Elemental Scientific LASERS
INNOVATION TO ILLUMINATE

Applications Note

Rapid and Accurate Determination of Trace Elements in Copper Concentrate Fused Beads

SolidSample ICPMS – Fully Automated Laser Ablation ICPMS

Matrix Matched LA-ICPMS Calibration Standards:

Linearity and accuracy previously only thought possible through dissolution.

Full Automation:

Continuous robotic sample feed with barcoded sample tracking, data reduction, and result output.

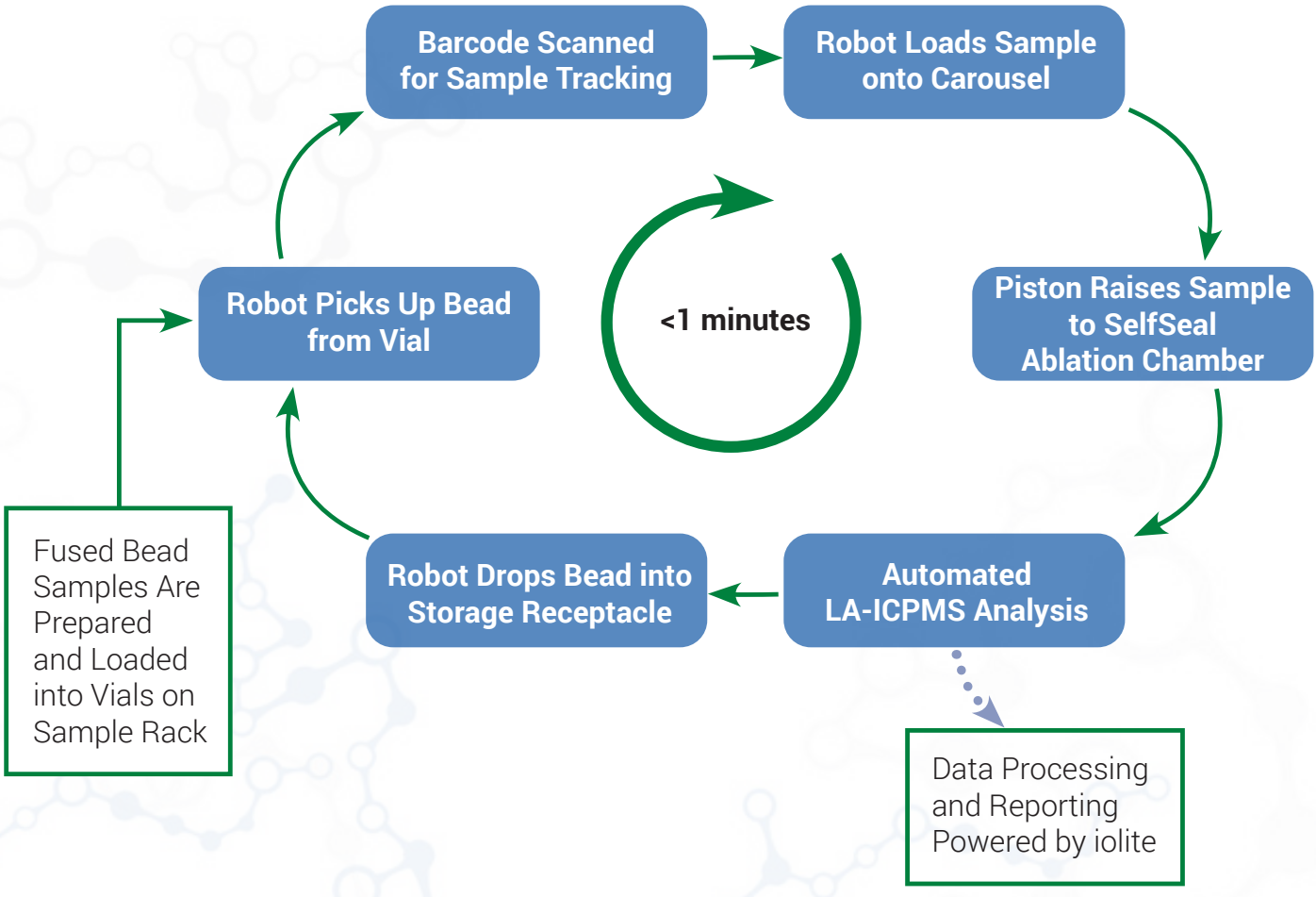
Faster Turn-Around Time:

Weeks of sample prep and analysis reduced to minutes!

