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Application Note

Analysis of Small Sample Volume Geochemistry Samples by Array Detector ICP-MS

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INTRODUCTION & OBJECTIVES

The British Geological Survey analyses thousands of samples every year and an increasing number of specialist applications require sample consumption of less than 1 mL. This application note examines the use of the Teledyne CETAC MVX-7100 µL Workstation to analyse shellfish microchemistry.

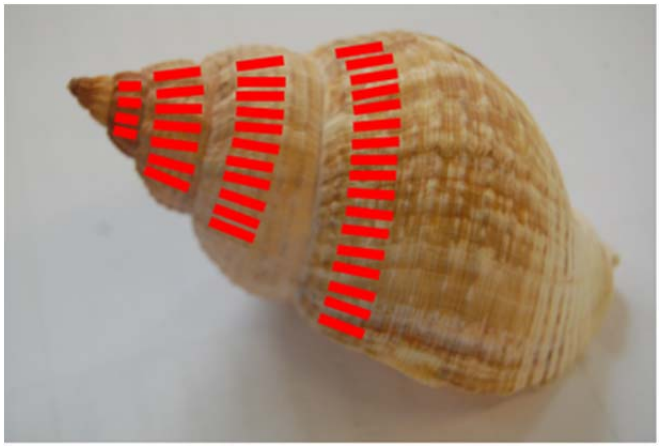

Figure 1: Example of whelk shell with sampling bands marked

The size and growth rates of whelk shells can be bio-indicators for a variety of scientific study from water quality to global warming. These shells as they grow will displace elements within the calcium carbonate matrix with elements in their environment. This exchange is the basis for developing hypotheses to answer the greater questions at hand. The challenge however is that the effective area sampled is very small and the amount of total solid sample that is drilled out is on the order of 100 micrograms. New methods and new technologies are required to attempt to answer these questions.

MATERIALS & METHODS

The MVX-7100 utilizes a metal-free flow injection sample pathway to deliver volumes as low as 5 µL. A septum piercing quartz probe allows samples to be capped and protected from evaporation and contamination. The system features a high precision x, y, z autosampler, syringe driven sampling and injection, and a modular design for right and left handed ICP-MS systems.

Figure 2: MVX-7100 µL workstation

Method development was carried out using standards prepared in a 1%HNO3 matrix. The MVX-7100 was used with the Teledyne CETAC Aridus II desolvating nebulizer in order to achieve sufficient signal enhancement.

Optimized sample injection inserted an air slug on either side of the sample slug in the flow injection loop. This method of injection gave the best results and also did not waste any sample – a very important requirement. The samples to be analysed were micro-drillings (100 µg) of whelks. The sample is drilled from the shell in sequential shell bands and the resulting material is digested for ICP-MS analysis.

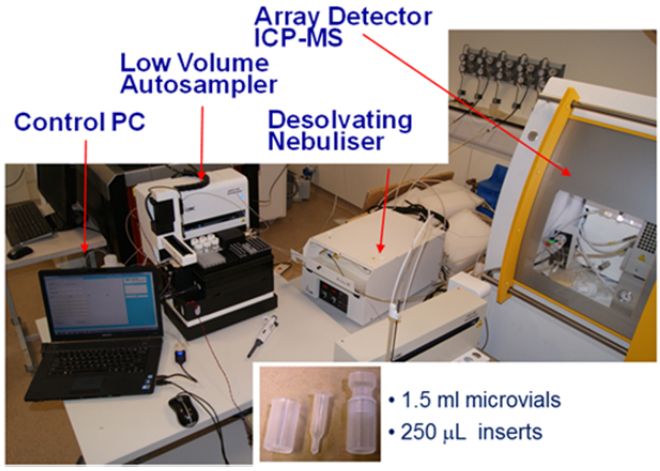

Figure 3: MVX-7100 on Array Detector ICP-MS

The method showed reproducibility of 1 - 3% RSDs with internal standardization. Figure 4 displays a series of transient signals from 10 separate injections.

