

Latest Advances in Tandem LA – LIBS Instrumentation: Leading the Innovation in Laser Ablation Chemical Analysis with Simultaneous LIBS and LA-ICP-MS using Femtosecond system

Chunyi Liu
Applied Spectra, Inc. Fremont, CA

Introduction:

The J200 Tandem LA – LIBS Instrument from Applied Spectra is a true innovation in laser ablation solid sampling that provides simultaneous LA-ICP-MS and LIBS analysis to expand elemental coverage and increase concentration dynamic range.

The new J200 Femtosecond IX tandem LA – LIBS instrument from Applied Spectra, Inc. provides the unique capability of combining the analytical benefits of both LIBS and LA-ICP-MS. Specifically, LIBS can be used for the analysis of H – Pu, which includes non-metals such as C, H, N, O, and halogens (e.g. F) that are difficult (or impossible) to be analyzed in conventional ICP-MS systems. Additionally, LA-ICP-MS complements the LIBS analysis by providing trace elemental and isotopic ratio compositions. This tandem instrument, in combination with a Analytik Jena PlasmaQuant® MS Elite ICP-MS, expands the dynamic range of analysis from sub-ppb levels with LA, to % levels with LIBS, while also increasing the elemental coverage (typical ICP-MS elements + H, Ar, He, O, and F detection with LIBS).

In this presentation, latest advance in LIBS detector, ultrashort laser source, and LA and LIBS sampling cell are presented for advanced analytical capability. These hardware advances combined with integrated data analysis software enables ground-breaking analytical capability to measure every element in the periodic table in one laser sampling including organic, halogens and others that are difficult for ICP-MS alone.

Most Compact Femtosecond system With superior capability

Figure 1. ASI J200 Femto IX Tandem System 343/1030 nm fs laser with dual ICCD detector

