A Low Volume Injection Autosampler For ICP-MS

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Introduction

The requirement to sample and inject decreasingly low liquid volumes is increasingly prevalent in scientific analysis and particularly in ICP-MS. Several fields exists where sample size is such that traditional sample introduction is not possible. Problems can arise during dilution, especially when analyte concentrations begin at trace levels. Biological cells, dust particles, atmospheric condensates are just a few examples of sample types that illustrate the need for an ICP-MS autosampler that can precisely sample and inject microlitre and nanolitre sized aliquots. In this study we present such an autosampler. The autosampler system is designed akin to the type of system that operates with a High Performance Liquid Chromatography (HPLC) system. The difference however is that each of the wetted parts are manufactured of non-metallic materials in order to be applicable to ICP-MS. Additionally, this system takes an aliquot and injects the entire sample into a sample loop, therefore no sample is ever wasted, such as is the case with a sample uptake line.

System Description

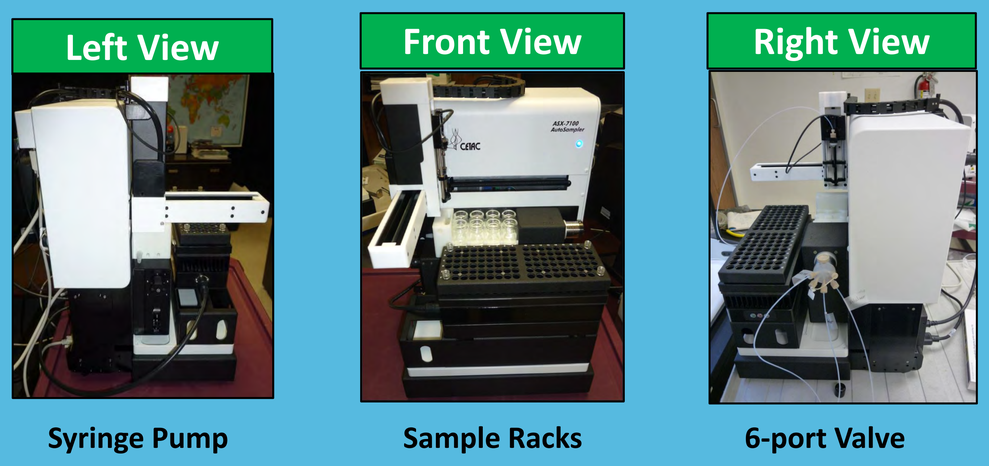

Figure 1: Front, right, and left view of low volume autosampler.

Experimental Setup

The ICP-MS used for this study was a Thermo iCAP Q ICP-MS. The autosampler was interfaced to the front of the instrument using the standard nebuliser and spray chamber arrangement. The peristaltic pump on the ICP-MS was used for the carrier solution, as well as to drain the spray chamber waste. A diaphragm pump on the autosampler delivered rinse solution to the dual rinse stations used to rinse the probe. Additionally, the probe was rinsed by use of the syringe pump to drive rinse solution through the inside of the probe, in this way the probe was washed on the inside and outside.

Figure 2: Thermo iCAP Q with low volume autosampler installed.

Results

The results shown here represent extremely preliminary work. The calibration curves for both the full loop injection and the partial loop injection could be improved. Optimization of both the sample aliquoting and the sample injection are continuing, along with additional figures of merit. There are several ways to inject a sample; filling the loop completely or partially. The sample can be sandwiched between air and carrier solution. In our tests the partial loop fill with air on either side of the sample gave a much sharper peak for integration.

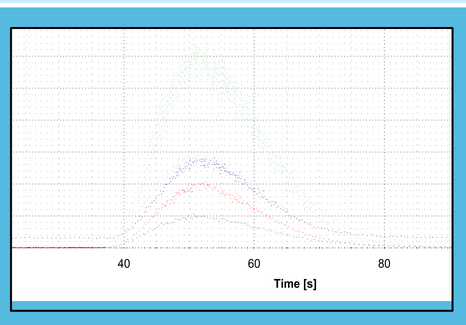

Figure 3 Full loop injection, 50 µL loop, 25 ppb

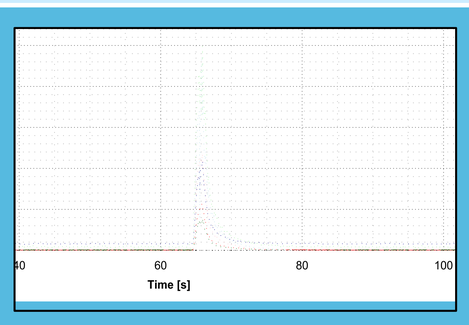

Figure 4 Partial loop injection sandwiched with air, 10 µL, 25 ppb

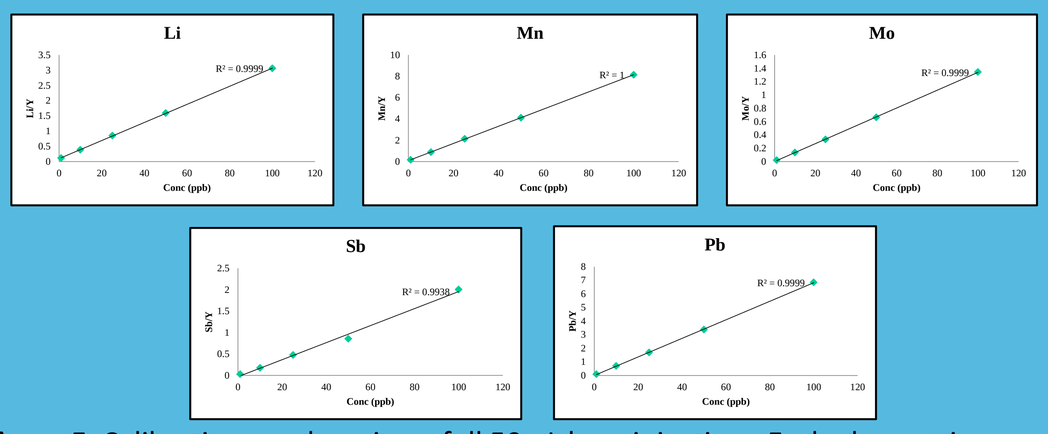

Figure 5 Calibration graphs using a full 50 µL loop injection. Each element is normalised to the internal standard (Y).

