prep*FAST* MC

Automated Column Chromatography for High Precision Isotope Analysis

Brief

The prep*FAST* MC is a fully automated, low pressure chromatography system that isolates elements of interest from the sample matrix and collects multiple discrete eluent fractions for precise isotopic analysis. The syringe-driven system allows sample loading, multiple acid washes, column conditioning and elution cycles all at user-defined intervals (time, volume and flow rate).

Features

- Fully automated
- Syringe control
  - Load exact volume
  - Dispense exact volume
  - Precise flow rates
- All fluoropolymer flow path
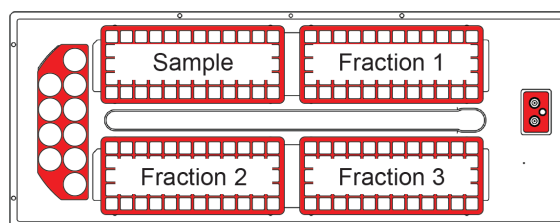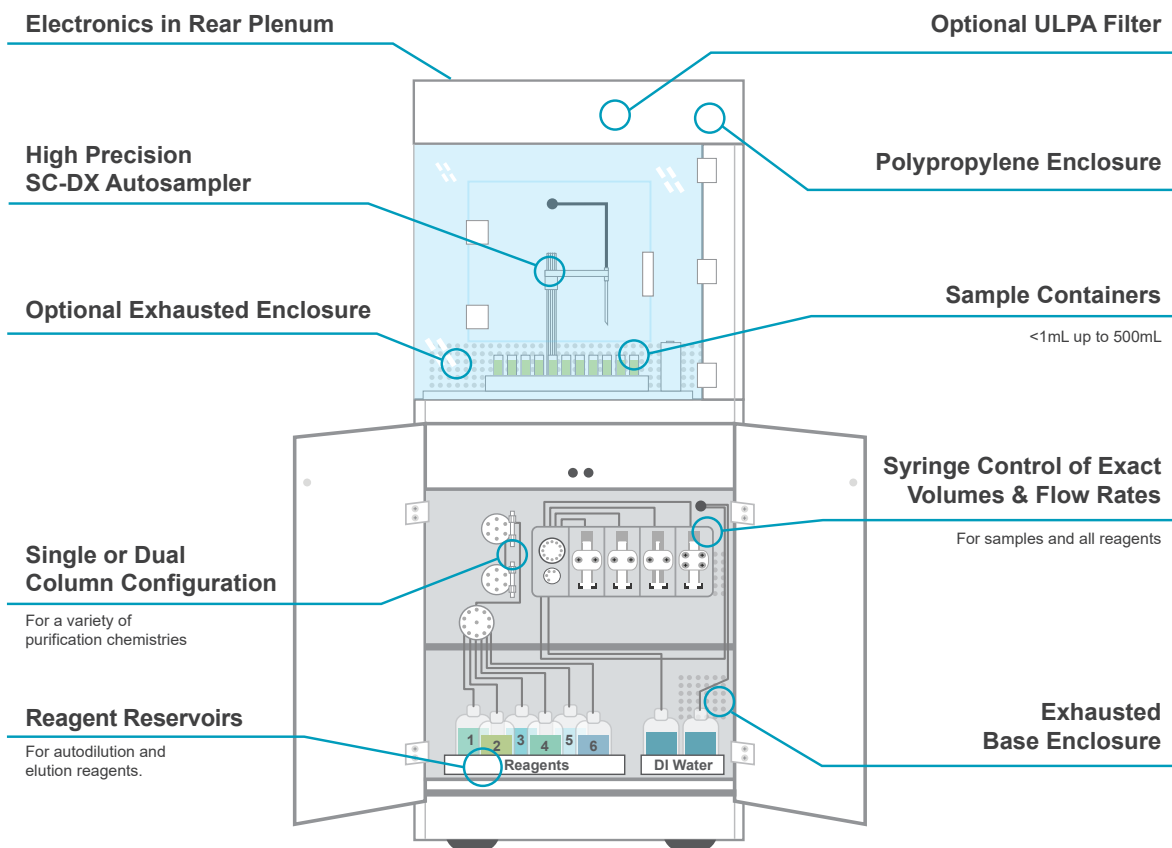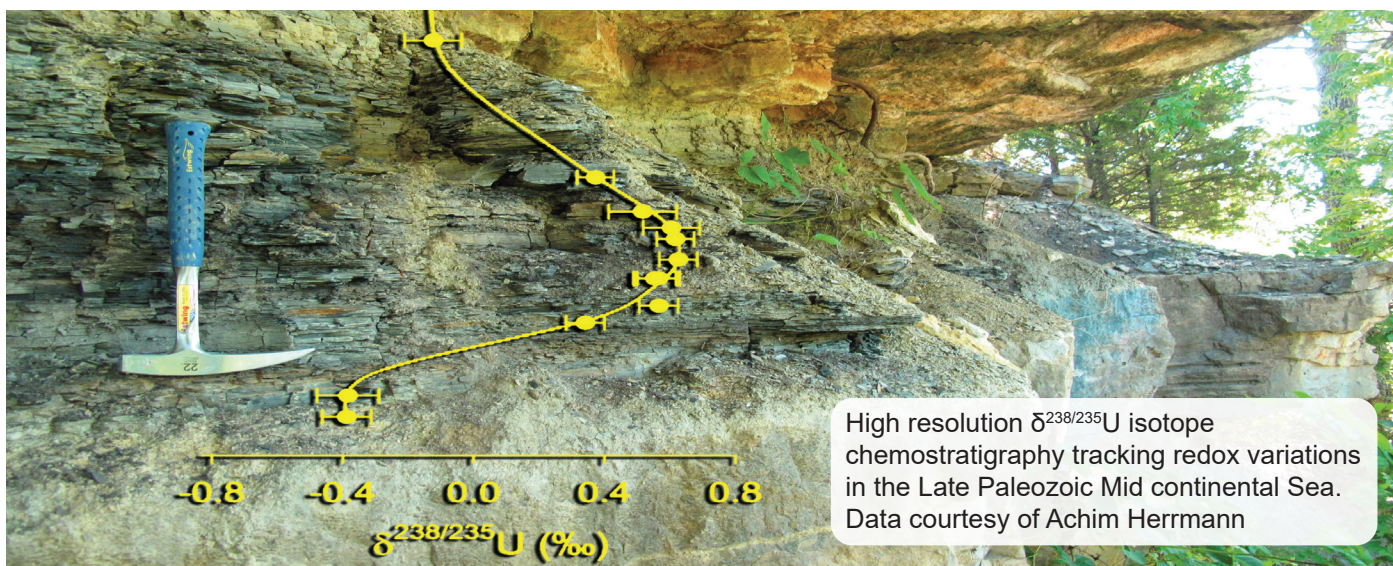- Multiple destination locations
- Flexible chemistry

