

Fully Automated, Intelligent, High-Throughput Elemental Analysis of Drinking Waters Using SQ-ICP-MS

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Key Words

Auto-Dilution, Drinking Water, ICP-MS, SQ-ICP-MS, He KED, Sample Preparation, U.S. EPA Method 200.8 Revision 5.5

Goal

To demonstrate robust high-throughput analysis of environmental samples using SQ-ICP-MS in He-KED mode, in accordance with the requirements of U.S. EPA method 200.8 Revision 5.5 and to demonstrate the performance of the Thermo Scientific™ iCAP™ RQ ICP-MS coupled to the ESI prepFAST auto-dilution system.

Introduction

EPA Method 200.8 analyses for the quantification of trace metals in drinking and waste waters are performed routinely in many laboratories. Thousands of analyses are performed per week to support the monitoring and control of drinking water contaminants and water quality. Due to the complexity of the standard operating procedure (SOP), skilled technicians are required to setup and prepare the daily analysis, as well as actively monitor the results and perform further sample manipulation as required throughout the analytical run. The need for technical staff is a factor that keeps the overall expense of routinely running the 200.8 method relatively high.

Recent advances in auto-dilution offer the potential to automate much of the sample preparation and data review with automated re-runs of any samples that do not meet predefined limits. By automatically creating a calibration set of standards from one stock standard and then diluting each sample to a predefined dilution level, an auto-dilution system can save valuable analysts' time and reduce costs overall, through the lowered consumption of utilities and lab supplies.

Fast sample throughput is another driving factor when implementing routine SOPs. Throughput in the method described herein is improved by the discrete sampling of the auto-dilution system, dramatically reducing uptake and washout time, as well as the use of a single measurement mode for the analysis of all the analytes in the method.



The use of kinetic energy discrimination with helium as a reaction cell gas (He KED) ensures comprehensive interference removal and confidence in the accuracy of the analytical results. Whereas other single quadrupole (SQ) ICP-MS systems require multiple methods for the analysis of drinking water, the iCAP RQ ICP-MS collision/reaction cell (QCell) has a high ion transmission across the mass range, so that all of the analytes in the method, including low mass analytes such as Li and Be can be measured in He KED mode. This eliminates the extra overheads of switching times between different modes and simplifies method development.

This application note describes the fully automated, intelligent, high throughput EPA 200.8 analysis of environmental samples using the ESI prepFAST™ auto-dilution system integrated with the iCAP RQ ICP-MS.

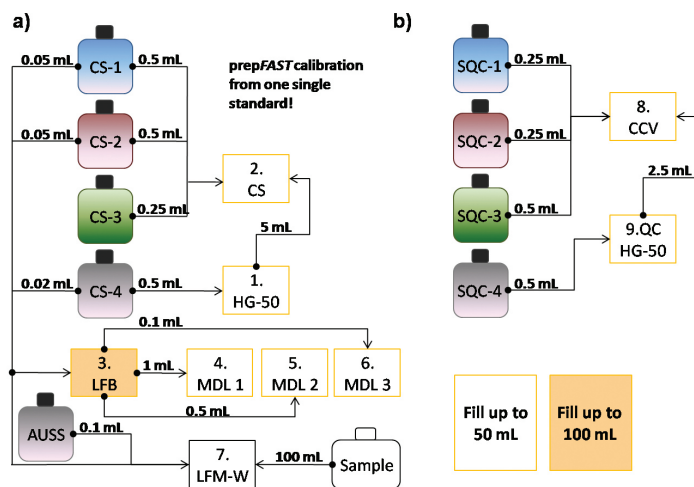


Figure 1. Scheme of (a) standard and (b) QC solutions required for EPA 200.8.

AUSS: Gold Standard Solution, CCV: Continuous Calibration Verification, CS-1 to 4: Calibration Standards, HG-50: Mercury Standard (50 ppb), LFB: Laboratory Fortified Blank, LFM-W: Laboratory Fortified Matrix, MDL-1 to 3: Solutions to determine Method Detection Limit, SQC-1 to 4: Standards for Quality control.

Table 1. Instrument conditions.

Parameter	Value
iCAP RQ ICP-MS	
Nebulizer	PFA-ST
Nebulizer gas flow	1.02 L·min ⁻¹
Interface setup	Ni Cones, High Matrix Skimmer insert
Cell gas flow	4.8 mL·min ⁻¹ He
KED voltage	3 V
prepFAST	
Sample loop	1.5 mL
Time per analysis	66 s



Figure 2. prep**FAST** auto-dilution system connected to the iCAP RQ ICP-MS (left). ESI SC-2DX autosampler (right).

