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Syringe Based Flow Injection MC-ICP-MS: High-Efficiency Sample Utilization with Increased Throughput

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Abstract

Sample-standard bracketing is a widely used standardization technique for the determination/comparison of precise isotope ratios by MC-ICP-MS, and for some non-traditional stable isotope systems, is the only method available. The frequency of standard bracketing increases with required precision and decreasing mass. This, combined with low sample introduction rates (typically < 100 µL/min), requires that a significant amount of time is needed for uptake and wash cycles. Sample throughput is therefore reduced.

A newly designed ESI micro*FAST* MC (Omaha, USA) precisely and accurately loads a loop and then smoothly injects solution to a µFlow concentric nebulizer at defined rates from 10-100 µL/min. The valve on the flow injection system selects from two discrete, parallel flow paths for standards and samples. This allows rapid switching between sample and standard solutions with minimal dead volume between the valve and the nebulizer.

Li isotope ratios were measured by a Thermo Scientific NEPTUNE Plus MC-ICP-MS (Bremen, Germany) using the micro*FAST* MC system. The following performance criteria were assessed: 1) internal precision, 2) external reproducibility of replicate measurements, 3) sample utilization (ions counted / atoms in solution consumed) and 4) sample throughput.

The micro*FAST* MC increases sample throughput, improving sample utilization for the lowest flow injection rates. External reproducibility is improved for the smallest absolute sample amounts using low volume solution aliquots.

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Experimental Conditions

- microFAST MC
  - 20 µl/min injection rate, ca. 63 µl per run
  - 0.5 ml sample aliquot per quintuplicate
- Thermo Scientific™ NEPTUNE Plus™ MC-ICP-MS
  - Jet Interface option (Jet & X cones)
  - 10^{12} ohm amplifier for ^{6}Li and 10^{11} ohm amplifier for ^{7}Li
  - 45 x 4.2-second integrations per analysis, ca. 3 minutes
  - Wet plasma
- 60 V/ppm Li sensitivity at 20 µl/min (ambient spray chamber)
- 100 V/ppm Li sensitivity at 20 µl/min (heated spray chamber)

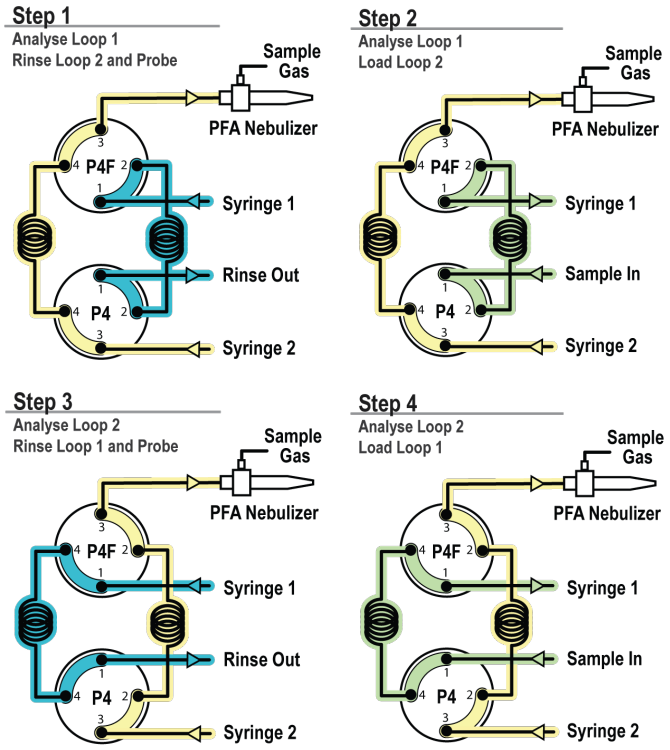

Figure 1. Flow paths illustrating the microFAST MC patented dual loop injection for rapid sample standard bracketing. Dual loop system allows one loop to be cleaned and loaded with sample while the other loop is injected and analyzed. Rapid switching between loops drastically reduces long uptake and wash times associated with low sample flow rate.

Total Sample Consumption

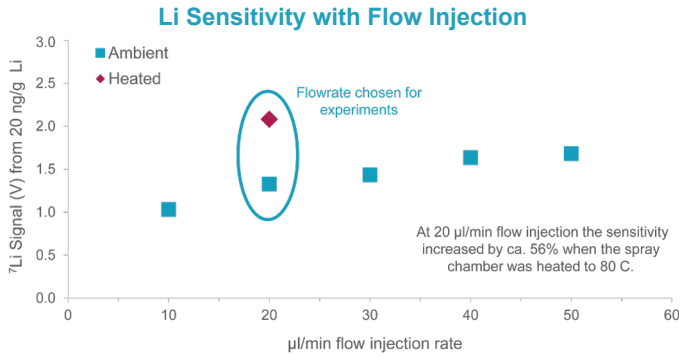

Figure 2. At ambient temperature, sensitivity gains with increased sample flow rate are minimal after 20 µL/min. The corresponding increase in sample waste stream would result in decreased absolute detection limits. The PC^{3x} allows heating of the spray chamber, which nearly doubles the sensitivity at 20 µL /min.