Figure 1. 4DX top view with locations for Sample, Fraction 1, Fraction 2, and Fraction 3. Vial sizes, styles and rack configurations are flexible.

Electronics in Rear Plenum

Optional ULPA Filter

High Precision SC-DX Autosampler

Polypropylene Enclosure

Optional Exhausted Enclosure

Sample Containers

<1mL up to 500mL

Syringe Control of Exact Volumes & Flow Rates

For samples and all reagents

Single or Dual Column Configuration

For a variety of purification chemistries

Reagent Reservoirs

For autodilution and elution reagents.

Exhausted Base Enclosure

Uranium Isotopes

High resolution $\delta^{238/235}$U isotope chemostratigraphy tracking redox variations in the Late Paleozoic Mid continental Sea. Data courtesy of Achim Herrmann

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High-resolution isotope ratio stratigraphy requires the routine analysis of large numbers of samples. To reduce interferences and minimize matrix effects, extensive purification procedures are used to isolate elements from their natural matrix. Current purification protocols require manually feeding gravity-driven separation columns, a process that is both costly and time consuming. This laboratory bottleneck is eliminated with the prep*FAST* MC, an automated, low-pressure ion exchange chromatography system that can process from 1 to 60 samples in unattended operation. The syringe-driven system automatically isolates elements of interest and collects up to 3 discrete fractions at user-defined intervals (time, volume and flow rate). The combination of maximizing sample throughput and minimizing costs associated with personnel and consumables provides 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.

Figure 2. Fully-automated chromatographic separation of uranium (red) from all other matrix elements in BCR-2 (Columbia River Basalt) using the prep*FAST* MC system. Elution profile was collected online in real-time using a Thermo XSERIES ICPMS. Data courtesy of Stephen Romaniello and Gwyneth Gordon (Arizona State University).
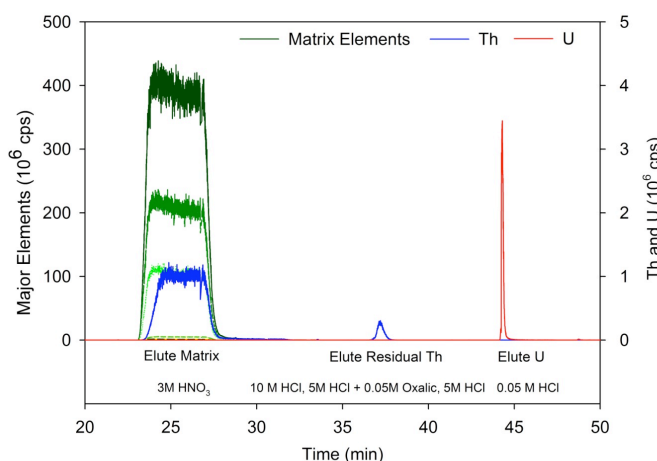
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ARIZONA STATE UNIVERSITY

