Fully Automated Matrix Removal and Sample Purification For ICPMS and MC-ICPMS

Elemental Scientific

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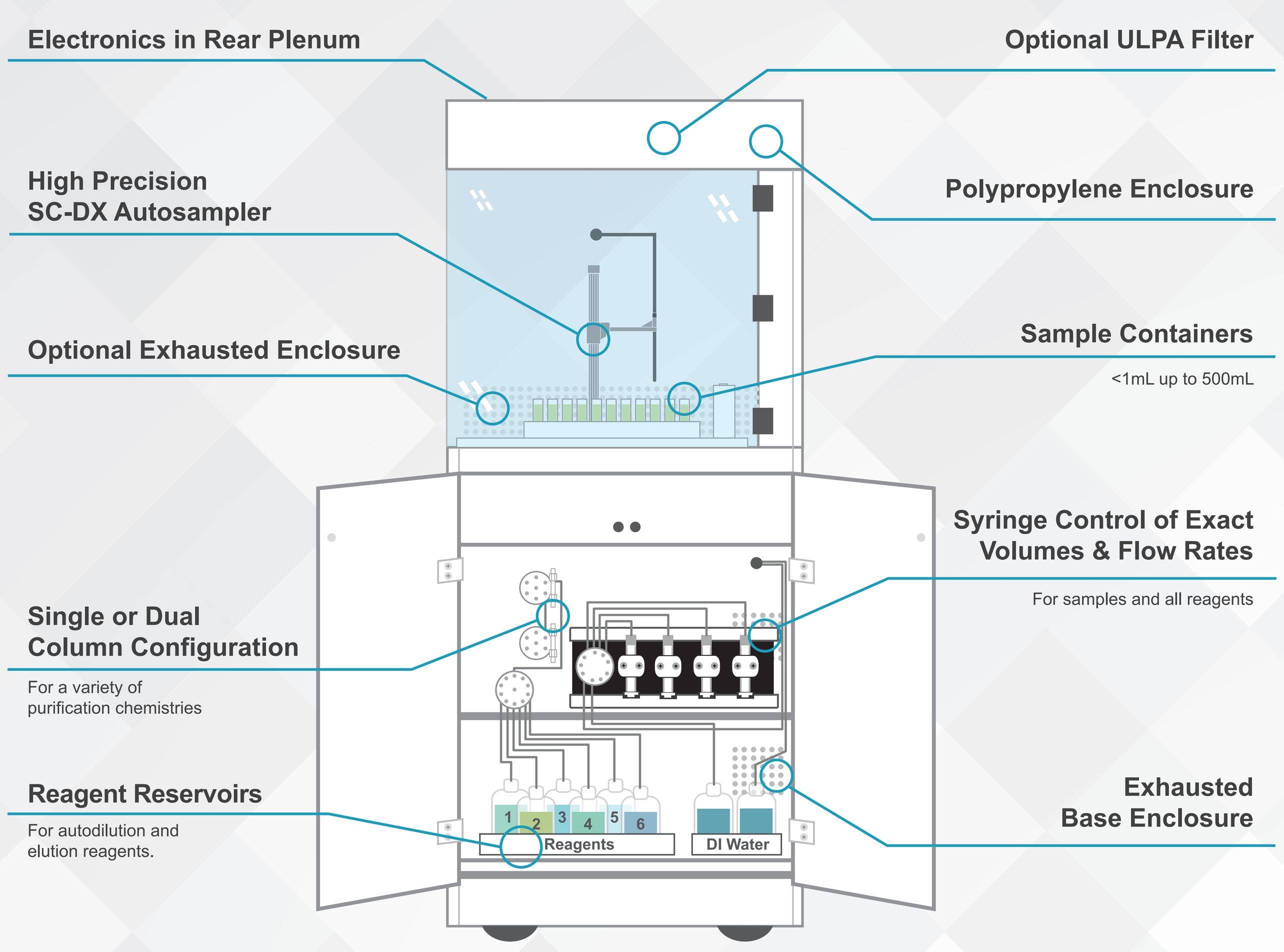
Abstract