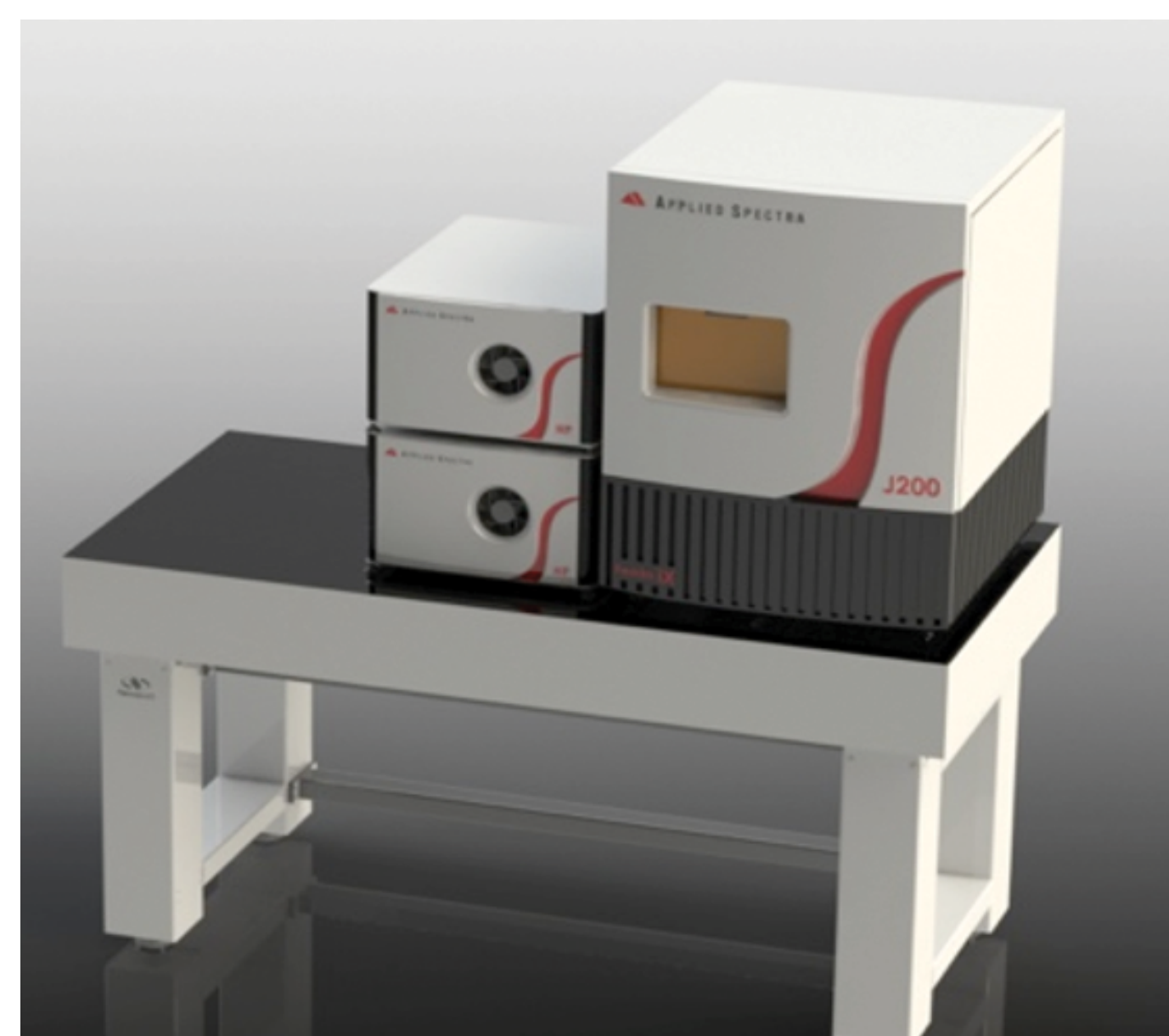


Figure 2. Long term laser energy stability.