Stephen Romaniello, Gwyneth W. Gordon, Achim Herrmann and Ariel D. Anbar
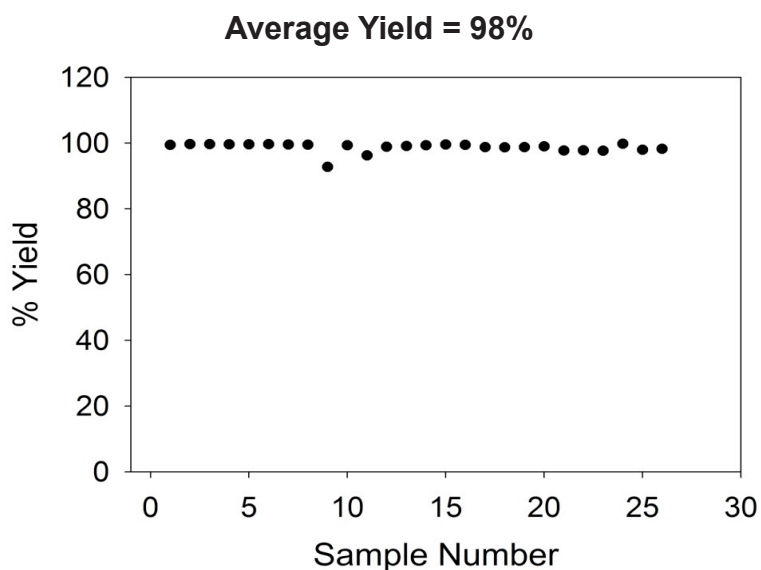
Average Yield = 98%

Figure 3. Uranium recovery for 26 consecutive samples with fully-automated cleaning and reuse of the separation column. The average yield was 98% and the minimum yield was 92%. Data courtesy of Stephen Romaniello and Gwyneth Gordon (Arizona State University).
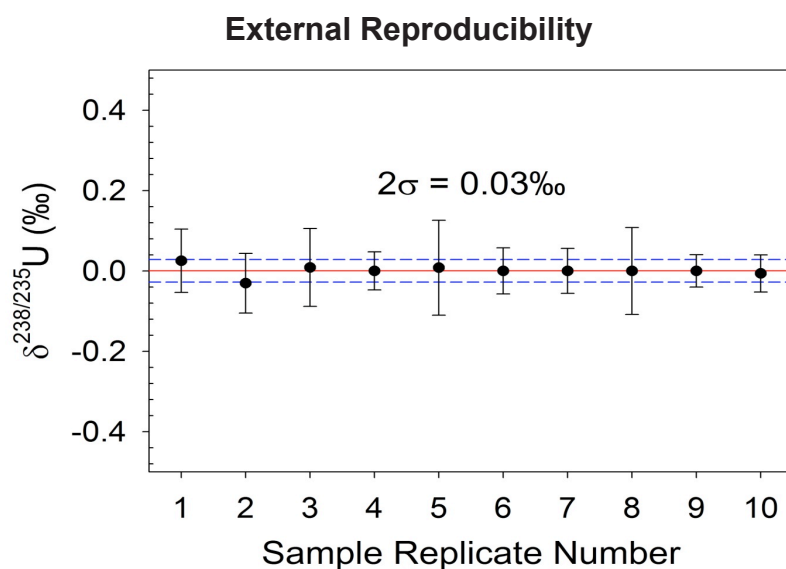
External Reproducibility

$2\sigma = 0.03‰$

Figure 4. External reproducibility of $\delta^{238/235}U$ for 10 aliquots CRM145 independently processed through chemistry over a 1 week period interspersed with natural samples using the prep*FAST* MC. Errors bars indicate the 2σ precision of replicate measurements on a single sample aliquot (n≥3). Blue dashed lines indicate the 2σ precision for the means of all sample aliquots. Data collected using a Thermo Neptune MC-ICPMS and courtesy of Stephen Romaniello and Gwyneth Gordon (Arizona State University).
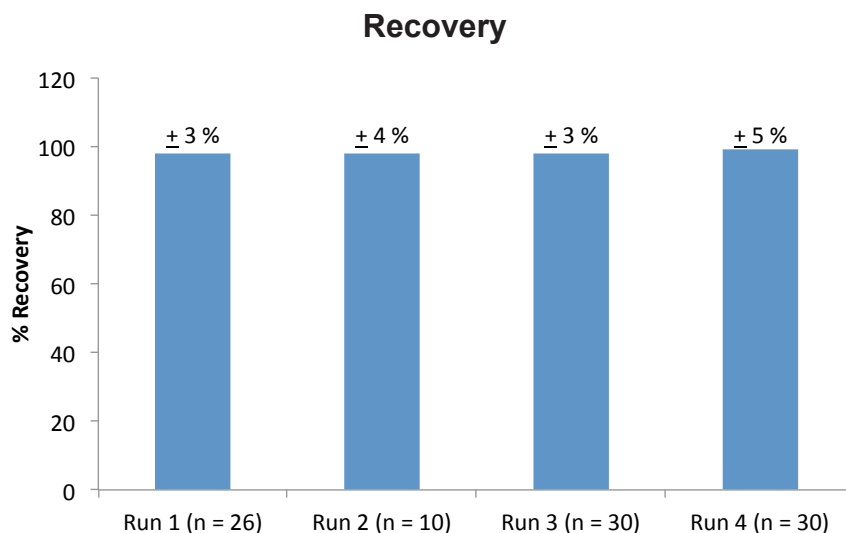
Recovery

Figure 5. Data collected over four runs of thirty automated extractions illustrates constant, complete recovery for within run, and run to run extractions. The column was replaced for each run. Within run replicates are indicated by "n" and recovery determinations are within analytical uncertainty.