To better understand bio, geo and biogeochemical processes, large quantities of high quality data are required. Over the past decade, advances in sample introduction techniques combined with ICP-MS and MC-ICP-MS instrumentation have improved analytical precision, accuracy and detection limits for trace metal concentration and high precision isotopic ratio determination. However, many analyses still require significant sample preparation for which the laboratory bottleneck remains. The recent development of automated sample preparation systems (sea*FAST*™ and prep*FAST* MC™, ESI, Omaha, USA) provide a platform to address this bottleneck for a range of sample types and applications. The system uses specially designed fluoropolymer valves, precise and accurate syringe control, and an autosampler all controlled by a flexible, yet simple, software package to automate tedious and time consuming sample preparation. The system allows sample loading, multiple acid washes, column conditioning and elution cycles necessary to isolate elements of interest and automatically collect or directly inject eluent fractions.

Case studies from existing labs will be used to illustrate the systems wide range of capabilities. The sea*FAST*™ is used to determine a variety of elements in seawater ranging from single digit ngL-1 concentrations of Mn, Fe, Co, Ni, Cu and Zn to ultra-trace levels (10s of pgL-1) of REEs. Newly developed protocols for the prep*FAST* MC™ automate the purification of B, Ca, Fe, Cu, Zn, Sr, Pb, U and Th for isotopic analysis from a variety of sample types.

The sea*FAST*™ and prep*FAST* MC™ maximizes sample throughput and minimizes costs associated with personnel and consumables providing an opportunity to greatly expand research horizons in fields where large isotopic data sets are required, including archeology, geochemistry, climate/environmental science, biomedical sciences and food authentication.