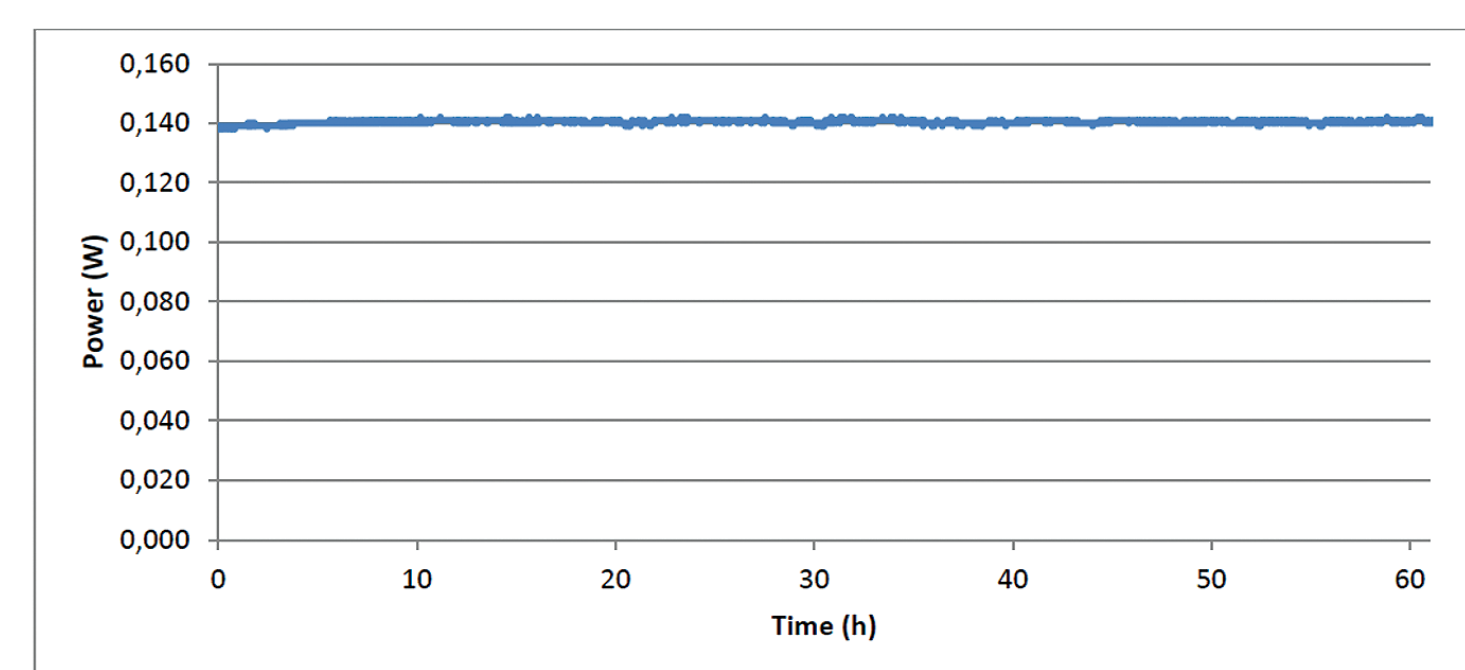


Figure 3. Example of a LIBS spectrum from NIST 610 Glass using J200 Femto IX system showing K, Rb and O.

