Poster Note 43312

Dilute or Die – How to Handle High Matrix Samples by ICP-MS

Marcus Manecki, Daniel Kutscher, Shona McSheehy Ducos
Thermo Fisher Scientific, Bremen, Germany

Overview

Purpose: To compare different dilution techniques and their advantages and disadvantages. To demonstrate robust high throughput analysis of environmental high matrix samples using Q-ICP-MS in He-KED mode with different dilution techniques.

Methods: Thermo Scientific™ iCAP™ Qc ICP-MS coupled to the ESI prep*FAST*™ auto-dilution system. Argon Gas Dilution (AGD) combined with direct online dilution.

Results: Internal Standard and analyte recoveries of 80-120% were achieved for different high matrix sample sets with different matrix dilution techniques (auto-dilution or gas dilution) for long term measurements.

Introduction

Dealing with high matrix samples in analytical measurements often means complicated analyte enrichment or matrix removal (e.g. reverse osmosis) techniques have to be used. Disadvantages of those methods are that they are expensive, time consuming and increase the risk of sample contamination. Dilution of the samples often leads to much better results as long as the analytes of interest are not diluted below the limit of detection of the analyzing instrument. However, skilled technicians are often required to setup and prepare the day's analysis as well as actively monitor the results and perform further sample manipulation as required throughout the analytical run.

Therefore, automated dilution approaches including Argon Gas Dilution (AGD) and intelligent dilution with the ESI prep*FAST* ™ auto-dilution system were evaluated as methods for handling higher matrix and overcoming some of the issues of manual sample preparation. We show the limitation of each dilution technique and present data which shows that the iCAP Qc ICP-MS and the ESI prep*FAST* auto-dilution system can run an entire analysis (>100 samples) against EPA 200.8 (encompassing preparation, calibration and measurement) with minimal manual intervention.

Methods

Analysis of environmental samples were performed to describe the capabilities of the following dilution techniques:

Argon Gas Dilution

Samples are diluted through a combination of a reduced nebulizer flow and an additional argon flow added to the spray chamber (Fig. 1). To set up the AGD mode, a dedicated tuning procedure was used to tune the system with the additional Ar gas flow to achieve optimal sensitivity for all analytes.

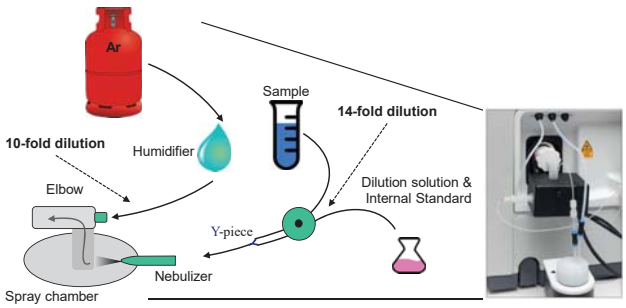

FIGURE 1 . Concept of an Argon Gas dilution approach coupled to an online dilution.

With the AGD approach a 25% NaCl solution spiked with 25 ppb of different analytes was measured over 4 h in He-KED mode. The recovery and internal standards were determined and the overall stability (RSD) of the system was calculated.

Thermo SCIENTIFIC

prep*FAST* auto-dilution

A fully automated system (prep*FAST* system, Elemental Scientific Inc.) was prepped with the necessary solutions (diluent, internal standard, rinse solution). The whole dilution process for all sample types is controlled by the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) software.

For the first dataset a long term measurement of >100 samples against the EPA 200.8 (Rev. 5.5) method for drinking water was performed. The dataset contains standards, samples and quality controls required by EPA 200.8.

In the second experiment semi saline waste water (up to 8% NaCl) was analyzed directly with the prep*FAST* system. The Internal Standard (IS) recovery limits of between 85 to 120 % were defined by the method. All samples were run with a 40-fold prescriptive dilution (PD) and if a sample was not in the defined range, an automated 10-fold auto-dilution (AD) of the sample was triggered.

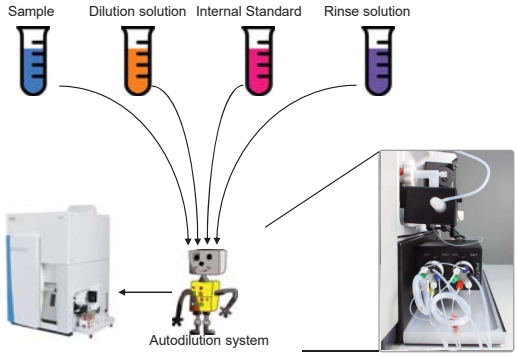

FIGURE 2. Concept of a fully automated dilution system, the prep*FAST* attached to the iCAP Qc ICP-MS.

Results

Argon gas dilution

AGD (10-fold dilution) was used in combination with a 14-fold online dilution. A 4 hour run with 23 elements was analyzed in a single He-KED mode. The analyte recovery over the whole run was between 80 and 120% for all analytes. After a short stabilization time the recovery of the internal standard varied between 80 and 120 % (Fig. 3) showing that it is possible to reliably analyze 25% NaCl with the iCAP Q ICP-MS equipped with AGD.