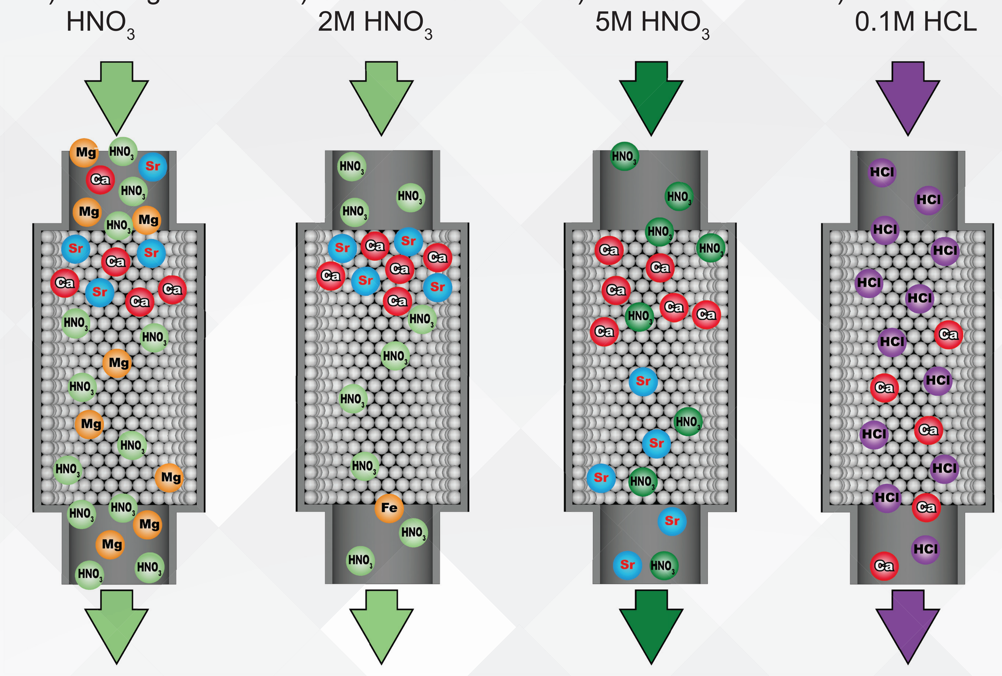
prep*FAST* MC™

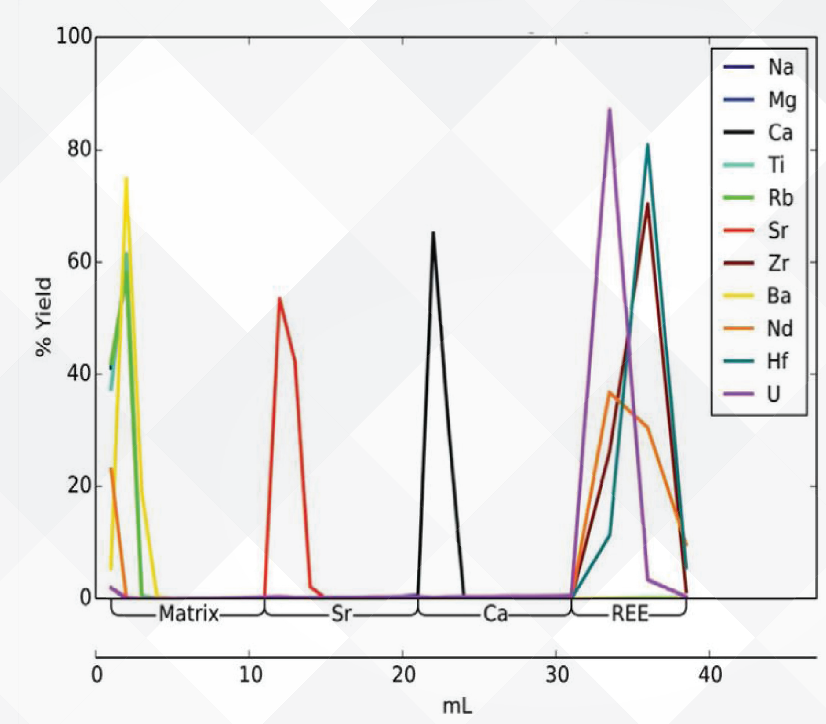


Applications

- Ca Sr Nd Pb: T. Zimmermann, et al. *Optimization of novel automated sample preparation systems for elemental and isotopic analysis of environmental samples via (MC) ICP-MS*
- Nd: N. S. Saji, D. Wielandt, C. Paton and M. Bizzarro, J. Anal. At. Spectrom., 2016, DOI: 10.1039/C6JA00064A.
- Sr: A. Retzmann, et al. *Optimized fully-automated Sr/matrix separation for Sr isotopic analysis in soil extracts to generate isoscapes of bioavailable Sr for archaeological migration studies*
- Cu: T. Gabriel Enge, M. Paul Field, Dianne F. Jolley, Heath Ecroyd, M. Hwan Kim and Anthony Dossetoa, J. Anal. At. Spectrom., 2016, DOI: 10.1039/C6JA00120C.
- Sr Ca: S. J. Romaniello, M. P. Field, H. B. Smith, G. W. Gordon, M. H. Kim and A. D. Anbar, J. Anal. At. Spectrom., 2015, DOI: 10.1039/C5JA00205B.
- B: E. De La Vega, Automation of boron purification for δ11B analysis with MultiCollector Inductively Coupled Plasma Mass Spectrometry