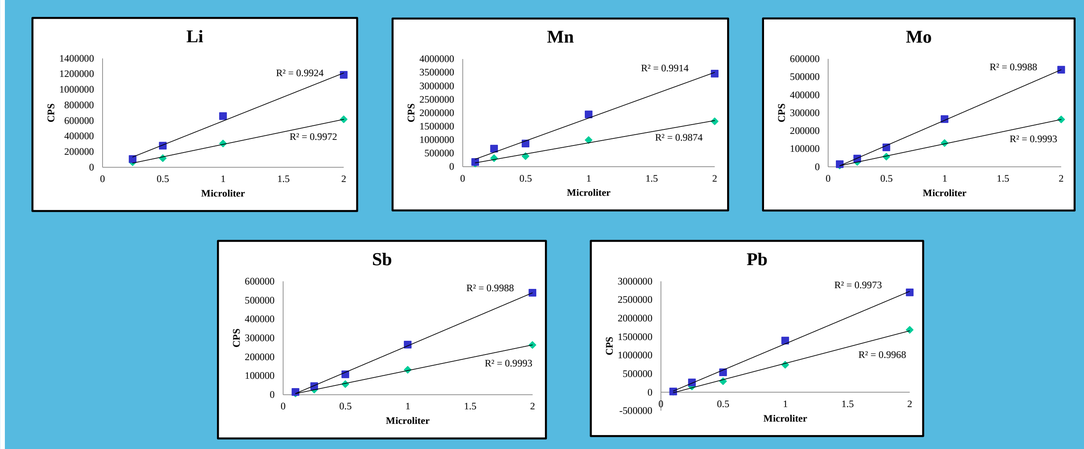

Figure 6 Calibration graphs using partial loop injection (of a 50 µL loop). Peak area (blue) peak height (green).

Future Work

Adding a syringe pump to drive the carrier solution allows for additional capability. Using a low pump rate a total consumption nebuliser such as the DS-5 can be used. Figure 7 shows the autosampler with an additional syringe pump as well as the DS-5 hooked up to the iCAP Q. Figure 8 shows a sample run using the DS-5.

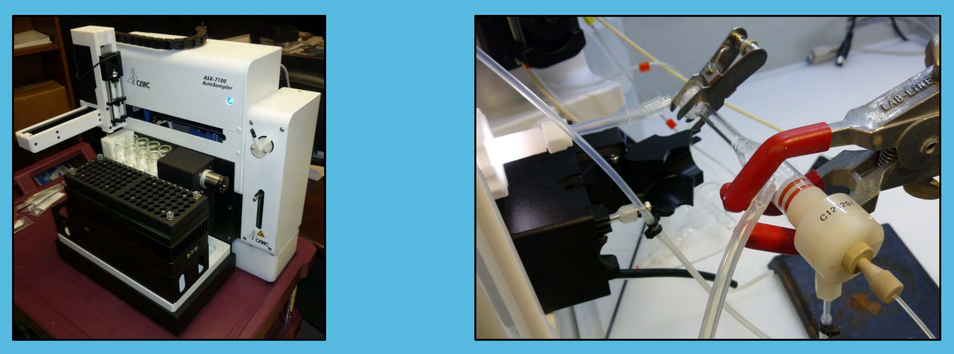

Figure 7 (left photo) Low volume autosampler with additional syringe pump. (right photo) DS-5 nebulizer attached to Thermo iCAP Q ICP-MS.

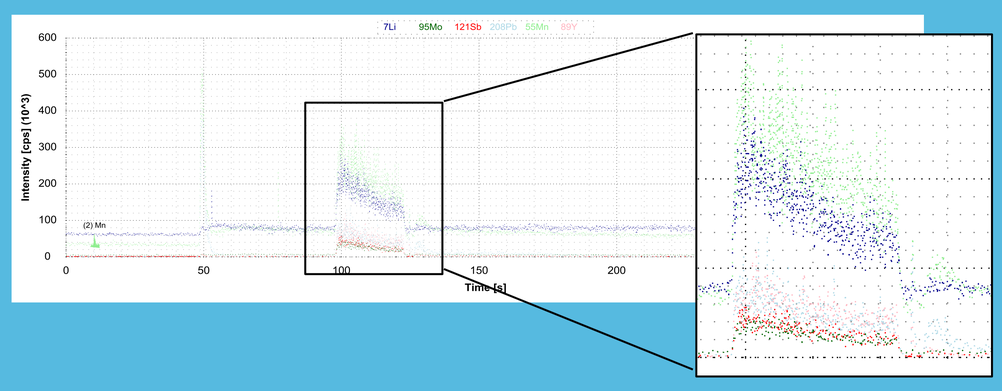

Figure 8 Time resolved analysis plot showing a 10 µL injection using a DS-5 total consumption nebuliser, the flow rate in this example was 50 µL/min.