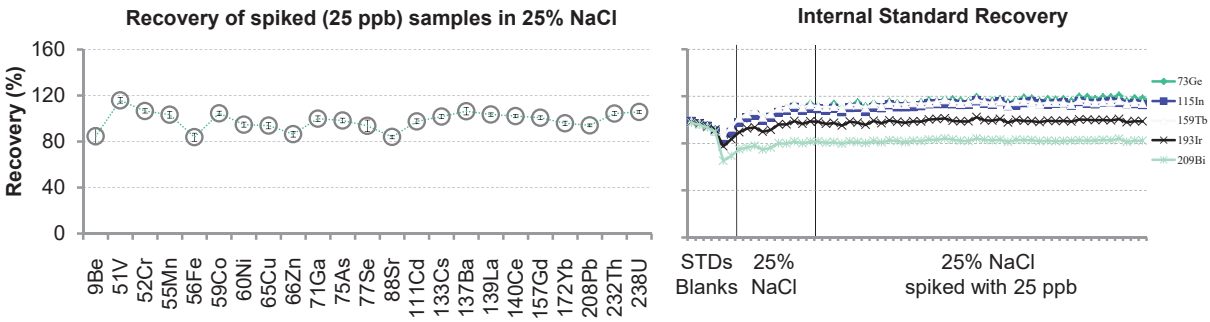

FIGURE 3. Analyte (left) and Internal Standard (right) recovery from a 4 h run with 25% NaCl. Black bars (left graph) show the RSD (N=24).

prep*FAST* and routine performance

Over 100 Samples were analyzed against EPA 200.8. The throughput in single measurement mode was 68s per sample. Concentrations of all analytes and internal standard recovery were measured over a period of 3 h (Fig. 4). All analytes remained within the recovery range of 90 to 110% which is required by EPA 200.8.

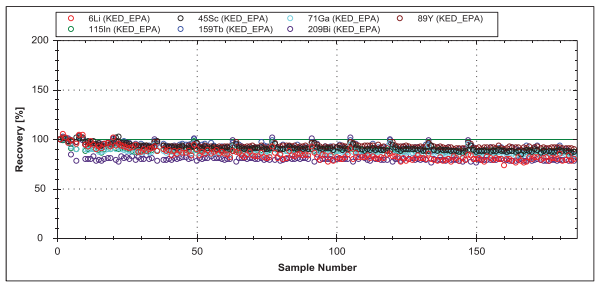

FIGURE 4. Screenshot of Qtegra ISDS software. Internal standard response of running tap water and QC samples. The recoveries are well within the 60 – 125% range specified in Method 200.8.

prep*FAST* auto-dilution for varying matrix samples

Different semi saline samples were measured directly with a PD of 40 (Fig. 5). Sample 24 triggered the auto-dilution process in the Qtegra ISDS software because the internal standard recovery was lower than the limits (85-120%) set in the method.
The first auto-dilution step increased the original PD factor by 10 and the sample was reanalyzed after a (40+10) 50-fold dilution. The internal standard recovery was still not in range and another two measurements were triggered. After the 3rd automated dilution the internal standard recovery was within the range of 80 to 120 percent and the system continued on to the next sample analysis without any manual intervention. Qtegra ISDS software provided all required features needed for this high throughput analysis of environmental samples.

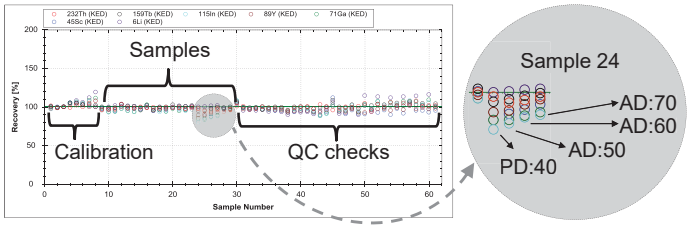

FIGURE 5. Screenshot of Qtegra ISDS software. Internal standard response of running semi saline water and QC samples (left). Automatically triggered auto dilution of sample 24 is shown in the right circle.

Conclusion

The iCAP Qc ICP-MS equipped with an ESI Autosampler and prep*FAST* auto-dilution system or the AGD module was tested for the analysis of different high matrix samples. Both systems delivered very reproducible data and showed a good overall performance.

- **High Throughput:** Both systems are ideal for measuring environmental samples in a high throughput laboratory.
- **Ease of Use:** AGD mode is simple to set-up and operate; a default tuning mode guarantees the reliable optimization. Auto-dilution settings are easily programmed into the workflow and provide the basis for prescriptive and intelligent dilution. Manual sample preparation and data post processing is significantly reduced when using auto-dilution, saving valuable time for skilled technicians to work on other lab tasks.
- **Flexibility:** With AGD, dilution of every sample is performed at a fixed level, but the dilution factor cannot be modified individually for single samples. The prep*FAST* auto-dilution system offers variable PD and is a solution which eliminates the need for manual intervention, even if samples fall beyond the analytical range or the internal standard pre-defined ranges. Auto-dilution is the ideal solution for handling high and variable matrix samples in high throughput, routine laboratories.