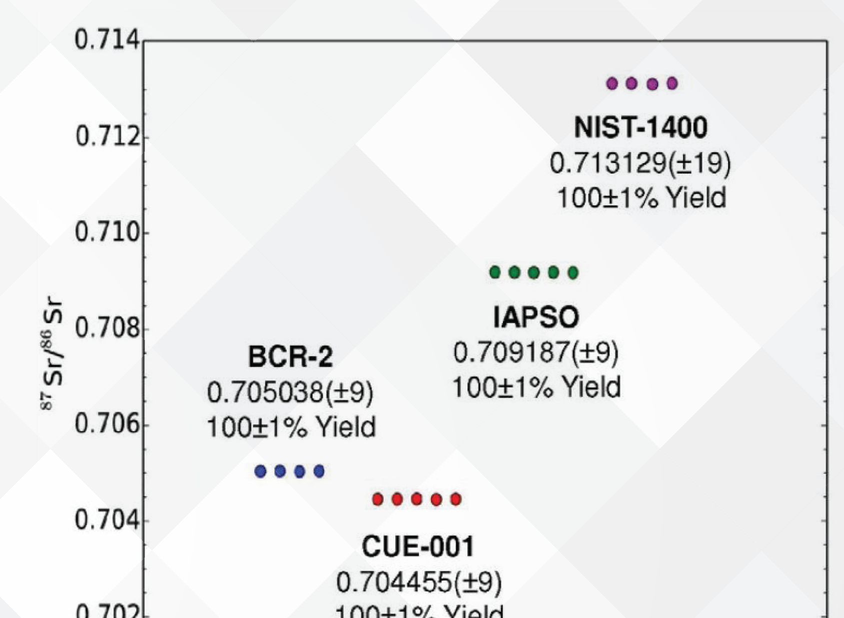
prep*FAST* MC Sr, Ca Column Chemistry





Elution profile for Sr Ca Method

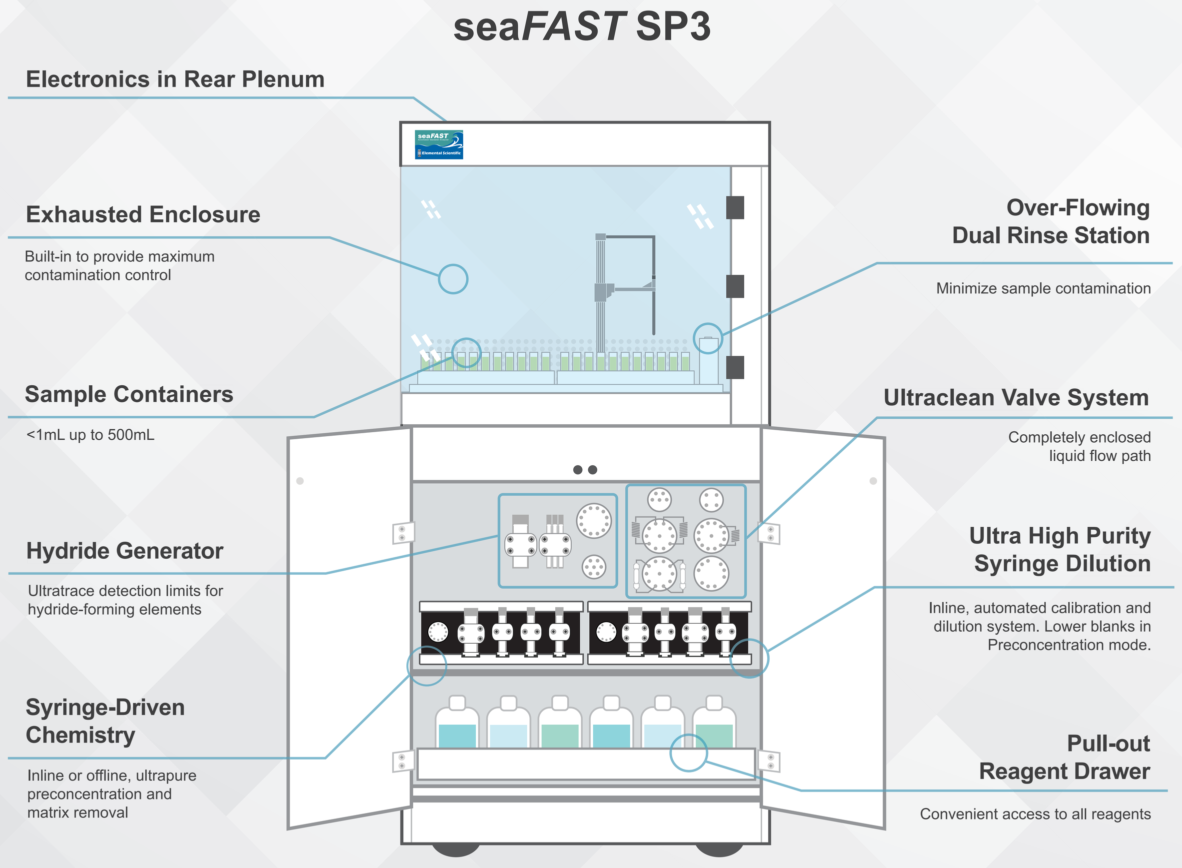
Column: 1mL ESI Ca-Sr Column			
Step	**Purpose**	**Volume**	**Reagent**
1	Condition Column	10 mL	2M HNO_3 +1% wt. H_2O_2
2	Load Sample	1 mL	2M HNO_3
3	Elute Sample Matrix	10 mL	2M HNO_3 +1% wt. H_2O_2
4	Elute Sr	10 mL	6M HNO_3
5	Elute Ca	10 mL	12M HNO_3
6	Elute REEs, Hf, Cd, U	10 mL	1M HF