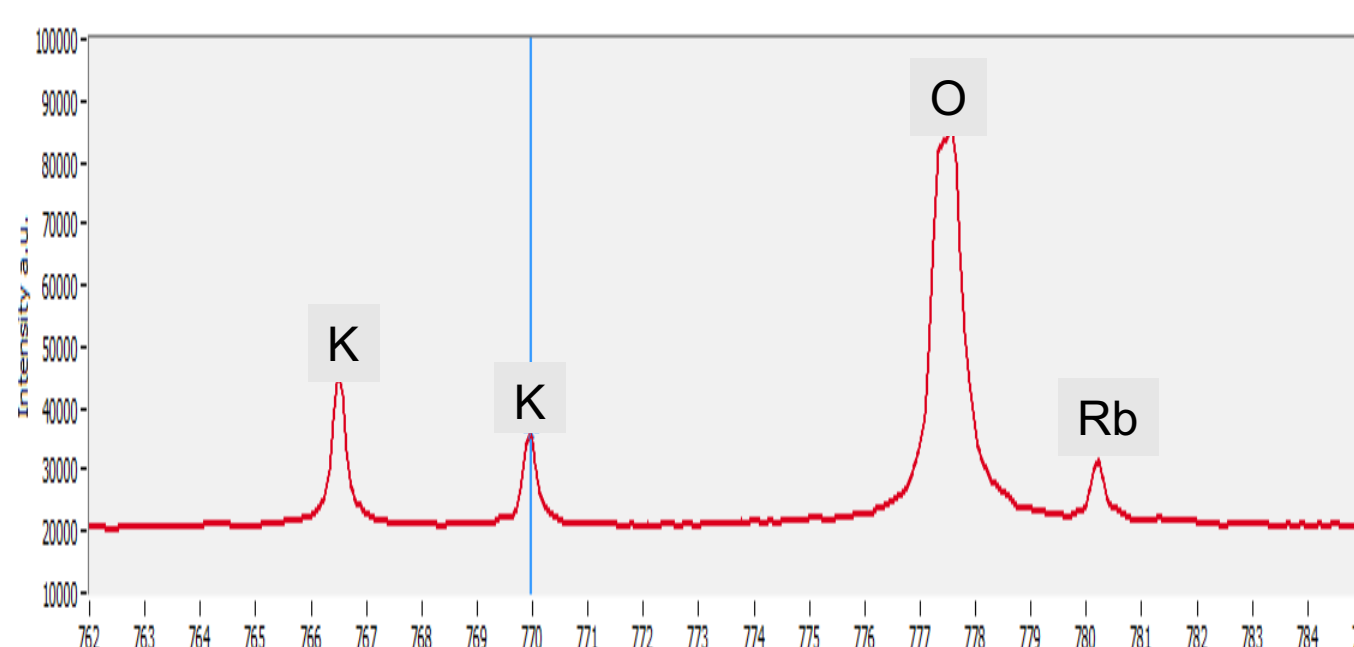
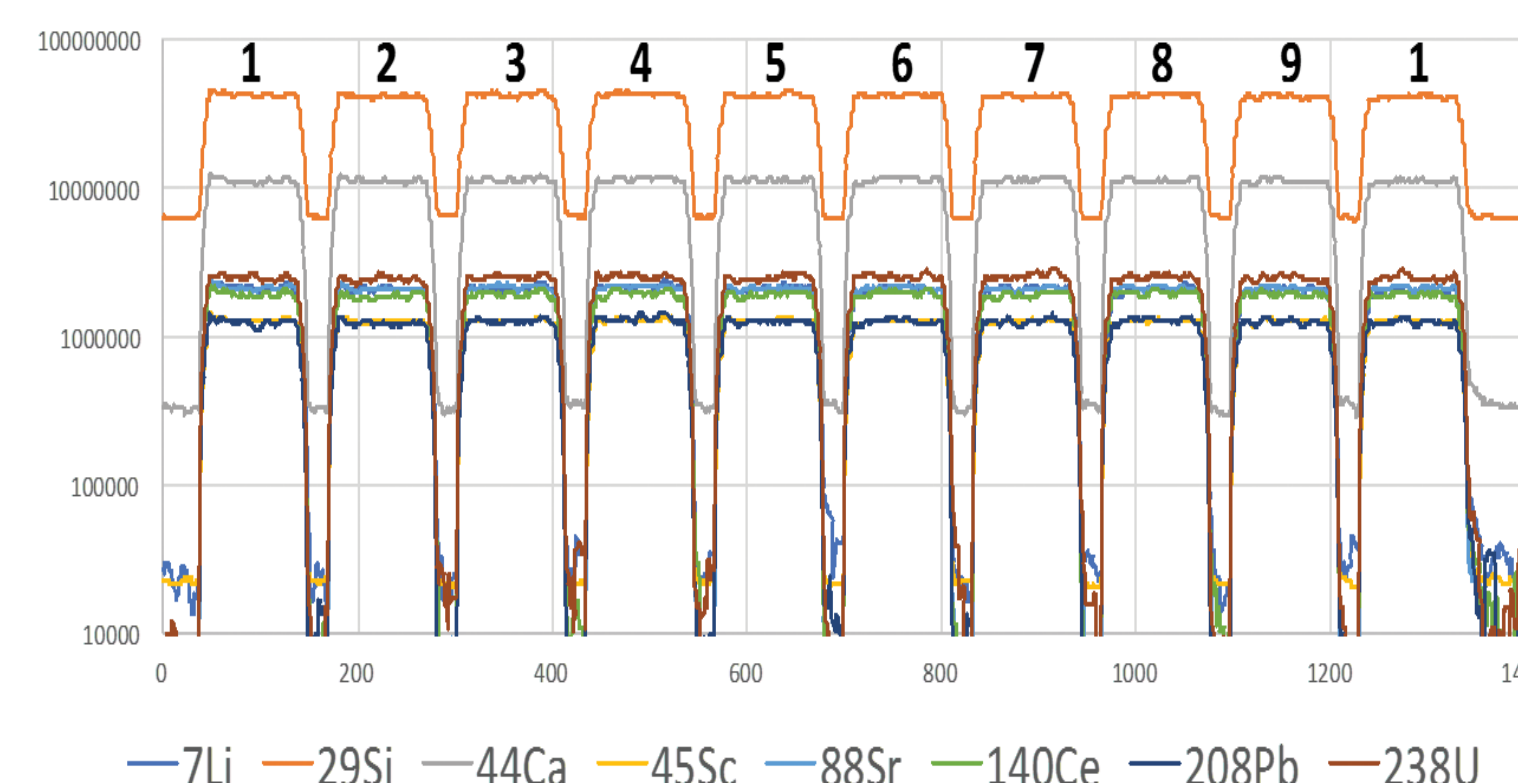