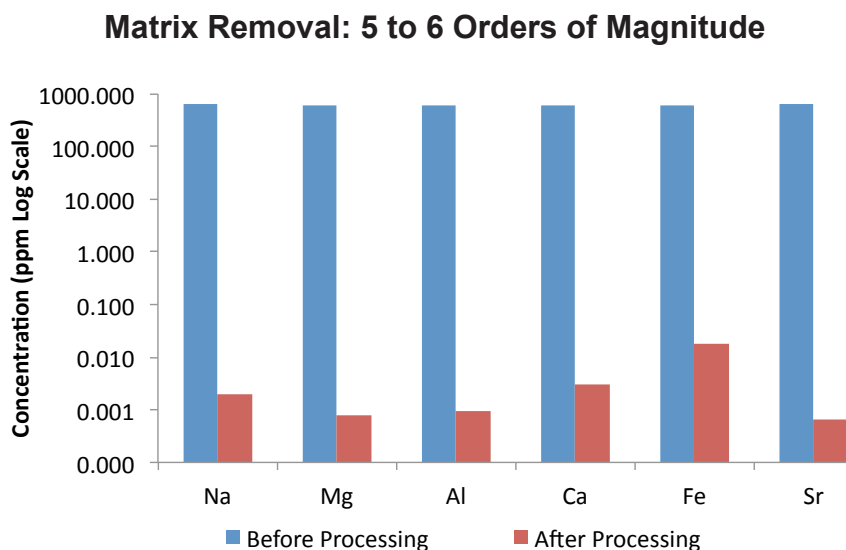## Matrix Removal: 5 to 6 Orders of Magnitude

Figure 6. The concentration of major matrix elements determined in final extractions indicates greater than 99.999% matrix removal. Initial concentration of 500 ppm matrix elements is reduced to less than 20 ppb or lower.

Strontium Isotopes

^{87}Sr / ^{86}Sr

	Determined	Reference
Rock (BCR-2)	0.70503 ± 2	0.70501 ± 8
Bone (NIST 1400)	0.71312	0.71310 ± 2
Seawater (IAPSO)	0.70915 ± 2	0.70917 ± 1

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High-precision isotope analyses in forensic anthropology, nuclear forensics and food authentication applications, requires the generation of large data sets. A new, fully automated Sr purification method for the prep*FAST* MC eliminates the sample preparation bottle neck. Reproducibility, reliability, recovery, blank and carry over are at required levels for the determination of Sr isotopes in a variety of natural samples. Potential applications of prep*FAST* MC are illustrated by accurate and precise determination of Sr isotopes in rock, bone, and seawater reference materials. Unattended, the prep*FAST* MC can process up to 48 samples in 24 hours and, (depending on chemistry), collect several purified extractions from each sample.
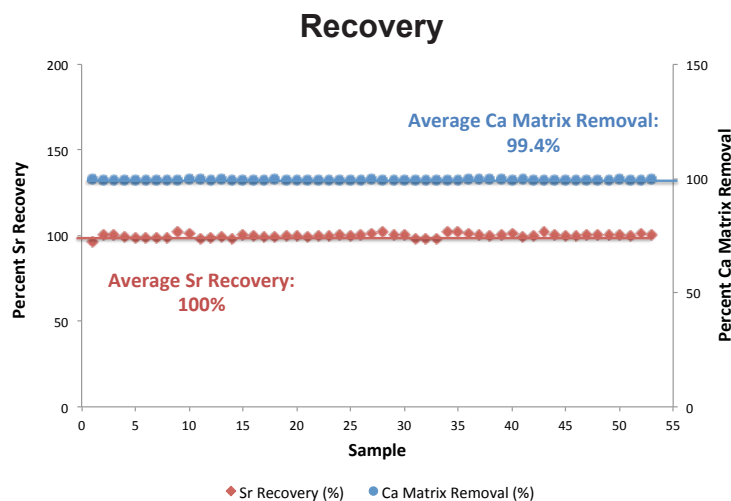
Figure 7. Strontium recovery for 55 consecutive, fully automated extractions is plotted. 100% recovery and near complete removal of Ca provides pure fractions for accurate and precise determination of isotopic ratios.