Average values and yields are given below standard labels for $^{87/86}Sr$ with a population averaged 2σ error. Randomly interspersed blanks (not shown) contained <100 pg Sr.

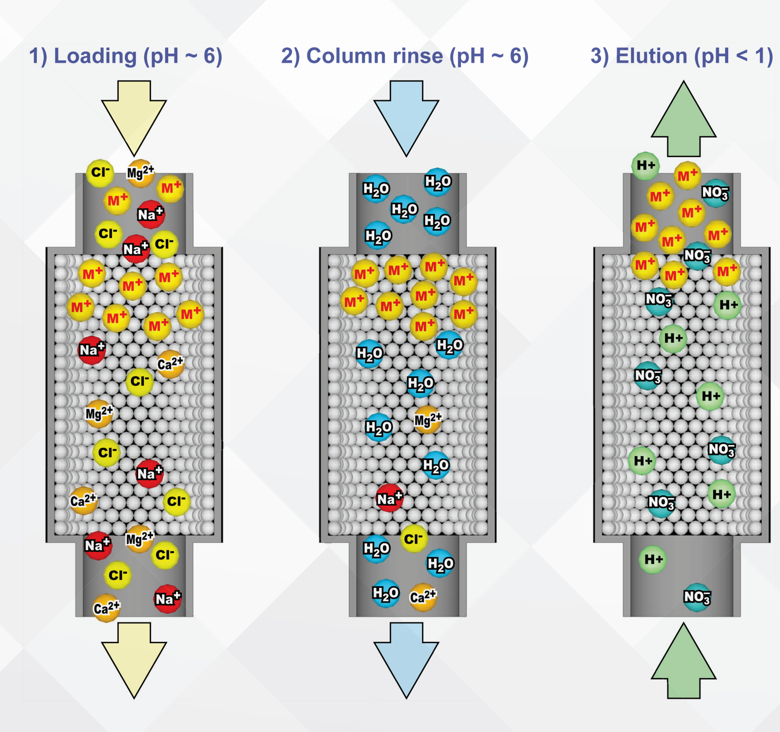
sea*FAST*

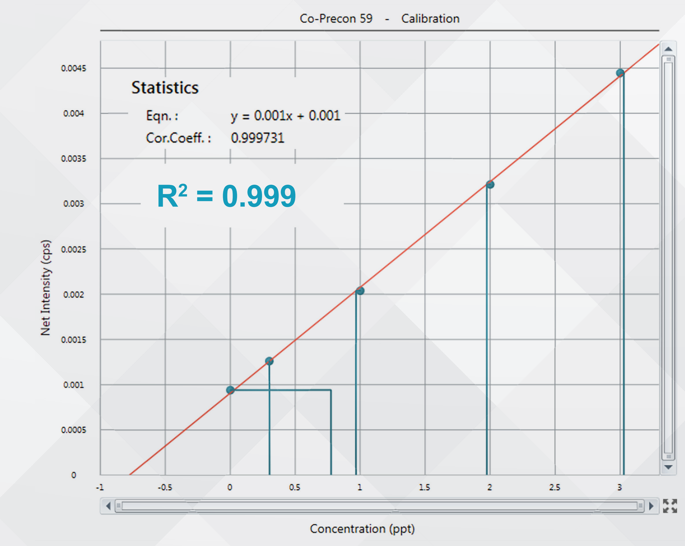
sea*FAST* SP3



Applications

- Offline REE: Behrens, M.K.; Muratli, J.; Pradoux, C.; Wu, Y.; Böning, P.; Brumsack, H.J.; Goldstein, S.L.; Haley, B.; Jeandel, C.; Paffrath, R.; Pena, L.D.; Schnetger, B.; Pahnke, K.; Marine Chemistry, 2016, DOI: 10.1016/j.marchem.2016.08.006.
- Online REE: Hathorne, E.C.; Haley, B.; Stichel, T.; Grasse, P.; Zieringer, M.; Frank, M.; Marine Chemistry, 2012, DOI: 10.1016/j.marchem.2016.08.006.
- Mn, Fe, Co, Ni, Cu, Zn: Lagerström, M.; Field, M.; Séguret, M.; Fischer, L.; Hann, S.; Sherrell, R.; Marine Chemistry, 2013, DOI: 10.1016/j.marchem.2013.06.001.