Automating Multi-elemental Analysis of Solid Samples by ICPMS using SolidSample ICPMS

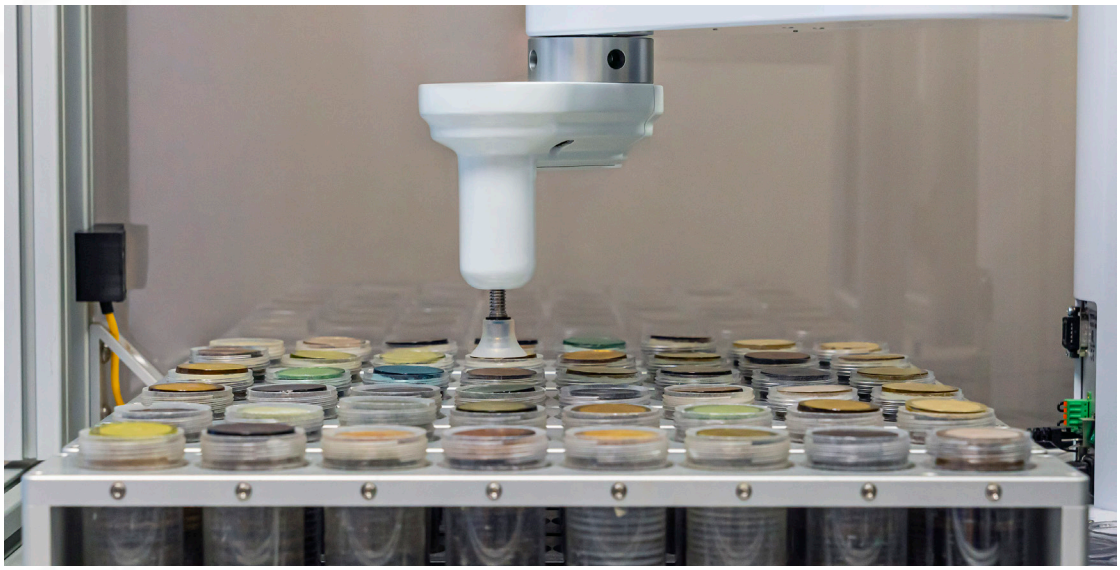
- Homogeneous, matrix-matched calibration standards can be used repeatedly for hundreds of analyses before refurbishing for continued use
- Accurate results and linear calibrations: Calibration standards produce R^2 values of ≥0.999 and recoveries within ± 5% in certified reference materials
- No acid dissolution, acid disposal, or exposure to hazardous materials. No more HF!
- Capable of analyzing >1000 samples in a single experiment
- Automatic quantification and result output powered by iolite data reduction software
- Sample to sample analysis time in <1 minute

SolidSample ICPMS Analytical Cycle



Robotic Arm for Sample Handling

Robotic arm uses vacuum suction cup to deliver samples to, and retrieve samples from rotary carousel.

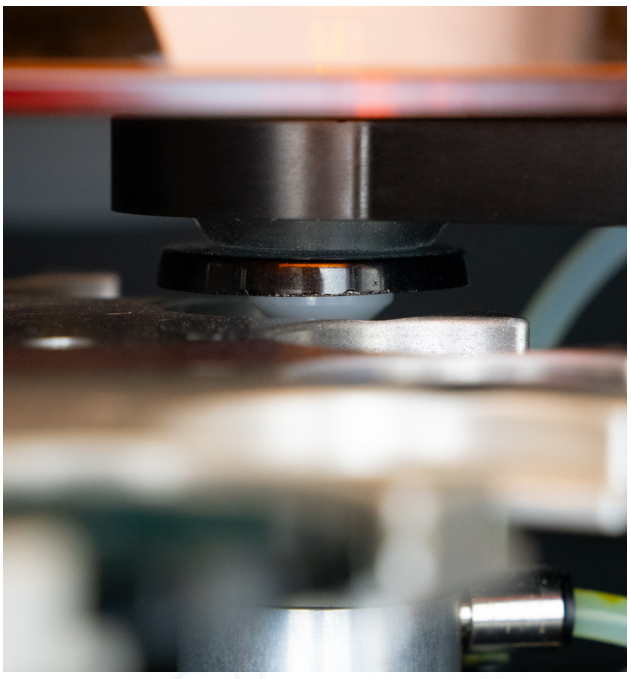


Rotary Carousel

Carousel rotates to present samples to the analysis position.

