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Savillex Technical Note

Performance of a PFA Cyclonic Spray Chamber with the Thermo ElementXR ICP-MS

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Summary

The Savillex PFA cyclonic spray chamber was evaluated for use with a Thermo ElementXR HR-ICP-MS for the measurement of multiple trace element/calcium ratios in foraminiferal calcite. Samples are run in an HNO3/HF matrix (Misra et al. 2014) which requires an inert, trace metal clean, sample introduction system. The signal stability of the cyclonic spray chamber was studied, along with its washout (particularly for boron which is a "sticky" element). The spray chamber was found to have good short and long term stability and fast washout (60 s for boron), making it well suited to the application.

Savillex PFA Cyclonic Spray Chamber on ElementXR

Application Requirements

Trace element/calcium ratio determinations in foraminiferal calcite are performed in the Cambridge laboratory using an ElementXR. Trace levels of Na, Mg, Sr, and ultra-trace levels of Li, B, Ba, Cd, Mn, U are measured in a calcium rich matrix and ratioed to calcium. Important requirements for this application are:

- A trace metal clean sample introduction system – essential for low blank determinations
- Good short term stability – necessary as the instrument cycles between elements during data acquisition
- Good long term reproducibility, since runs can take several hours
- Fast washout with minimum memory effect between samples

Sample introduction system performance has a major influence on all of the requirements above. Samples are run in 0.1M HNO3/ 0.3M HF to prevent boron carryover between samples, hence an inert sample introduction system is required. Due to poor wettability and design limitations in the manufacture of fluoropolymers such as PTFE and PFA, inert spray chambers generally have inferior performance to quartz spray chambers. We evaluated the recently introduced PFA cyclonic spray chamber from Savillex to see if advancements in technology have improved the performance of inert sample introduction systems for ICP-MS.

System Configuration

A Thermo ElementXR (Thermo, Bremen, Germany) was fitted with a demountable torch and 1.8 mm ID platinum injector, a PFA cyclonic spray chamber (no surface treatment) with baffle, PFA elbow connection with make up gas port, and a C100 PFA concentric nebulizer (PFA parts all from Savillex Corporation).

Method

An acid blank and three standards prepared in 0.1M HNO3/ 0.3M HF with concentrations reflecting the composition of foraminiferal calcite were used: concentrations of standards, Std 0, Std 4 and Std 8, are shown in *Table 1*. Trace element concentrations in Std 0 are from the high purity single element Ca solution used.

Element	Ca	Li	B	Mg	Al	Sr	Cd	Ba	U	Na	Mn
Units	ppm	ppt	ppb	ppb	ppb	ppb	ppt	ppb	ppt	ppb	ppb
Std 0	10	0.6	0.002	0.02	0.004	0.1	0.1	0.002	0.005	0.04	0.002
Std 4	10	13.6	0.26	10.3	0.57	24.5	4.1	0.16	2.8	26.8	1.3
Std 8	10	37.1	0.74	32.0	1.78	45.8	12.0	0.54	31.5	54.5	9.3

Table 1
Standard Concentrations.

The ElementXR operating parameters are shown in *Table 2*. Sensitivity was optimized using Std 4. After the initial data acquisition for all standards, the acid blank, Std 0 and Std 8 were measured 17 times each in sequence, to check for reproducibility, drift and washout of boron after aspiration of the most concentrated standard, Std 8, to the blank.

Parameter	Value	Parameter	Value
Sensitivity: 10 ppm Ca (Ca 43)	*25 Mcps*	*Sample consumption per analysis*	*~220 uL*
Sensitivity: 0.26 ppb B (B 11)	*95,000 cps*	*Number of repeat measurements*	*17*
Uptake time	*90 seconds*	*Total run time including initial calibration*	*5 hours*
Wash time	*60 seconds*	*Drift correction*	*not used*

Table 2
Operating and Method Parameters.

Results

The cyclonic spray chamber was found to have good short term stability with RSDs typically in the range of 0.5-1% in Std 8. Longer term reproducibility was also good with RSDs for 17 replicates of Std 8 over a 4 hour period following the initial calibration in the range 1.5 – 2.9% for Li7, B11, Mg25, Al27, Ca43, Sr87, Cd111 and Ba137 measured in low resolution. The plot (figure 1) below shows the calcium signal in Std 8 (measured in low resolution at m/z 43). RSD of the Ca43 signal was 2.02%. No drift correction was applied.

