

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

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## Key Words

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

## Goal

To demonstrate robust high throughput analysis of environmental samples using 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 Qc ICP-MS coupled to the ESI prepFAST auto-dilution system.

## Introduction

EPA Method 200.8 analyses are now routinely performed in many laboratories for the analysis of trace metals in drinking and waste waters. 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 have strong potential in automating much of the sample preparation and data review with automated re-runs of any samples that do not meet predefined limits. Auto-dilution systems can automatically create a calibration set of standards from one stock standard and automatically dilute each sample to a predefined dilution level, eliminating the need for an analyst to spend valuable time on these activities and saving cost on water, analytical reagents as well as analytical lab-ware that would be used in a manual preparation.



Fast sample throughput is another driving factor when implementing routine SOPs. Throughput in the approach applied here is improved by the discrete sampling of the auto-dilution system which dramatically reduces uptake and washout time as well as the use of one measurement mode for the analysis of all the analytes in the method. The use of kinetic energy discrimination with helium as a cell gas (He-KED) ensures universal interference removal and confidence in the accuracy of the analytical results. Whereas other ICP-MS systems require multiple methods for the analysis of drinking water, the Thermo Scientific™ iCAP™ Q 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 the 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 greatly the method development.

This application 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 Q ICP-MS.

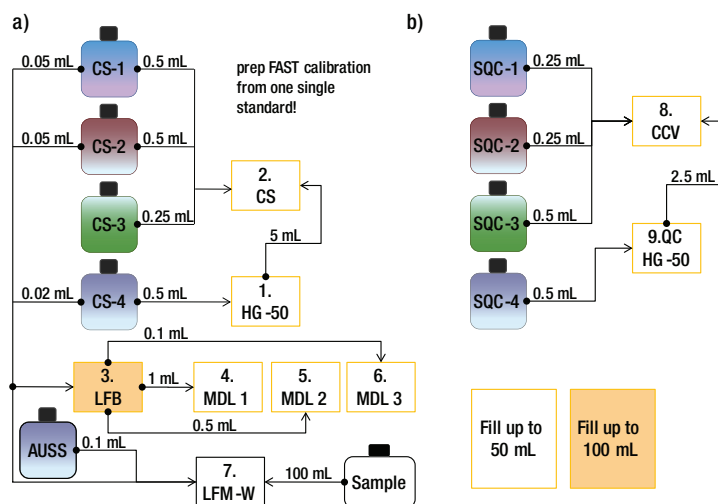


Figure 1. Scheme of (a) standard and (b) QC solutions required for EPA 200.8.

Table 1. Instrument conditions.

Parameter	Value
<b>iCAP Q ICP-MS</b>	
Nebulizer	PFA-ST
Nebulizer gas flow	1.02 L·min <sup>-1</sup>
Interface setup	Ni Cones, High Matrix Skimmer insert
Cell gas flow	4.8 mL·min <sup>-1</sup> 100% He
KED voltage	3 V
<b>prepFAST</b>	
Sample loop	1.5 mL
Time per analysis	68 s



Figure 2. prepFAST auto-dilution system connected to the iCAP Q 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 determination of dissolved analytes in drinking water, 3 L of tap water was collected in an HDPE tank and acidified to 1% v/v HNO<sub>3</sub> (Optima grade acid, Fisher Chemicals). Aliquots (20 mL) from the tank were filled into 50 mL polypropylene centrifuge tubes and for analysis.

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

### Mass Spectrometry

The iCAP Qc ICP-MS coupled to the prepFAST Auto-dilution System (Figure 2) with an Elemental Scientific SC-2DX autosampler was used for acquisition of all data. The iCAP Qc 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 is to enter the number of samples to be analyzed in the analytical batch. All parameters that need to be monitored and achieve certain criteria to comply with EPA 200.8 are automatically checked by the software. Samples that do not pass all criteria (e.g. ISTD recovery rates or over-range of analyte concentrations) will be automatically diluted at a suitable level that is calculated or defined within the software and the measurement repeated.

### Intelligent Auto-Dilution with prepFAST

Dilution factors of up to 400-fold are reliably and accurately performed 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 will dilute the sample and re-measure only the affected analytes without manual interaction.

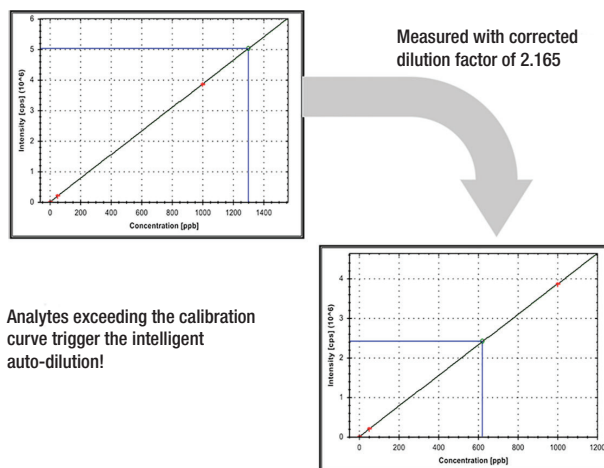


Figure 3. Analyte concentration re-analyzed by intelligent auto-dilution. Original sample (left), reanalyzed analyte with dilution factor 2.165 (right).