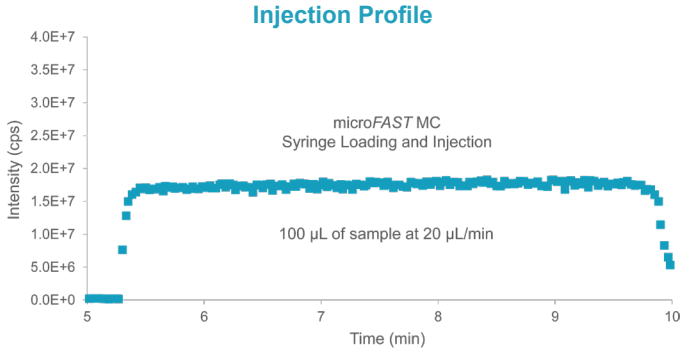

Figure 3. Injection profile of a 100 µL sample at 20 µL/min.

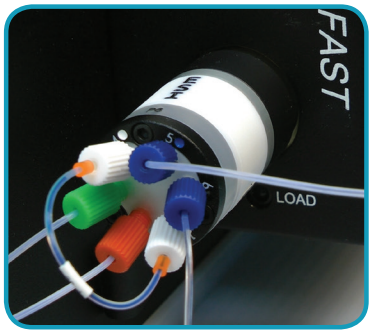

Figure 4. Accurate and precise loading of the sample loop with the exact volume required for analysis.

Reproducibility

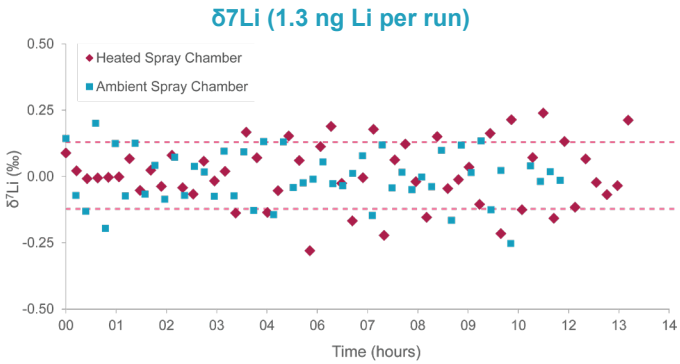

Figure 5. Sample standard bracketing over 12 hrs illustrating ±0.24‰ (2SD) precision for 1.3ng samples of Li

∂7Li | 1.3 ng Li | n>50		
	Heated	Ambient
Mean	0.01	-0.01
SD (‰)	0.12	0.12

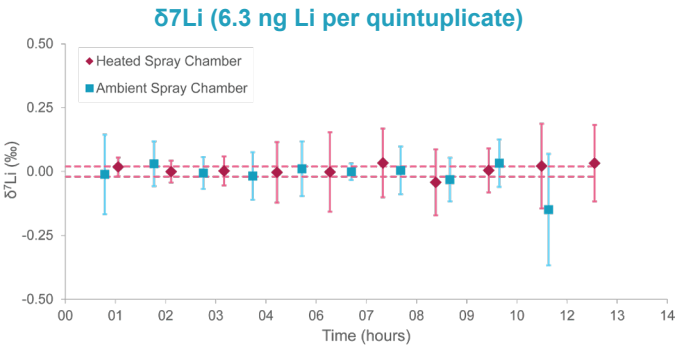

Figure 6. Quintuplicate data requires 6.3ng of Li and provides ± 0.04‰ (2SD) precision over 12 hrs

∂7Li (quintuplicate) | 6.3 ng Li | n=10		
	Heated	Ambient
Mean	0.01	-0.01
SD (‰)	0.02	0.05

Mass Bias

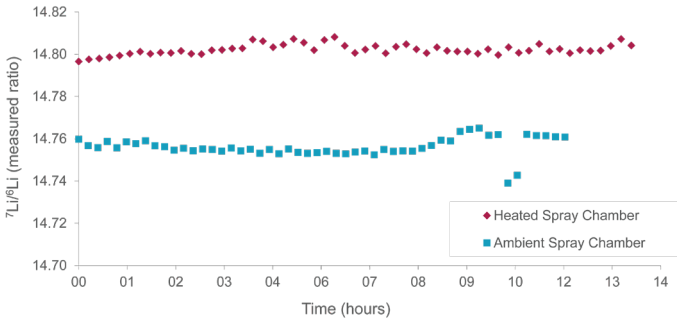

Figure 7. micro*FAST* MC combined with either ambient or heated PC^{3x} exhibits stable mass bias over >12 hrs.

Summary

- Reliably run small volume samples at micro flow rates
  - Syringe loading
  - Syringe injection
  - Set exact sample flow rate
- Dual loop rapid injection system
  - Reduce uptake and wash time
  - Improve time between sample standard bracketing
- Stable MC-ICP-MS
- Precision of ± 0.04‰ for 6.3 ng of Li.

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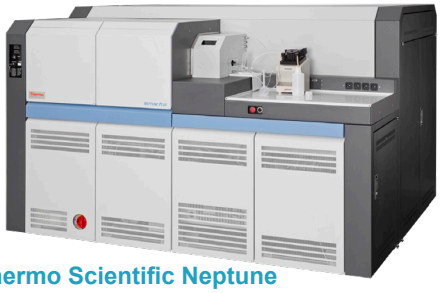
micro*FAST* MC

PC^{3x}



Thermo Scientific Neptune