SelfSeal Ablation Chamber

Samples are driven up to the self-sealing ablation chamber via piston, creating an air-tight seal for solid sample analysis.



Sample and Standard Preparation

A series of Cu Concentrate matrix-matched, Lithium Tetraborate fused beads were created at ESI for calibration standards, using a proprietary method.

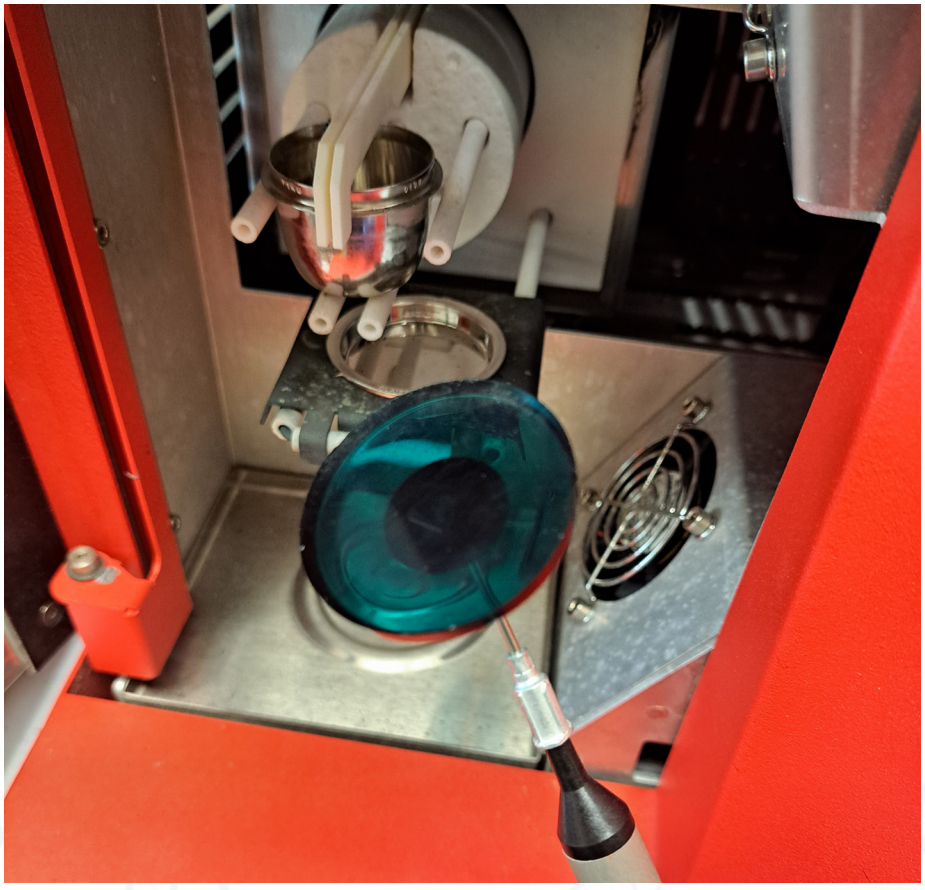
Tungsten was added to the beads for internal standardization.

8 spiked calibration standards (S1-S7) were created in accordance with table 1.

OREAS166 Cu Concentrate reference material was fused and treated as an unknown.

Table 1. ESI in-house fused bead calibration standard concentrations.

	Fused Bead Concentrations						
Calibration Standard ID	S1	S2	S3	S4	S5	S6	S7
Concentration (ppm)	0.39	2.3	4.6	9	19	33	50



Experimental Design

The following experiment was designed to:

1.) Verify the quality of calibration with ESI in-house Cu Concentrate calibration standards.