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Sr, Ca Column Chemistry

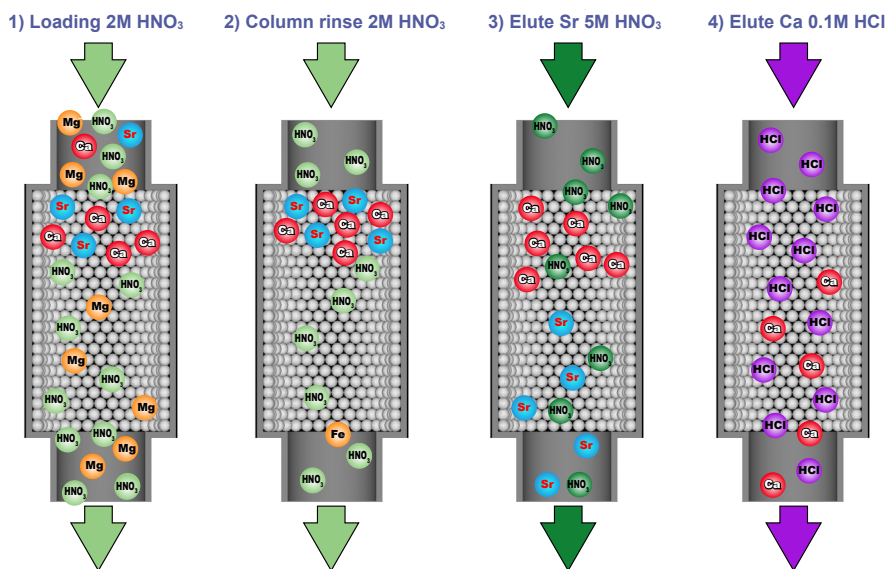1) Loading 2M HNO₃   2) Column rinse 2M HNO₃   3) Elute Sr 5M HNO₃   4) Elute Ca 0.1M HCl

Figure 8. Illustration of fully-automated chromatographic separation of Sr from Ca and other matrix components.
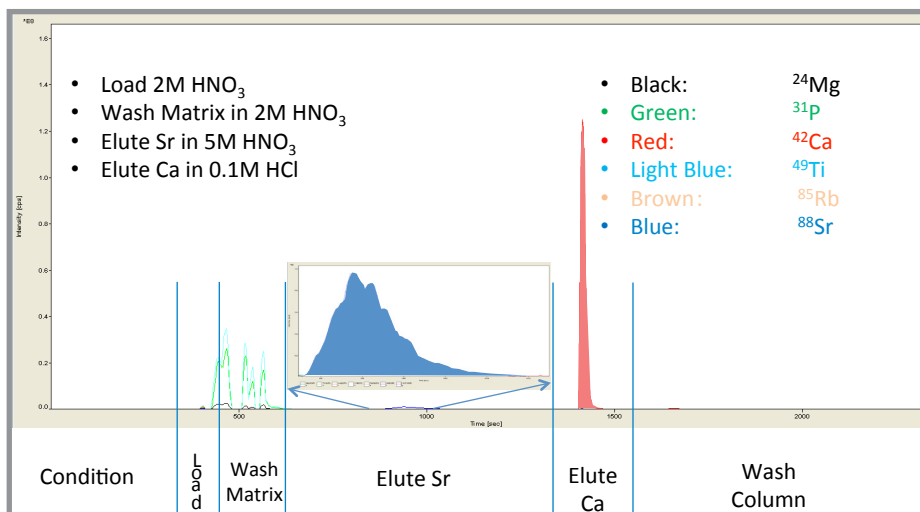
Sr and Ca Elution Profile

- Load 2M HNO$_3$
- Wash Matrix in 2M HNO$_3$
- Elute Sr in 5M HNO$_3$
- Elute Ca in 0.1M HCl

- Black: ^{24}Mg
- Green: ^{31}P
- Red: ^{42}Ca
- Light Blue: ^{49}Ti
- Brown: ^{85}Rb
- Blue: ^{88}Sr

| Condition | Load | Wash Matrix | Elute Sr | Elute Ca | Wash Column |

Figure 9. Online, real-time elution profile collected on the Element 2 (Thermo) illustrates the fully-automated chromatographic separation of Sr and Ca from matrix components. In carbonate samples it is possible to also collect the Mg fraction during sample load and wash cycles.
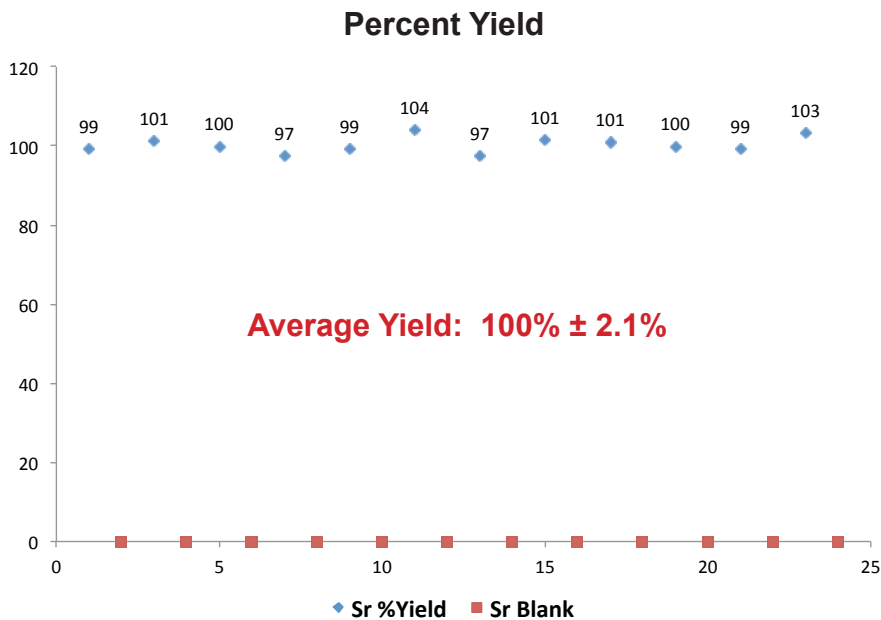
Percent Yield