Methods

Sample Preparation for U.S. EPA 200.8 Rev 5.5

All samples were prepared according to the EPA 200.8 method. For the determination of dissolved analytes in drinking water, tap water was collected in an HDPE tank and acidified to 1% v/v HNO₃ (Optima grade acid, Fisher Chemicals). Aliquots (20 mL) from the tank were filled into 50 mL polypropylene centrifuge tubes for analysis.

The standards and quality control (QC) solutions were prepared according to the protocol outlined in Figure 1.

Mass Spectrometry

The iCAP RQ ICP-MS coupled to the Elemental Scientific prepFAST Auto-dilution System with an SC-2DX autosampler (Figure 2) was used for acquisition of all data. The iCAP RQ ICP-MS was operated in He KED mode for all analytes. Instrumental parameters are listed in Table 1.

Data Analysis

Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) software was used for quantitative assessment of the data. Working from a predefined EPA 200.8 template, the only user action needed is to enter the number of samples to be analyzed in the analytical batch. All parameters that must be monitored and achieve certain criteria to comply with EPA 200.8 are automatically checked by the Qtegra ISDS software. Samples that do not meet all criteria e.g. ISTD recovery rates or over-range analyte concentrations, are automatically diluted to an appropriate level as calculated or defined within the software and the measurement automatically repeated.

Intelligent Auto-Dilution with prepFast

Dilution factors of up to 400-fold are performed reliably and accurately, with all flows controlled by high precision syringe pumps. With the intelligent dilution, Qtegra ISDS software registers every analyte that falls outside of the defined quality control requirements. If an analyte exceeds the calibration range (Figure 3) the intelligent auto-dilution dilutes the sample and re-measures only the affected analytes without manual interaction.

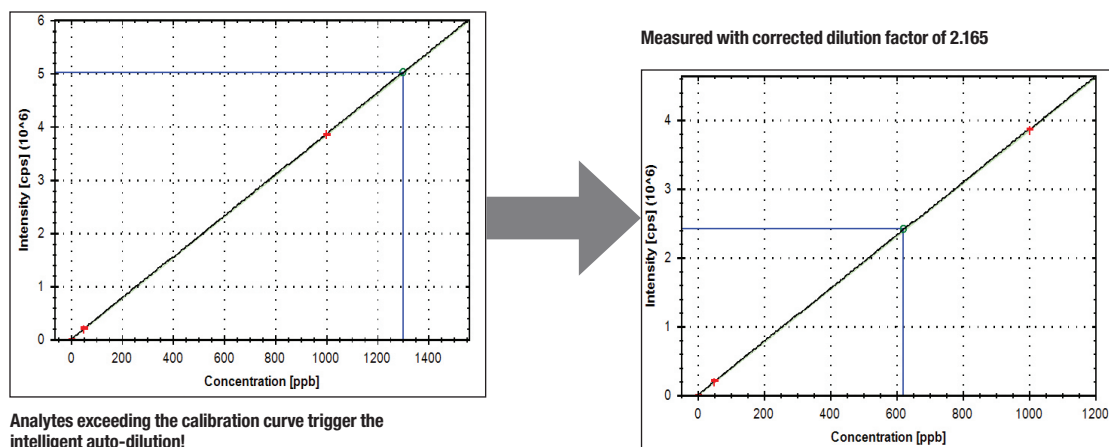


Figure 3. Analyte concentration re-analyzed by intelligent auto-dilution. Original sample (left), reanalyzed analyte with dilution factor 2.165 (right).

Results

Routine Performance of the iCAP RQ ICP-MS

Over 320 tap water samples were analyzed according to method EPA 200.8. The analysis time was on average 66 s per sample. The concentration of all analytes and their internal standard recovery was monitored throughout the whole analysis time. In total 508 analyses were run in less than 10 h. Internal standard recovery was well within the EPA 200.8 method requirements of 60 to 125 % (Figure 4).

Quality Control (QC) Samples

During the analysis run, a Continuing Calibration Verification (CCV) QC sample was analyzed every 10 samples to assess the accuracy of the calibration.

The EPA 200.8 method requires that the recovery of this QC must be within $\pm 10\%$ or within the acceptance limits of the method (EPA 200.8, rev 5.5, Table 8). All elements were found to be accurate to within $\pm 10\%$ of the known concentration, as well as the acceptance criteria and were stable over all repeated analyses (Figure 5).