2.) Demonstrate long term accuracy over a 75 hour run, emulating high-throughput workflow of industrial mining analysis.

Unknown & calibration standards ran every 45 samples

- 1 set of calibration beads (S1-S7) were used repeatedly over the entire experiment (n=20)
- 1 Unknown bead (OREAS166) was used repeatedly over the entire experiment (n=20)

Blank fused beads used to fill entire sample rack.

1000 sample acquisitions

Experimental Conditions

Each sample was treated with a pre-ablation pass to remove any surface contamination.

Each time a bead was sampled, it was ablated with a single line scan.

He cell gas was used in the ICPMS to suppress polyatomic interferences.

Data processing was performed automatically using built-in SolidSample ICPMS data reduction application powered by iolite.

Table 3. SolidSample ICPMS and ICPMS Parameters

SolidSample ICPMS	
Preablation	
Spot size	150 µm
Repetition rate	10 Hz
Fluence	3.5 J/cm^2
Stage speed	150 µm /s
Ablation	
Spot size	100 µm
Repetition rate	40 Hz
Fluence	5.5 J/cm^2
Stage speed	100 µm /s
Transfer gas (He) flow	800 mL/min
ICPMS	
Masses monitored	^{24}Mg, ^{27}Al, ^{44}Ca, ^{47}Ti, ^{51}V, ^{55}Mn, ^{59}Co, ^{60}Ni, ^{72}Ge, ^{75}As, ^{95}Mo, ^{107}Ag, ^{115}In, ^{118}Sn, ^{121}Sb, ^{137}Ba, ^{208}Pb, ^{209}Bi, ^{238}U
Dwell time per amu	20 ms
Neb gas (Ar) flow	800 mL/min
Cell gas (He) flow	2.5 mL/min

Results

The resulting data indicates that analytical goals were met. Calibration curves for most elements showed R^2 values greater than 0.999. Most elements recovered within ± 5% of expected values. Crucial elements for Cu Concentrate analysis, such as As, Ag, Sb, Bi, and Pb showed exceptional results.

Results: Recovery and LOD

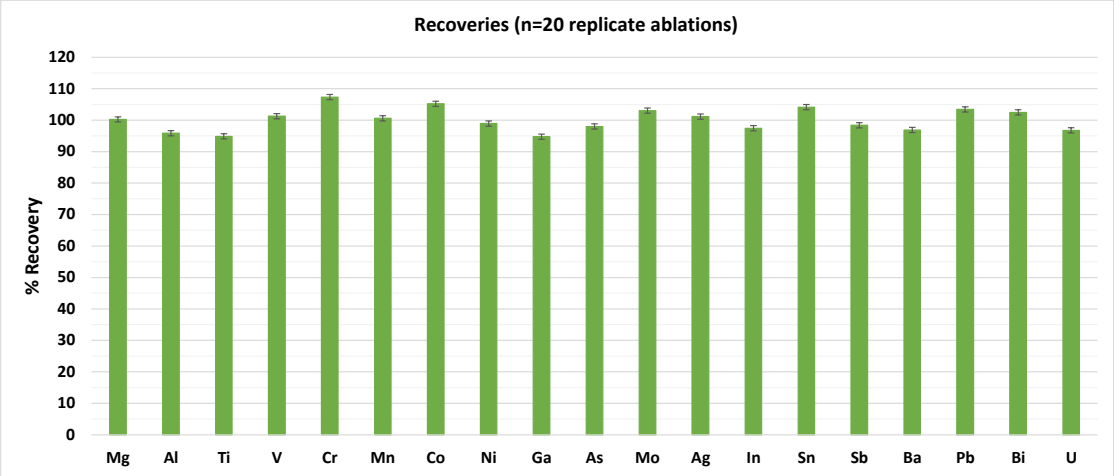


Figure 4. Recoveries. Error bars indicate standard error of 20 replicate ablations.