Average Yield: 100% ± 2.1%

Figure 10. Strontium recovery for 24 consecutive, fully automated extractions with cleaning and reuse of the separation column. Constant high yields and no carryover (below detection) illustrate the ability to reuse the resin.
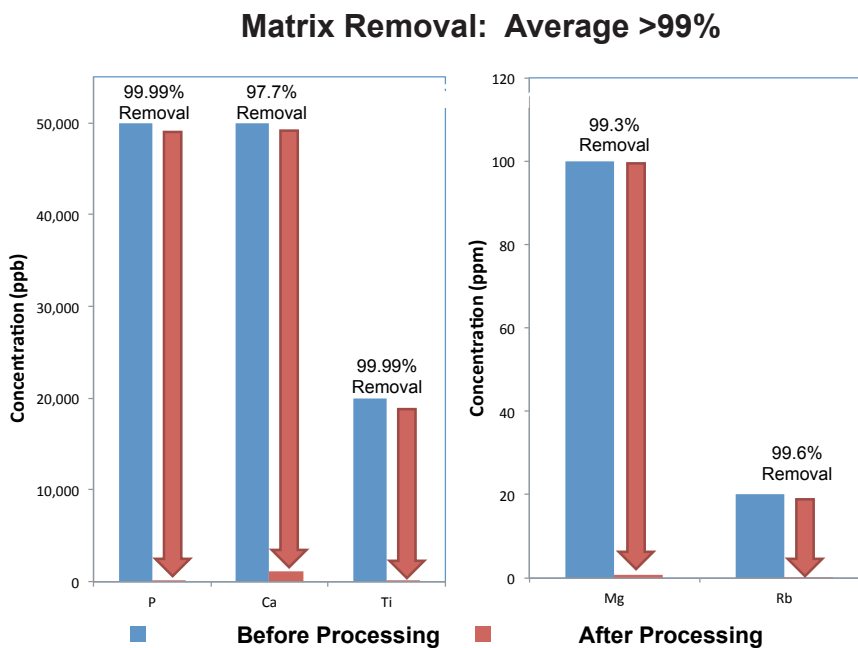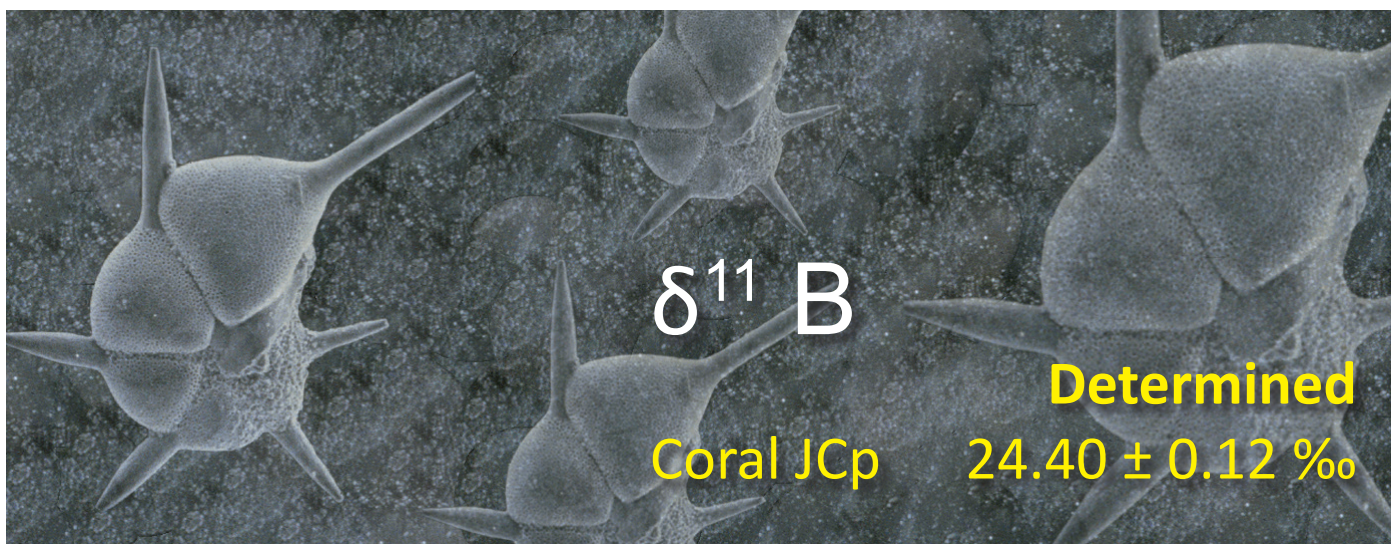
Matrix Removal: Average >99%

Figure 11. Online, real-time elution profile collected on the Element 2 (Thermo) illustrates the fully-automated chromatographic separation of Sr and Ca from matrix components. In carbonate samples it is possible to also collect the Mg fraction during sample load and wash cycles.