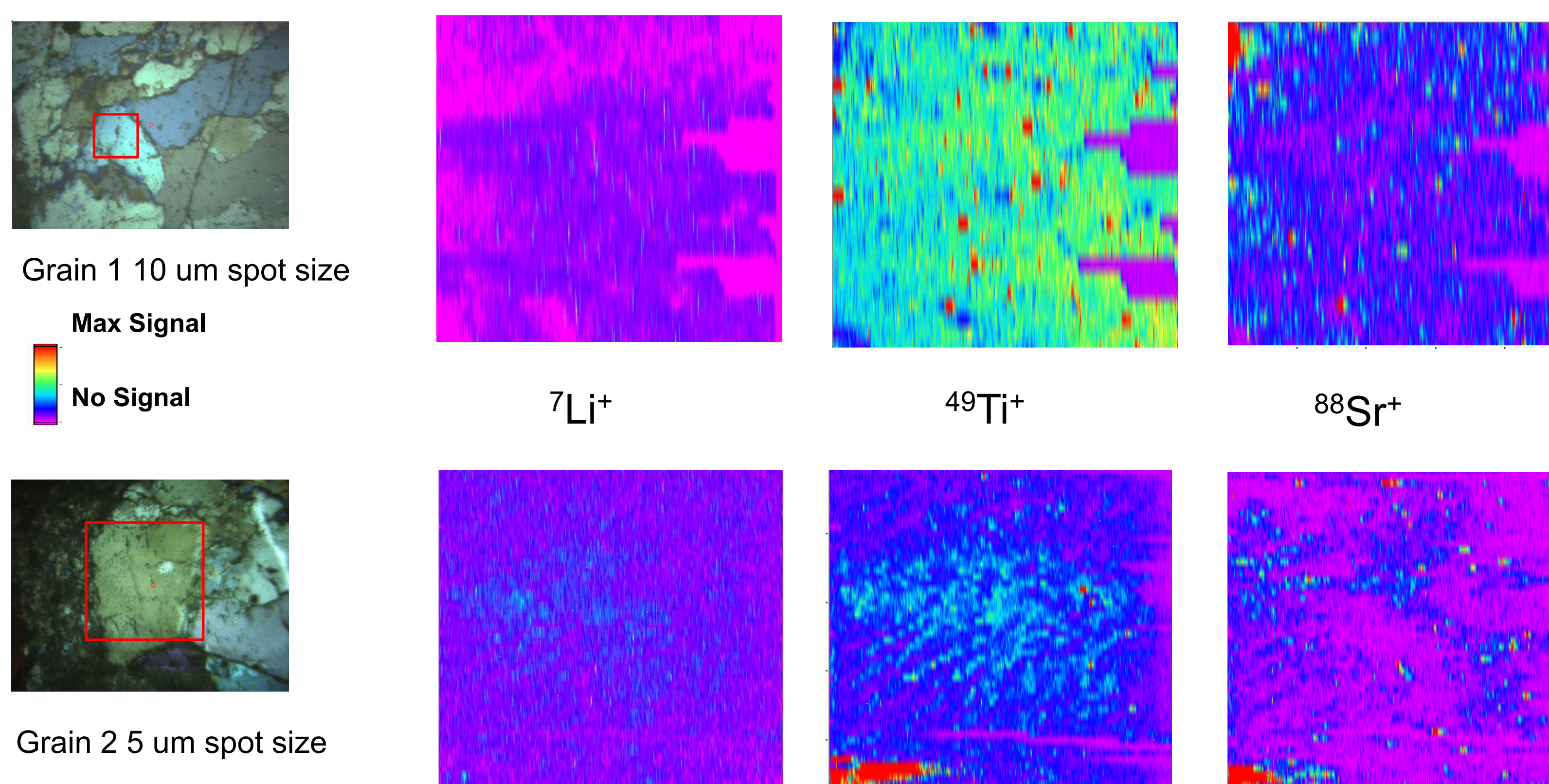
Figure 4. Excellent reproducibility of signals obtained for light to heavy isotopes based on line scans over nine different areas in Vertex™ sample chamber.