Table 6. Measured vs Accepted Values

Long-term Accuracy (ppm)					
Elements	Measured	Accepted	% Diff	% Recov	R2
^{25}Mg	743.6	741.7	0.2	100	0.9997
^{27}Al	512.4	534.5	-4.2	96	0.9995
^{47}Ti	17.1	18	-5.1	95	0.9989
^{51}V	0.4	0.4	1.3	101	0.9999
^{52}Cr	2.7	2.5	7.3	107	0.9997
^{55}Mn	15.6	15.5	0.6	101	0.9993
^{59}Co	151.6	144.1	5.2	105	0.9994
^{60}Ni	4.9	5	-1.1	99	0.9990
^{72}Ga	0.1	0.1	-5.3	95	0.9999
^{75}As	217.3	221.7	-2	98	0.9999
^{95}Mo	0.6	0.6	3	103	1.0000
^{107}Ag	0.7	0.7	1.2	101	0.9998
^{115}In	0.3	0.3	-2.6	97	0.9990
^{118}Sn	0.4	0.4	4.1	104	0.9995
^{121}Sb	1.6	1.6	-1.6	98	0.9999
^{137}Ba	1.6	1.7	-3.1	97	0.9951
^{208}Pb	10.3	10	3.4	103	0.9999
^{209}Bi	2.6	2.5	2.5	102	0.9999
^{238}U	0.1	0.1	-3.2	97	0.9976

Table 7. Limits of Detection

LOD (Pettke) (ppb)																		
^{24}Mg	^{27}Al	^{47}Ti	^{51}V	^{52}Cr	^{55}Mn	^{59}Co	^{60}Ni	^{71}Ga	^{75}As	^{95}Mo	^{107}Ag	^{115}In	^{118}Sn	^{121}Sb	^{137}Ba	^{208}Pb	^{209}Bi	^{238}U
727.9	496.0	55.1	4.8	31.9	33.4	5.6	233.8	4.0	23.4	3.7	4.2	5.0	332.1	2.9	3.8	1.5	0.3	0.3

