A Technique for Improving Long-Term Stability in High Matrix Sample Analysis by ICP-MS

Bill Spence[1], Simon Nelms[2], Michael Sgroi[3], Dhinesh Asogan[4]
[1] CETAC Technologies, 17 Clearwater Drive, Manchester, UK. [2] Thermo Fisher Scientific, Stafford House, Boundary Way, Hemel Hempstead, UK.
[3] CETAC Technologies, 14306 Industrial Road, Omaha, NE, USA. [4] CETAC Technologies, 9 Alderman Walk, Stanford-le-Hope, UK

14306 Industrial Rd.
Omaha, NE 68144
USA
PHONE 402.733.2829
FAX 402.733.5292
WWW CETAC.com

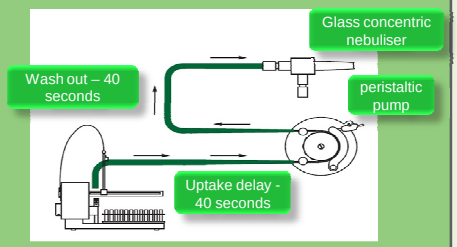
Introduction

High concentrations of dissolved matrix components pose a challenge to ICP-MS analyses, as their introduction over long periods leads to the deposition of matrix-based material around the interface apertures. This process causes changes in the shape and size of the ion beam sampled from the plasma which, in turn, causes changes in the relative sensitivity achieved with time. This characteristic makes successful, accurate long-term analyses of matrices such as sea water and clinical samples challenging, time consuming (if they are to be analysed in small batches with an increased cleaning schedule) and costly (due to the decreased life expectancy of cones, increased analysis time and the previously mentioned increased maintenance schedules).

Recent innovations in sample introduction techniques for the improvement of long-term stability are based on reducing the exposure of cones to high levels of matrix. Traditional sample introduction options (figure to right) often expose the instrument to high levels of matrix for longer than necessary as they pump sample through a long length of uptake tube. The ***ASXPRESS™ PLUS*** rapid sample introduction system dramatically reduces exposure by introducing the sample for the absolute minimum amount of time.

A comparative study was undertaken of a standard sample introduction set up (Experiment 1), and the ***ASXPRESS™ PLUS*** vacuum pump rapid sample introduction system (Experiment 2) for the analysis seawater and urine.

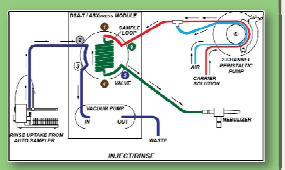
TRADITIONAL SAMPLE INTRODUCTION
(As per experiment 1)

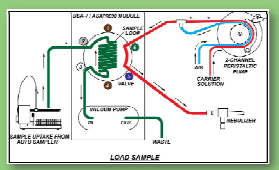


ASXPRESS™ PLUS Principle of Operation

Load Mode:
Simultaneous actions performed –
1. Sample rapidly loaded onto Teflon loop.
2. Sample introduction system rinsed with segmented air/carrier solution.

Then –
- Valve position switched to Inject Mode.





Inject Mode:
Simultaneous actions performed –
1. Sample pushed off loop into nebuliser by carrier stream from peristaltic pump. (Measurement performed).
2. Autosampler probe and uptake line rinsed by air/rinse solution.

Experimental Details

Autosampler: CETAC ASX-520 (with ***ASXPRESS™ PLUS*** for Experiment 2)
ICP-MS: Thermo Scientific X Series 2
Sample uptake rate: 800 uL/min

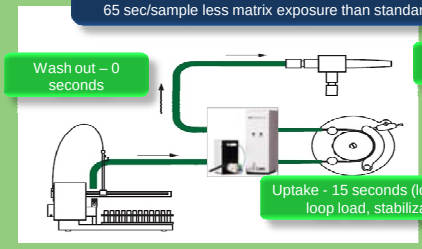
Nebuliser: glass concentric
Spray chamber: glass impact bead
Torch: one-piece quartz, 1.5 mm injector
Cones: Xt

Nebuliser gas: 0.91 L/min
Coolant gas: 13 L/min
Auxiliary gas: 0.7 L/min
RF Forward power: 1400 W

Standard optimization for best sensitivity (CeO/Ce = 0.015) –
no steps taken to improve matrix tolerance or drift resistance

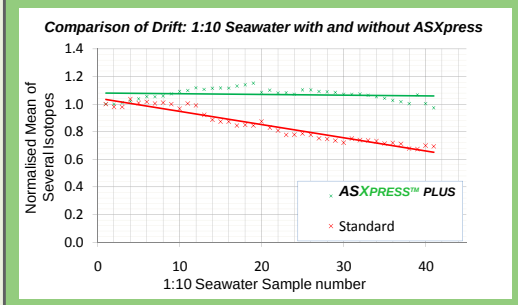
RAPID SAMPLE INTRODUCTION
(As per experiment 2)

Uptake time saving – 25 seconds
Wash out time saving – 40 seconds
65 sec/sample less matrix exposure than standard



Results

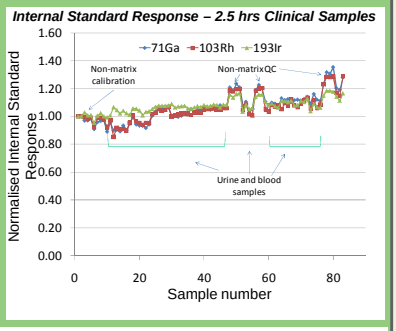
Comparative study of seawater analysis and the effect on drift

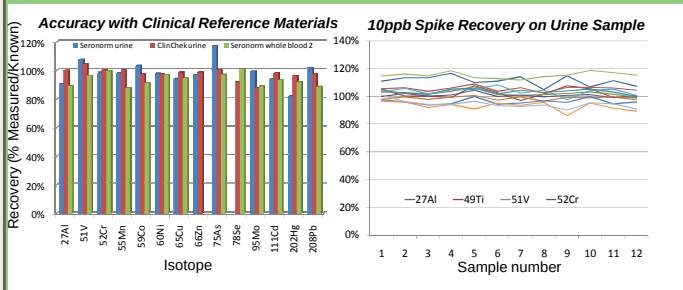


- 1:10 diluted seawater (+ trace nitric acid) + 5ppb Be, Co, In, Ce, Pb, Bi and U
- A calibration was performed, then 5ppb seawater solution was analysed as a sample 40 times
- Measurement time = 2 min/sample

Precision and accuracy of Clinical CRM sample analyses with ***ASXPRESS™ PLUS***

- Run of 80 mixed urine and whole blood samples at 1:20 dilution
- Diluent: 1% methanol, 0.5% HNO_3
- Internal standards: ^{71}Ga, ^{103}Rh, ^{193}Ir, at 50 ppb
- ICP-MS operated in helium KED collision cell mode
- ***ASXPRESS™ PLUS*** used to reduce matrix exposure, demonstrating freedom from drift, and good accuracy and precision





Conclusions

- The ***ASXPRESS™ PLUS*** reduced the non-productive matrix exposure of cones by over 60%.
- The instrumental drift associated with high matrix samples was significantly decreased, allowing 4 times as many samples to be analysed within a single batch.
- Long-term precision and accuracy in high matrix runs can be significantly improved.
- Additional benefits: Cost Savings – analyses time decreased, increased longevity of cones, lower argon and electricity consumption, less instrument wear.

Further information: www.cetac.com/automation/asxpress