- Ti, Zr, Nb, V, W, Mo: Poehle, S., Katja Schmidt , Andrea Koschinsky; Deep Sea Research Part I: Oceanographic Research Papers, 2015.

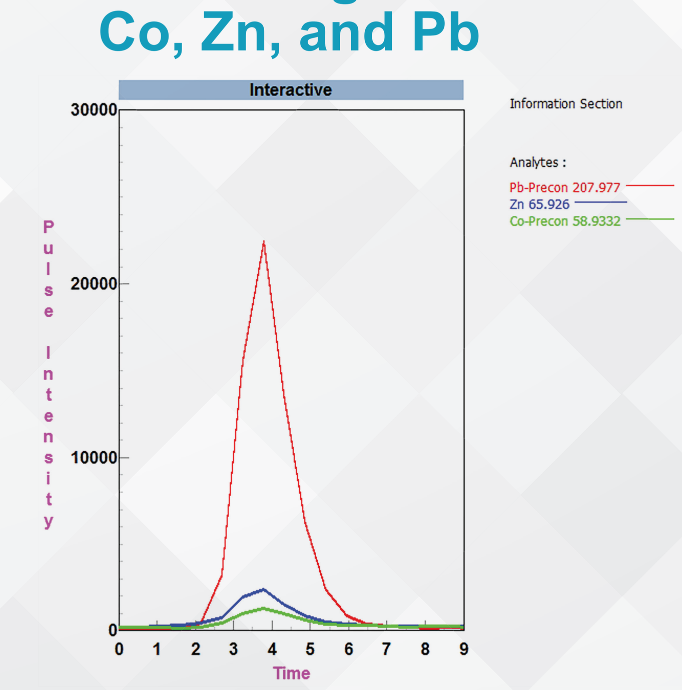




Automatic standard addition calibrations in GSP for Co, no blank subtraction.

sea*FAST* Preconcentration Column

Transient Signals for Co, Zn, and Pb



GSP sample elution profile.

D1 North Pacific Intercalibration Sample, 1000m									
	Consensus Value			sea*FAST* HR-ICPMS			sea*FAST* Q-ICPMS		
Fe	38	±	2.3	38.1	±	0.5	37.2	±	0.6
Co	2.74	±	0.3	2.58	±	0.05	2.89	±	0.04
Ni	516	±	16	519	±	7			
Cu	148	±	7.2	138	±	2			
Zn	496	±	23	505	±	4	488	±	7
Ag	na			2.6	±	0.2	2.4	±	0.2
Pb	5.88	±	0.6	5.2	±	0.1	5.70	±	0.02

Results for D1 measured with sea*FAST* and HR-ICPMS and Q-ICPMS are compared with consensus values, showing excellent agreement. Ni and Cu are >30 ppt in both GSP and D1, so they were not calibrated with the low concentration elements by Q-ICPMS. Ag has no consensus value.