Boron Isotopes

$\delta^{11}B$

Coral JCp — **Determined** 24.40 ± 0.12 ‰

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The merits of a new prep*FAST* MC method for the purification of boron from carbonate samples are demonstrated. The fully automated syringe-driven system cleans the column ($0.5M$ HNO_3), conditions the column (DI H_2O), loads the sample ($0.5M$ HNO_3 buffered with Na acetate/acetic acid), elutes the matrix (DI H_2O) and elutes the sample ($0.5M$ HNO_3) from the column. User-defined reagent volumes and flow rates result in near complete removal of sample matrix (>99% of Ca) and the quantitative recovery of boron in ~600 µL (~100% ± 2.7%, n=10). Full automation, precise/accurate syringe control, identical flow path, and one column/resin bed all contribute to the reproducibility of extraction. The concept of resin reuse is validated by >30 replicate extractions with high yield and minimal carryover (<0.01%; below our detection). The prep*FAST* MC processes a sample in approximately 30 mins (48 samples per day) automating timely extraction techniques that will aid the advance of boron isotopes as a mainstream research tool.
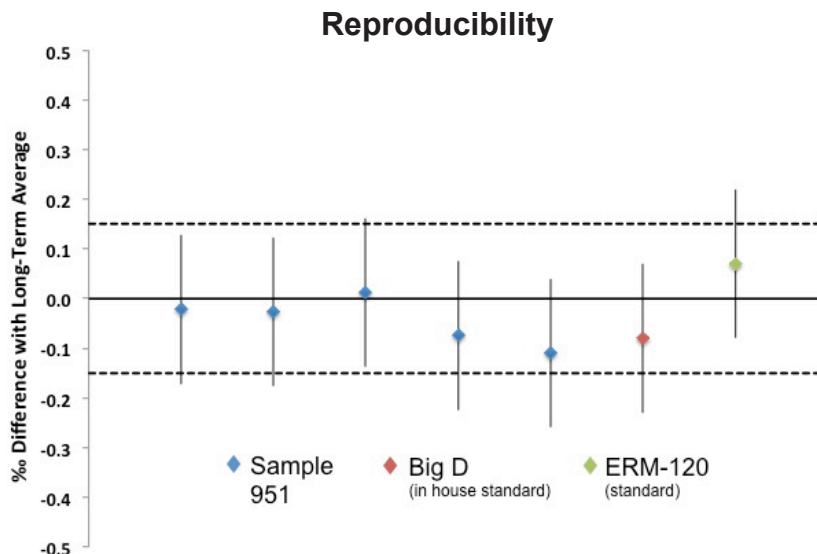
Figure 12. Online, real-time elution profile collected on the Element 2 (Thermo) illustrates the fully-automated chromatographic separation of Sr and Ca from matrix components. In carbonate samples it is possible to also collect the Mg fraction during sample load and wash cycles.

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Reproducibility

‰ Difference with Long-Term Average

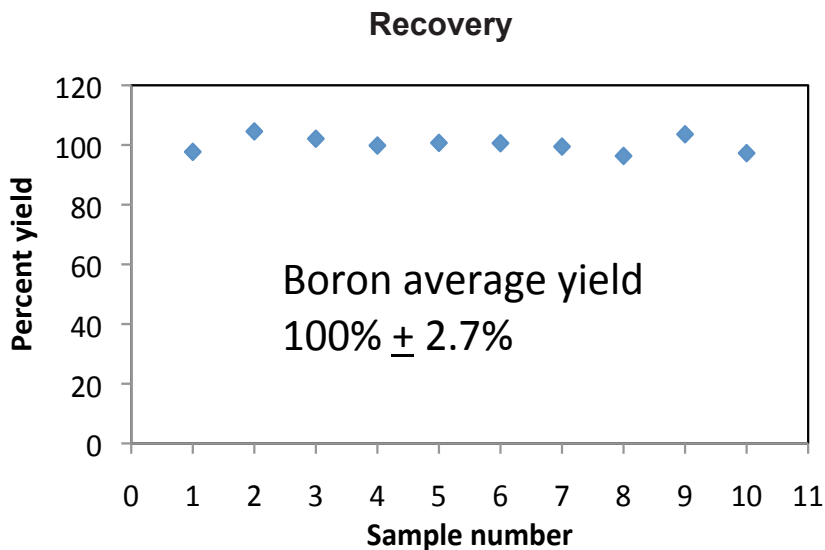♦ Sample 951   ♦ Big D (in house standard)   ♦ ERM-120 (standard)