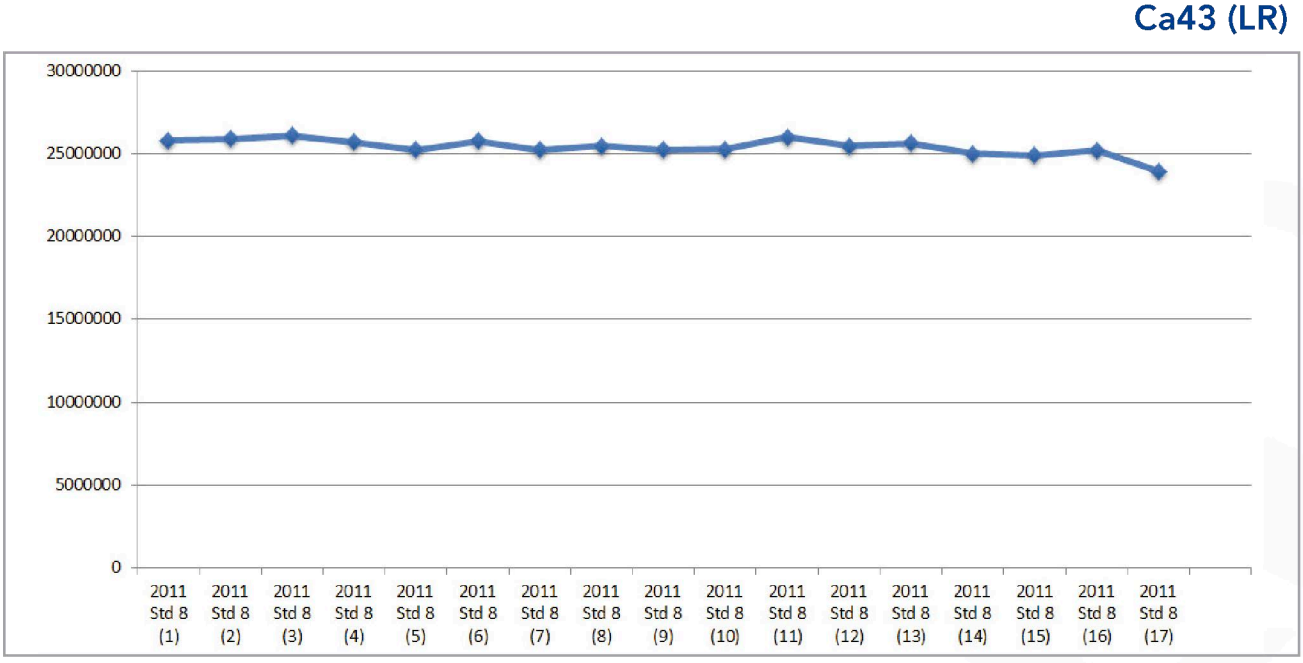

Figure 1
Calcium (10 ppm) Signal in Std 8 Over 4 Hours.

To evaluate washout and potential carryover of boron, which is one of the more challenging elements with respect to washout, the B11 signal (low resolution) was plotted for the blank and all standards – see figure 2 below. Washout from Std 8 (0.74 ppb B in a 10 ppm Ca matrix) to blank levels was achieved within a 60 sec. wash time. Point number 5 is Std 4. Following this, the blank, Std 0 and Std 8 were measured 17 times in sequence. Std 0, containing 0.002 ppb B (2 ppt) can clearly be measured above the blank, demonstrating the excellent sensitivity of the system fitted with the PFA cyclonic spray chamber.