* Acquired using NIST 610 glass
** RSD across different scan less than 2%

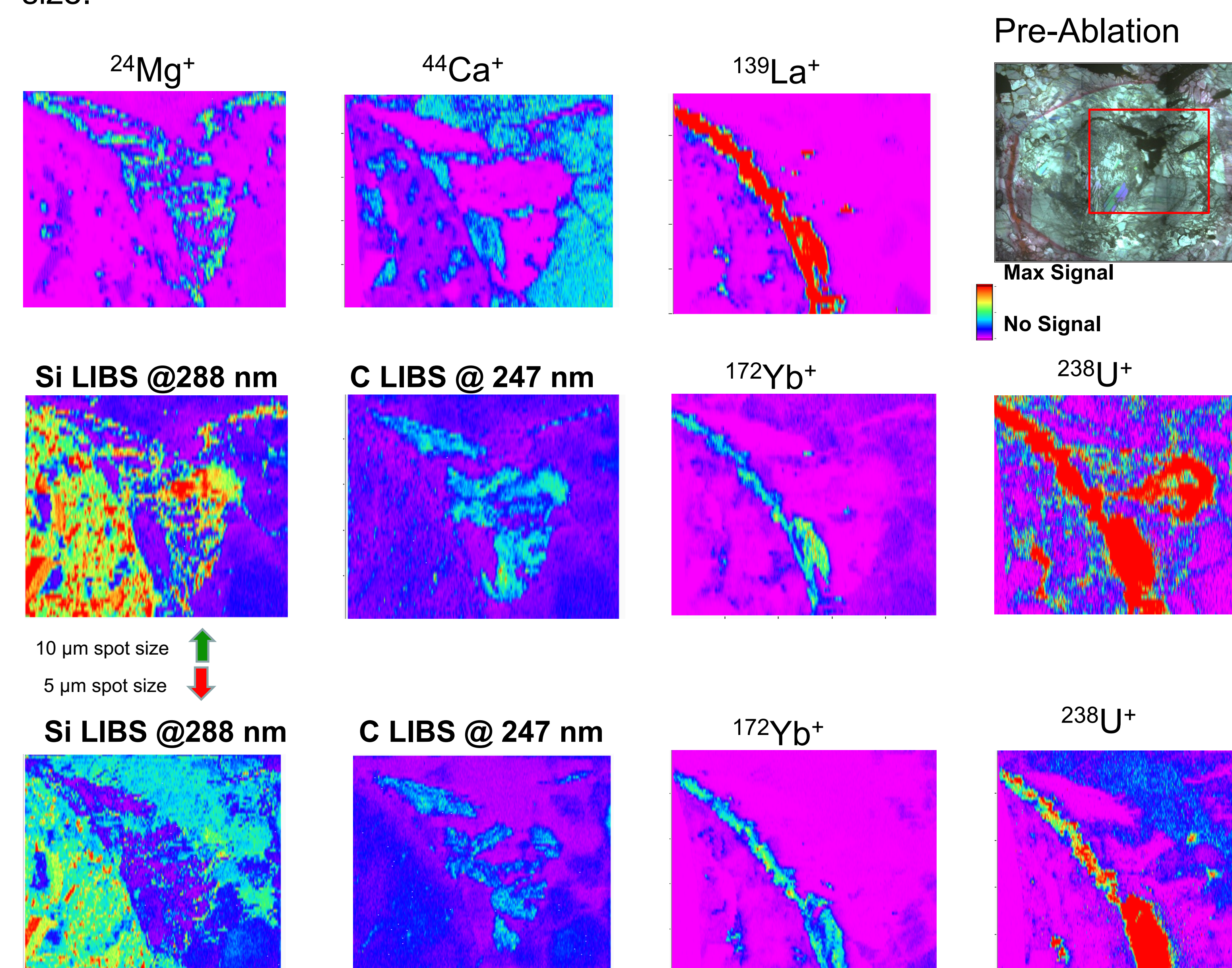
High Resolution Single Grain Quartz Mapping :

Figure 5. Chemical contour mapping inside two individual quartz grain sample is acquired using 343 nm fs laser and ICP-MS, condition used as 10 µm laser spot size in grain 1 and 5 µm in grain 2. 20 Hz rep rate, 0.008 mm/sec speed on a 0.43x0.43 square with 0.018 mm spacing.