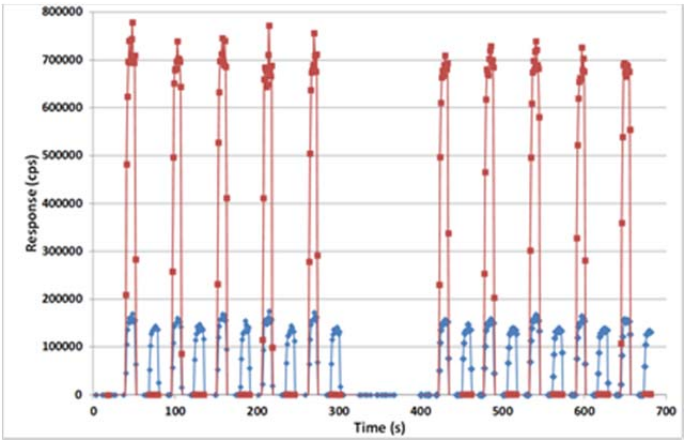

Figure 4: Repeated 200 µL injection from a 250 µL loop

The method development also compared rinse solution, segmented rise, and air. Figure 5 displays the comparison of the three rinses, with the full rinse performing the best.

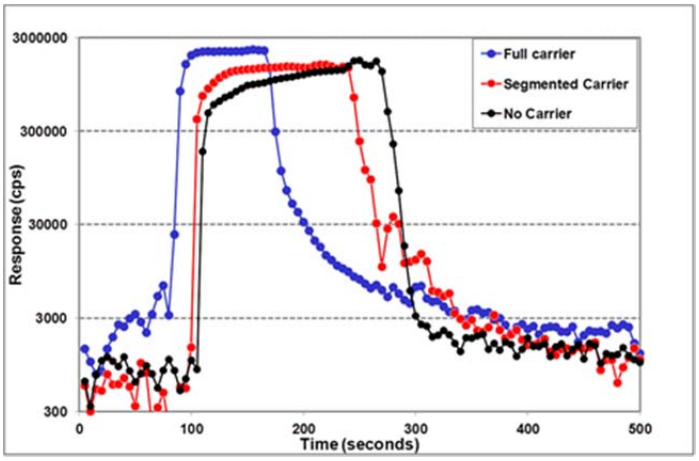

Figure 5: Comparison of three carrier streams

RESULTS & DISCUSSION

The results from the welk study indicate that unexpected elements of Fe, Mn, and U have significant concentration. Additionally these elements appear to change on an annual cycle and increase with age (figure 6).

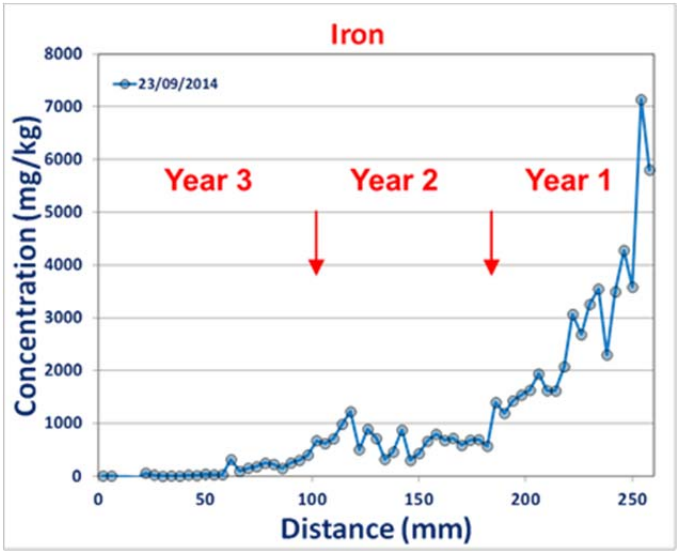

Figure 6: Results of micro-drillings from welk sample

CONCLUSIONS

The Teledyne CETAC MVX-7100 mL Workstation is designed to precisely sample a specific aliquot and deliver the entire aliquot to the ICP-MS. This capability was able to deliver results which may not have been possible without a high precision aliquot workstation.