Results: Calibration Curves

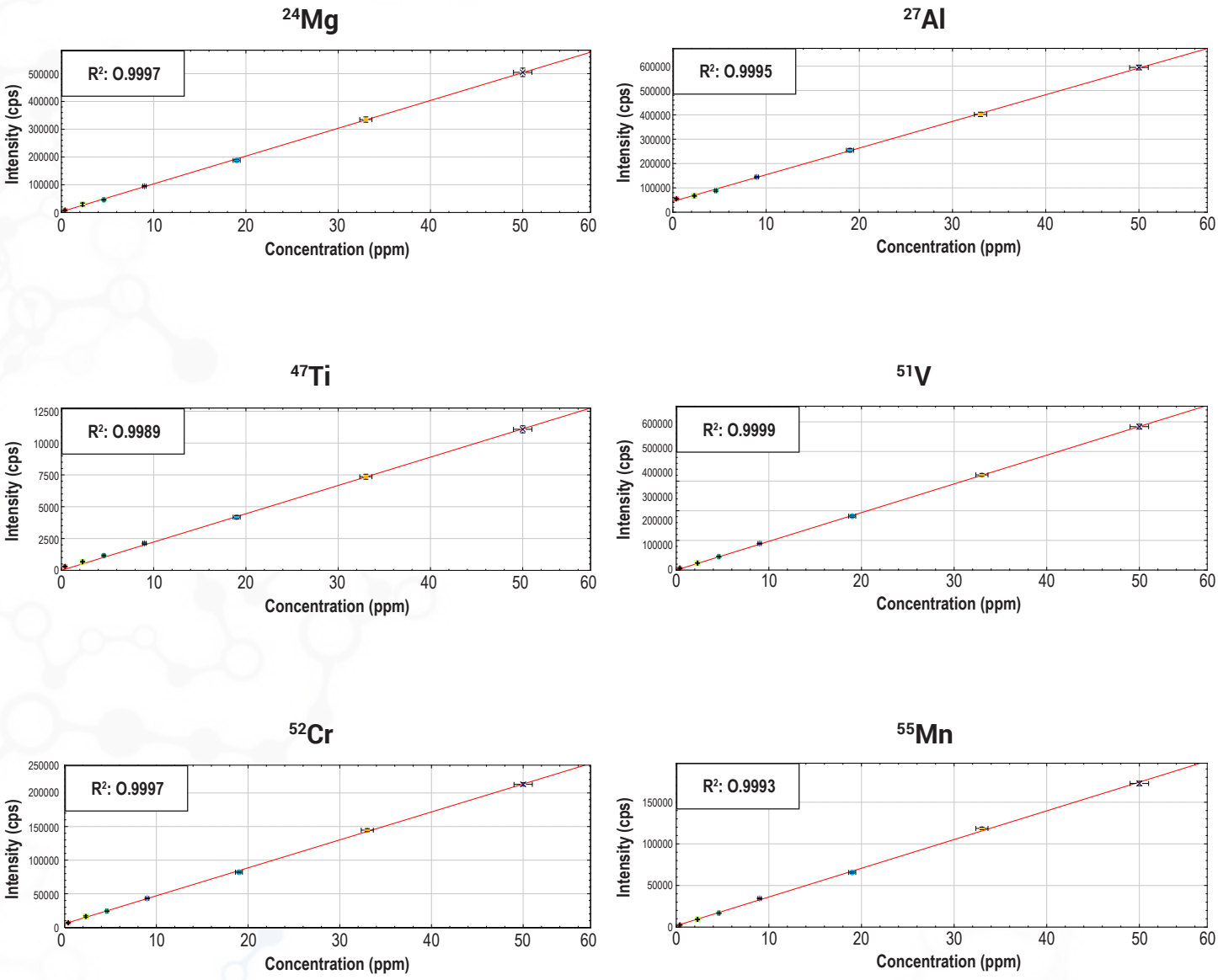


Figure 5a. Calibration Curves

Results: Calibration Curves

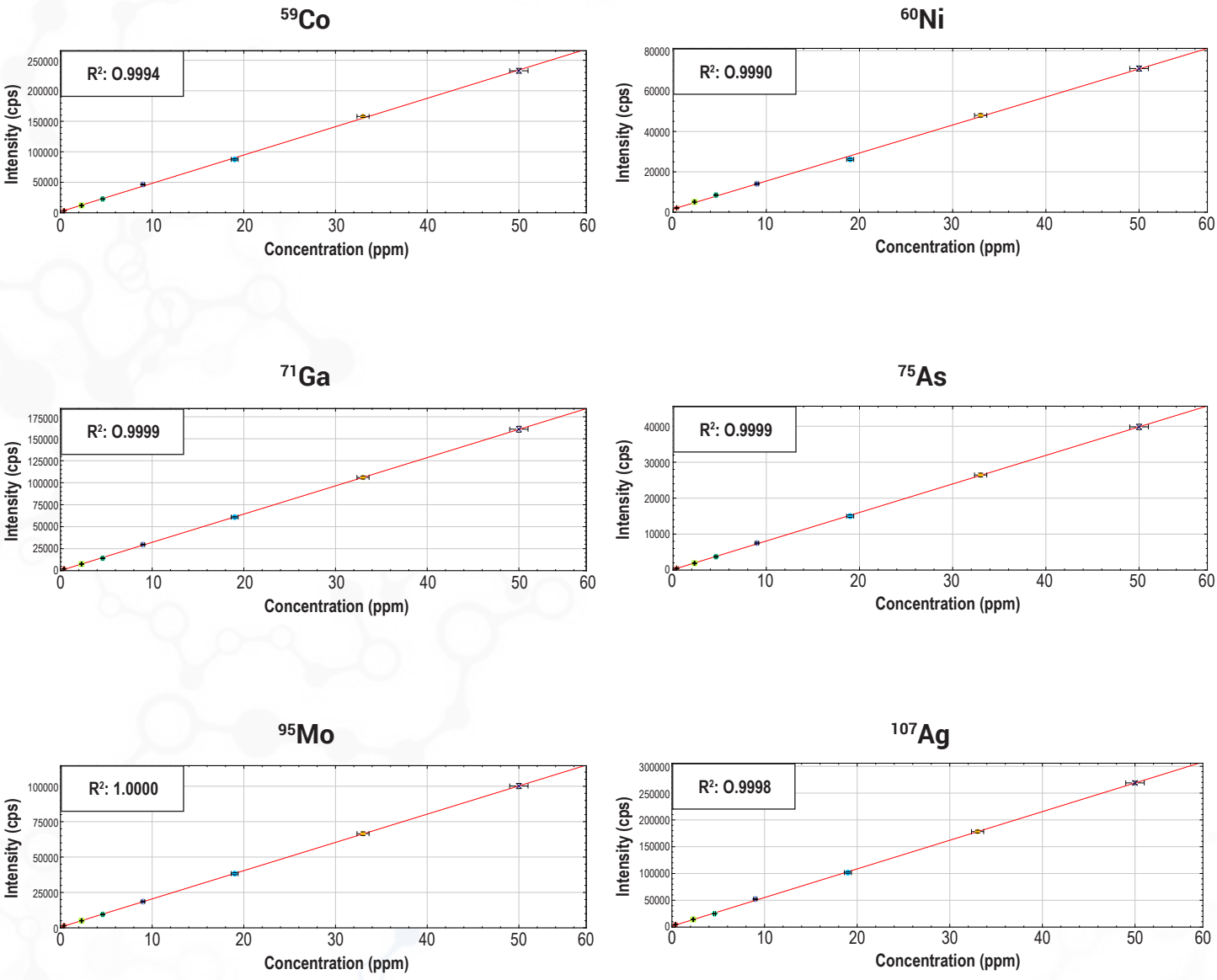


Figure 5b. Calibration Curves

Results: Calibration Curves

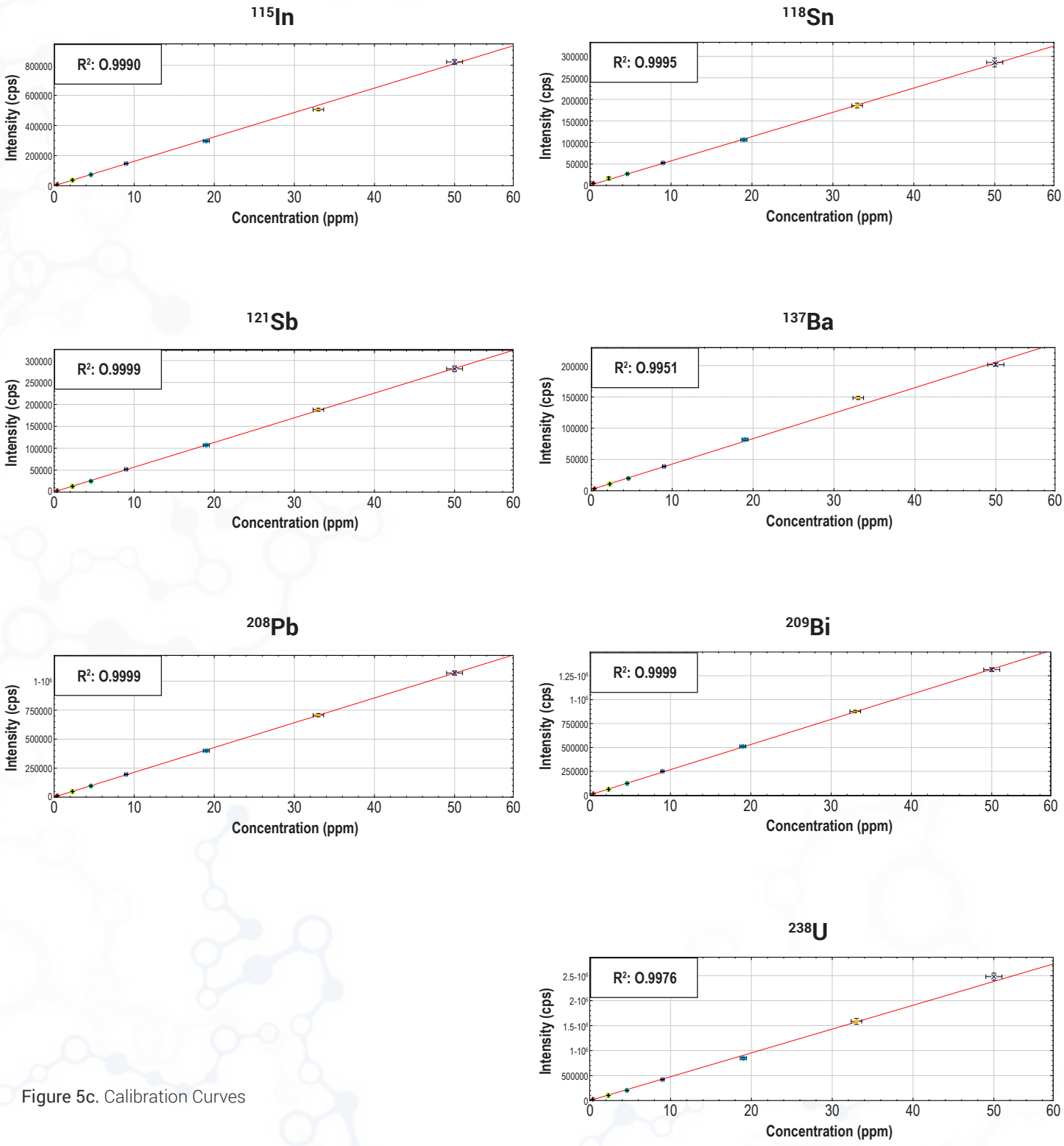


Figure 5c. Calibration Curves

Conclusion

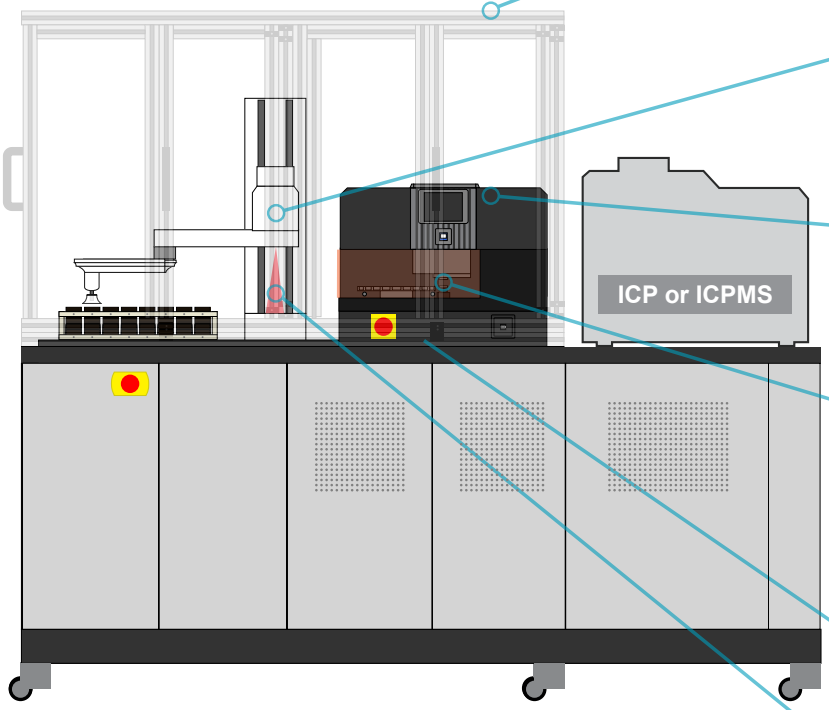
SolidSample ICPMS has been demonstrated to provide accurate and precise data for trace elements Cu Concentrate fused bead samples.

ESI Cu Concentrate calibration standards accurately quantified OREAS166 Cu Concentrate samples.

- Calibration curves for most elements showed R^2 values of better than 0.999.

- Almost all elements recovered within ±5% of known values.

SolidSample ICPMS can meet the rigorous demands for high throughput mining samples with a lower cost of ownership compared to other techniques.



Class 1 Laser Interlocked Enclosure

- Customizable for sample volumes
- Add/remove samples during operation
- Extraction available

Robotic Sample Handling

- Reduced operator input
- Fully-automated sample handling
- 24/7 operation

Laser Module

Various laser modules are available depending on the application requirements

SelfSeal Sample Chamber

- 5 s gas purge per sample
- Automated chamber cleaning
- High sample transport efficiency
- Enhanced signal response
- Rapid sample changeover

High-Throughput

- Up to 5X faster than conventional laser ablation

Barcode Scanner

- Reads sample ID
- Two-way LIMS communication
- Auto-builds real-time ICPMS run list

Figure 1. SolidSample ICPMS features diagram.

Elemental Scientific LASERS

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