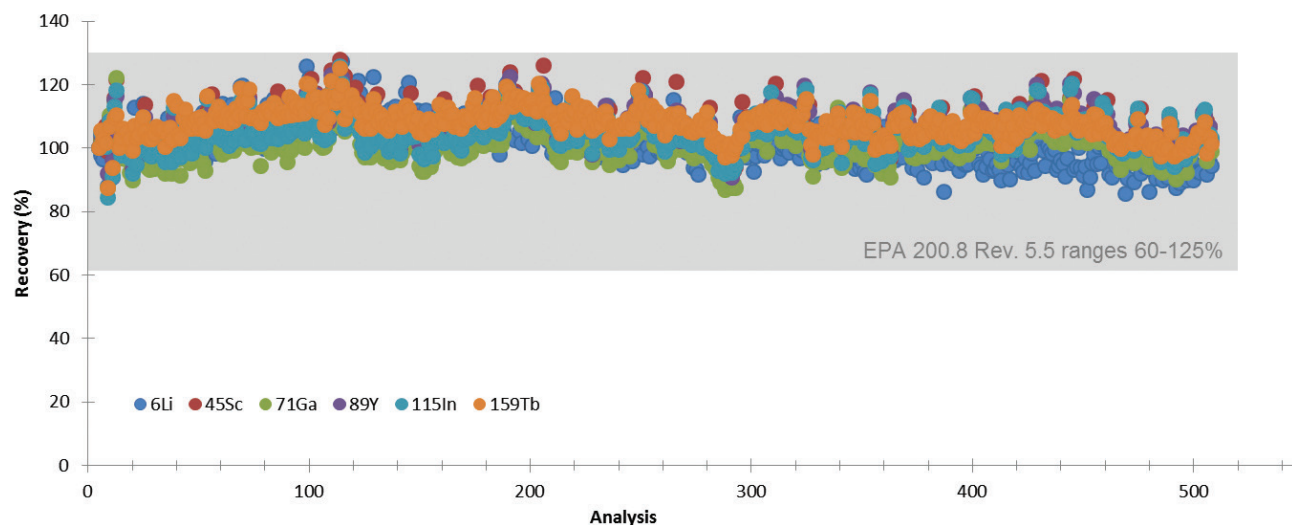


Figure 4. Internal standard response of running tap water samples and QCs showing recoveries well within the 60 – 125% range specified in EPA Method 200.8.

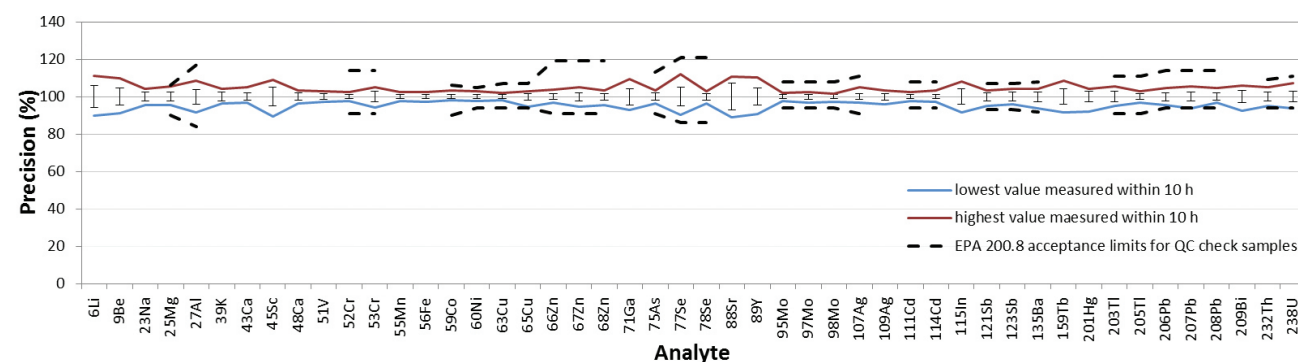


Figure 5. QC recovery and stability of the continuous calibration samples over the entire batch.

