



Direct Analysis of Glass Samples by Laser Ablation ICP-MS

Analytical Performance of the CETAC LSX-100 Laser Sampling System

Technique overview: Laser ablation (LA) offers the capability of direct analysis of both conductive and non-conductive samples. As a technique LA-ICP-MS shows considerably more potential than other laser sampling methods separating as it does, the sample volatilization and ionization processes. In addition, the ICP-MS spectra obtained are much simpler with a "dry" plasma as compared with spectra from more conventional solutions based introduction techniques and further, the detection limits achievable are generally at ng/g(ppb) levels

System overview: The CETAC LSX-100 is a unique and flexible laser sampling system providing the capability to perform both bulk and spatial analysis at either of three selectable wavelengths. The laser is designed to operate in TEM₀₀ single mode providing a Gaussian beam profile superior to laser systems operating in the more commonly used multi-mode. The design is a compact benchtop, allowing for elegant interfacing to all commercial ICP instruments.

- Advantages:**
- w Spot sizes from <10um to 100 um for both spatial and bulk analysis.
 - w TEM₀₀ mode giving true Gaussian (95%FTG) beam profile.
 - w Capability to operate at 266 nm for optimal coupling to most materials.
 - w Software selectable laser power and repetition rate settings.
 - w Interfaceable to all ICP-MS spectrometer systems.
 - w Computer driven zoom facility with continuous magnification to x800 for optimal location of sample features.
 - w High precision x-y-z translation stages for precise and reproducible location of samples.
 - w Unique cell design for rapid signal equilibration and washout.

Analytical performance: To illustrate the general analytical performance of the system, the standard reference material SRM 612 glass from National Institute of Standards and Technology (NIST) was analyzed. Accuracy, precision and detection limits were determined as general figures of merit. Table 1 illustrates typical detection limits measured at the 3 sigma confidence interval. Data for ⁵⁶Fe is of course limited by the contribution from the Si₂ species. The data confirms detection to low ng/g levels for most elements. The detection limit data is in some part influenced by the ICP-MS spectrometer used (HP4500) but also by the reproducibility of the UV ablation process. To evaluate accuracy, the glass was analyzed using a calibration derived from the other NIST series glasses. Data is shown to be typically being within +/- 10% of recommended values.

Short term stability data can be visually assessed from the temporal plots shown on Figure 1. As can be seen, the signal rapidly reaches a steady state after 15 seconds and further demonstrates excellent precision throughout the ablation process. Precision data for boron as an example was measured at <1.5% RSD level, this being typical for data at the 30 ppm concentration level in this particular glass standard. The unique cell design ensures that signals equilibrate with good precision then and clear out rapidly with minimal memory.

LSX-100 Laser Ablation Signal Stability

ICP-MS: HP 4500, Laser Energy: 2 mJ, Pulse Rate: 20 Hz

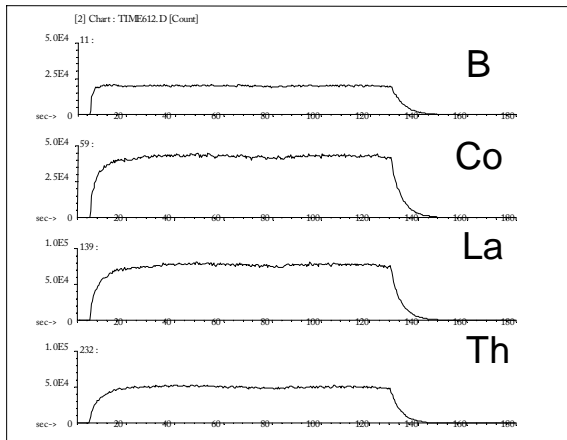


Figure 1. Temporal stability plots for elements in SRM 612

Conclusions: Analysis of NIST glass has become the *de-facto* standard when evaluating the quantitative performance of laser ablation ICP-MS. The CETAC LSX 100 system provides data which is comparable in accuracy to other systems but with the additional advantage of improved precision due in part to the superior beam profile from the TEM₀₀ operating mode.

Element	Recom	Measured	Det. limit
	ppm	ppm	ppb
B	32	31	10
Ti	50	45	55
Mn	39.6	40	31
Co	35.5	34	4
Ni	38.8	37	14
Cu	37.7	38	7
Rb	31.4	31	3
Sr	78.4	74	2
Ag	22	21	3
Ba	41	33.6	8
Ce	39	37	1
Nd	36	34.9	2
Yb	42	41.4	3
Au	5	4.6	3
Tl	15.7	16.3	2
Pb	38.6	33	5
Th	37.8	36	1.3
U	37.4	36	0.5

Table 1. Accuracy and detection limits for NIST SRM 612 glass measured with CETAC LSX 100 laser sampling system and ICP-QMS.