Mapping with Tandem System:

Figure 6. Chemical contour mapping of Graphite sample using 343 nm fs laser, data collection using both HP ICCD and ICP-MS, 10 µm laser spot size, 10 Hz, 0.01 mm/sec speed. 55 line on a 1x0.8 mm region. Maps display the different elemental response. After the first scan, same region was scanned again using 5 µm laser spot size.



Figures of Merit:

Table 1. Femto IX Tandem LA-ICP-MS and LIBS

LIBS Data was acquired using NIST SRM 610-614, ASI J200 Femto IX instrument, 10 laser pulses, 35 µm laser spot size. LA-ICP-MS data was calculated by measuring ablated mass vs. MS Intensity using NIST 612.

fs-LIBS		fs-LA-ICP-MS			
Element	LOD (ppm)	NIST 612			
		Reference Values* (mg/kg)	Isotope	Absolute Mass (pg)	LOD (ppb)
Si	15	40.2	⁷ Li	80.6	3.24
Al	4.7	44	⁴⁷ Ti	961	42.3
Be	0.56	35.5	⁵⁹ Co	56.8	2.02
Ca	19	78.4	⁸⁸ Sr	6.2	0.486
Cu	4.8	34.7	¹²¹ Sb	19.4	0.673
Zn	0.55	38.4	¹⁴⁰ Ce	5.54	0.213
Li	0.28	36.7	¹⁷⁷ Hf	11.2	0.411
K	0.55	2.51	¹⁹⁵ Pt	133	0.334
Rb	0.42	4.77	¹⁹⁷ Au	36.7	0.175
		38.57	²⁰⁸ Pb	3.93	0.152
		37.79	²³² Th	3.57	0.135
		37.37	²³⁸ U	0.917	0.0343

Table 2. LIBS signal Precision from NIST glass series using Femto IX Tandem system

	610		612		614	
	Intensity	RSD (%)	Intensity	RSD (%)	Intensity	RSD (%)
K	7.70E+05	2.68	6.63E+05	0.84	6.64E+05	0.55
Rb	3.85E+05	1.74	3.43E+05	0.87	3.40E+05	0.88
Ca	1.39E+06	0.93	1.38E+06	0.83	1.37E+06	0.81
Si	1.95E+05	0.54	1.94E+05	0.44	1.93E+05	0.40
Al	1.45E+05	0.81	1.44E+05	0.79	1.44E+05	0.82
Li	1.22E+06	0.86	1.01E+06	0.41	9.46E+05	0.79

Conclusions:

- The ASI tandem J200 LA – LIBS system provides the ability to perform simultaneous LA-ICP-MS and LIBS measurements.
- New Femto IX is compact yet very powerful
- LIBS allows for fast data collection and processing (seconds – minutes).
- LIBS provides a fast detection for elements that are more difficult such as F, H, N, and O.
- Femto LIBS detection yields new capability which not available before and with great precision and detection limit
- Chemical maps provided detailed spatial information and now with high resolution, even on previous difficult to ablate samples Quartz and Graphite.
- ASI's Data Analysis software allows fast elemental mapping of both LIBS and LA-ICP-MS data.

All data was collected using the J200 Femto IX Tandem LA – LIBS Instrument with Axiom Software from Applied Spectra, Inc. in combination with Analytik Jena PlasmaQuant® MS Elite ICP-MS.

Acknowledgements:

Thank you to CAGS for providing the quartz and graphite samples.

Thank you to Analytik Jena for supplying the PlasmaQuant® MS Elite ICP-MS instrument that is being used at the ASI facility.