Figure 13. Data collected from twenty automated extractions, alternating between sample and blank using the prep*FAST* MC boron method, are plotted. The data illustrate the ability to maintain both consistent and complete recovery of boron from multiple injections on one column.
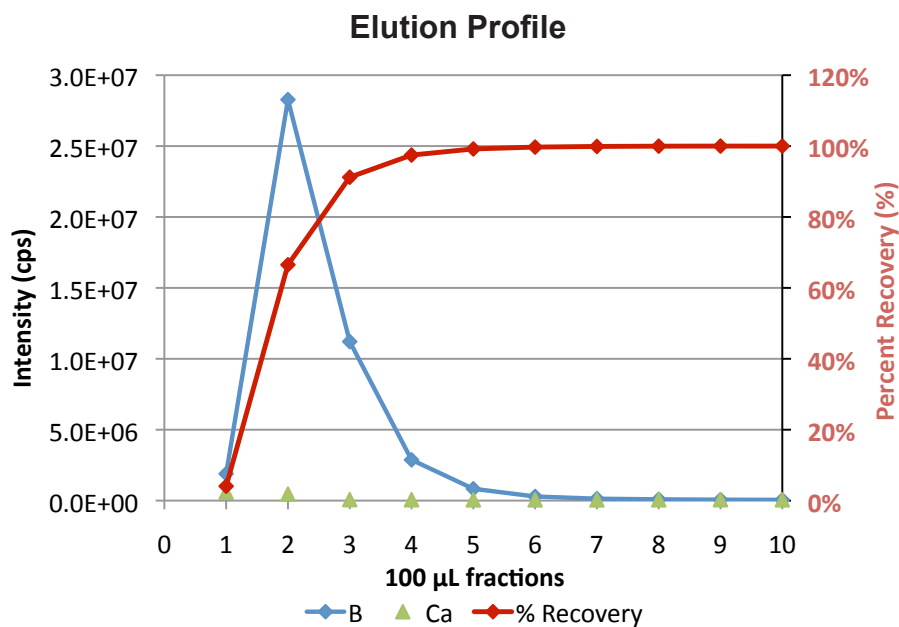

Figure 14. Ten 100 µL fractions collected from the prep*FAST* MC are used to generate an elution profile. Baseline calcium and high recovery (>99.9% in 600 µL) indicate sufficient matrix removal and quantitative boron elution. Quantitative recovery in a small final volume maintains boron concentrations at sufficient levels for isotopic analysis.
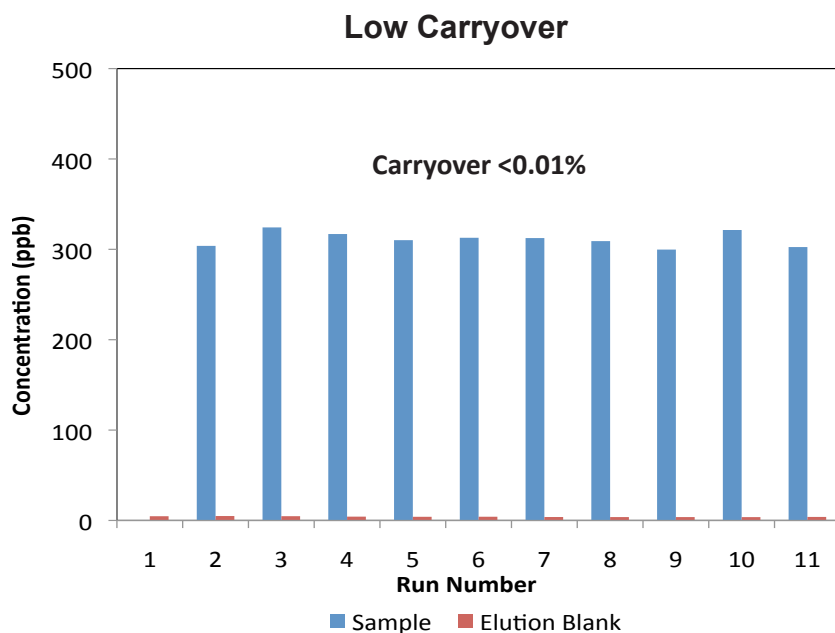
Boron Isotopes *(Continued)*

Low Carryover

Figure 15. Analysis of alternating blank – sample fractions from the prep*FAST* MC indicate boron values return to baseline concentrations for each blank. When baseline boron concentration is subtracted from each blank the data indicate greater than 4 orders of magnitude washout. Less than 0.01% carryover immediately after processing a 300 µg/L sample demonstrates no cross contamination from column reuse.

Benefits

- Fully automated
- User defined
  - Sample size
  - Elution volume
  - Wash/load/elute rates
- Multiple fractions
- High throughput (30-50 samples/day)
- Stand alone system runs 24 hrs unattended

prep*FAST* MC System