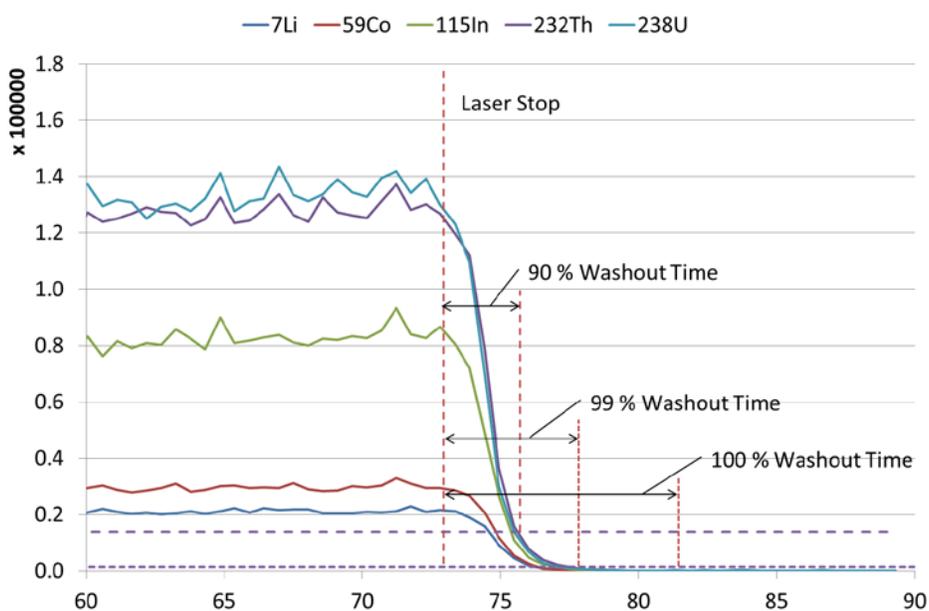


Figure 3. Washout of the LSX-213 G2 standard ablation chamber coupled to the XSeries 2 ICP-MS

Short washout times are desirable to ensure applicability to all three major method types. The washout of the standard ablation cell, averaged for all analytes using the three stability scans, are summarized in Table 5 below. This shows that the standard setup has excellent washout characteristics appropriate for most laser ablation studies.

Table 5. Washout times for the LSX-213 G2 standard ablation chamber coupled to the XSeries 2

| Washout Level | Time |
|---------------|--------------|
| 90 % | 3.90 ± 0.47 |
| 99 % | 5.67 ± 0.26 |
| Baseline | 14.97 ± 3.01 |

Conclusions

The LSX-213 G2 couples easily to the X-Series 2, both physically and electronically. Good sensitivity, stability and detection limits were obtained. This is due in part to the good speed of washout obtained with the standard cell design.

The LSX-213 G2 is an uncomplicated but fully-featured laser ablation system that offers excellent value.