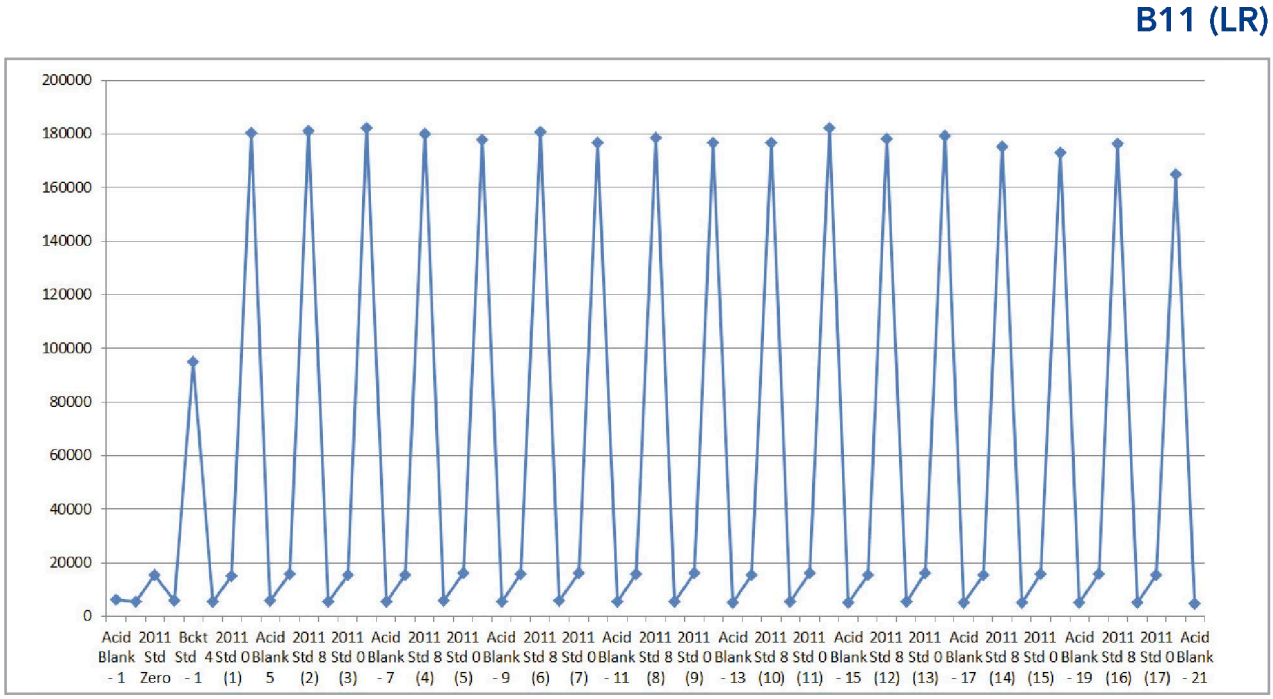

Figure 2
Boron signal over 5 hours – blank and all standards shown.

Sensitivity of the system can be more clearly seen by excluding the higher concentration standards. Figure 3 shows data from *Figure 2* for the acid blank and Std 0 only. Reproducibility for Std 0, containing only 2 ppt boron, is excellent, and the boron signal returns to the blank level, indicated by acid blanks 1 and 2, each time after aspirating Std 8 (not shown).

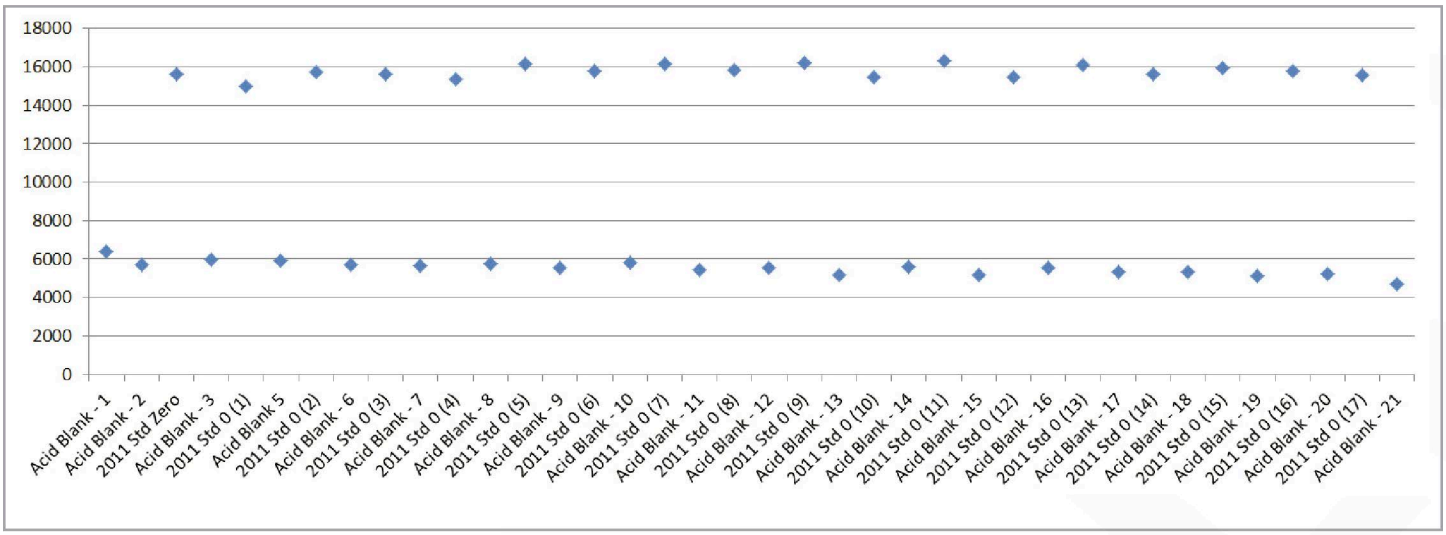

Figure 3
Boron signal over 5 hours – blank and Std 0 (2 ppt B). Std 4 and Std 8 points have been excluded.

Summary

The Savillex PFA cyclonic spray chamber combines high sensitivity with good stability and reproducibility, making it well suited to this application. Fast washout for difficult elements like boron also reduced overall run time and improved data quality.

References

1. Misra, S., M. Greaves, R. Owen, J. Kerr, A. C. Elmore, and H. Elderfield (2014), Determination of B/Ca of natural carbonates by HR-ICP-MS, Geochem. Geophys. Geosyst., 15, 1617–1628, doi:10.1002/2013GC005049.

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