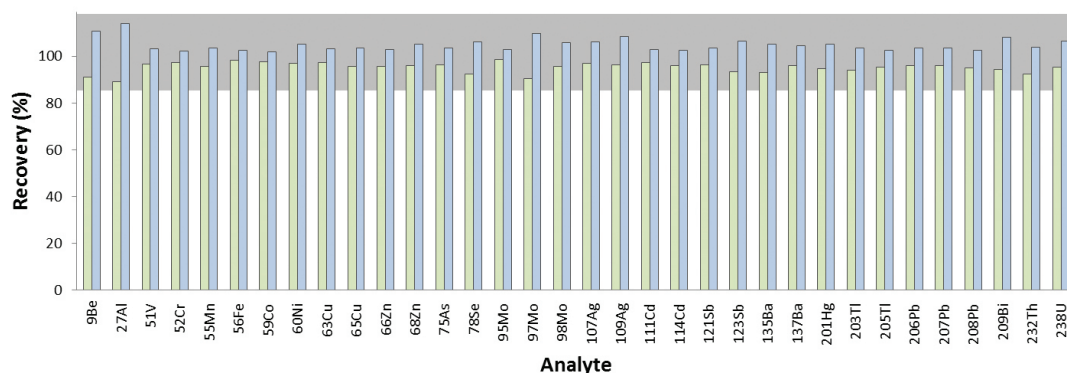


Figure 6. Laboratory Fortified Blank (LFB) recoveries from measurements. Blue bars show the highest (green lowest) recovery of the analyte measured during the 10 h run. Grey bar represent the EPA 200.8 acceptance range (85-115%) for LFB recoveries.

Laboratory Fortified Blank (LFB) and Matrix (LFM) Recoveries

The recovery of a fortified blank with known added amounts of analytes (Figure 1 a, solution 3) must be measured at least once per batch of samples. During this measurement the LFB was analyzed 32 times. In Figure 6 the calculated recovery rates are shown. All analytes show recoveries within the limits (85–115%) of EPA 200.8. Similar to the LFB recovery for every batch, one sample must also be spiked with a known amount of analytes, (Laboratory Fortified Matrix sample; LFM). All 32 LFM (Figure 1 a, solution 7) samples were within the EPA 200.8 recovery limits (75-130%).

Driven by Qtegra ISDS Software Fully Integrated

The Qtegra ISDS software provides all required features needed for the high-throughput analysis of environmental samples. Together with the fully integrated prepFAST system, Qtegra ISDS software offers:

- Prescriptive dilution of samples and calibration standards.
- Continuous monitoring of all quality controls (fortified matrix and blank recoveries or duplicate sample verification)
- LabBook feature that starts an intelligent sequence, with full QA/QC protocol and processes and reports results.
- Comprehensive, user definable reports enabling flexible export to external LIMS software packages.

Intelligent auto-dilution for samples exceeding the calibration range is fully integrated. Samples re-measured by the Qtegra ISDS software are added automatically to the sample list and clearly identified by a plus sign (Figure 7).

Conclusion

The Thermo Scientific iCAP RQ ICP-MS equipped with an ESI Autosampler and prepFAST Auto-dilution System was successfully validated for use with US EPA Method 200.8. With the robust iCAP RQ ICP-MS paired with an ESI prepFAST auto-dilution system, it is possible to run the entire analysis (encompassing sample dilution, calibration and measurement) with minimal manual intervention. After optimizing the uptake and washout parameters, the high sensitivity and stability of the iCAP RQ ICP-MS readily achieved the goal of 52 EPA Method 200.8 analyses per hour.

Robustness

The iCAP RQ ICP-MS delivers reliable analysis of drinking water with minimal drift when equipped with the high matrix insert. For extra robust operation in the face of higher matrix samples, the system can be equipped with the robust plasma interface.

Productivity

The iCAP RQ ICP-MS in combination with the ESI prepFAST Auto-dilution System is the ideal system to measure environmental samples in a high-throughput laboratory.

Simplicity

With the prescriptive and intelligent dilution capabilities provided by the system, manual sample preparation and data post processing is minimized.

No Impact on Bench Space

The integrated dual valve assembly is mounted directly beneath the sample introduction system, minimizing sample pathways.

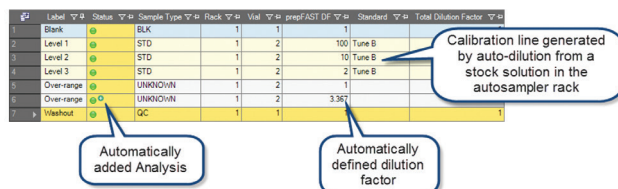


Figure 7. Screenshot of the intelligent auto-dilution process in Qtegra ISDS Software.

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