Routine Performance of the iCAP Q ICP-MS

Over 100 samples were analyzed according to method EPA 200.8. The analysis time was on average 68s per sample. The concentration of all dissolved analytes and their internal standard recovery was monitored throughout the whole analysis time. Internal standard recovery of the internal standards 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 as to assess the accuracy of the calibration. The EPA 200.8 method requires that the recovery of this QC must be within +/- 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 ( $< 2.5\%$  RSD) over all repeated analyses (Figure 5).

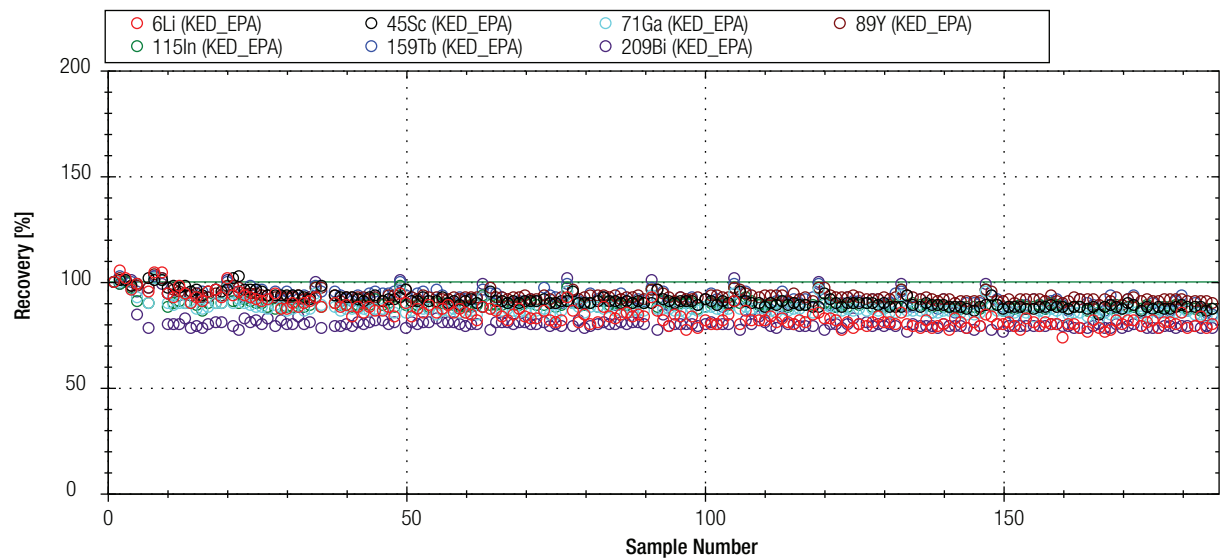


Figure 4. Screenshot of Qtegra ISDS software. Internal standard response of running tap water and QC samples showing recoveries are well within the 60 – 125% range specified in Method 200.8.

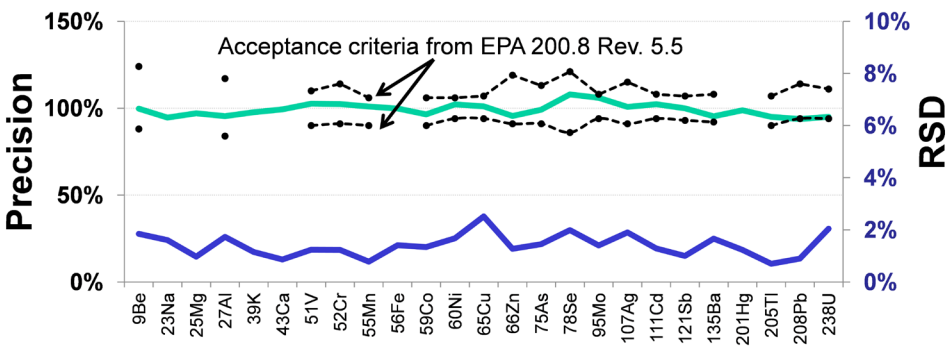


Figure 5. QC recovery and stability of the continuous calibration samples over the entire batch (n=11).

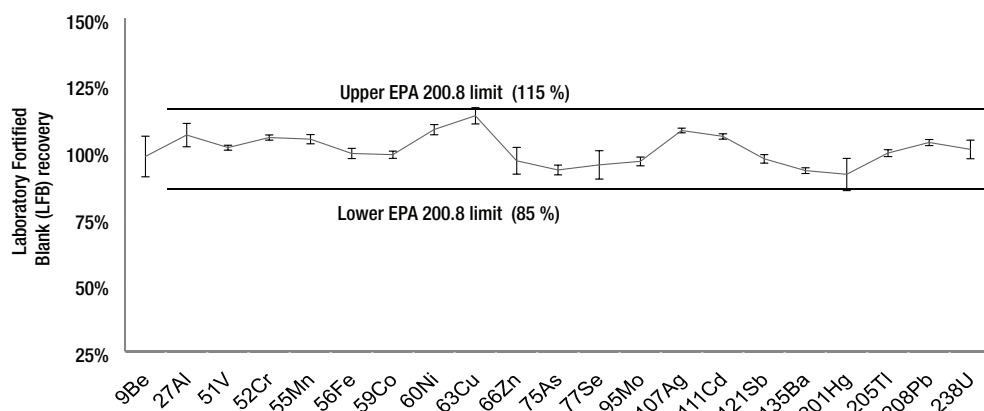


Figure 6. Laboratory Fortified Blank recoveries from QC samples of four measurements. The error bars represent the RSD.

### Laboratory Fortified Blank (LFB) Recoveries

The recovery of a fortified blank with known added amounts of analytes (Figure 1 a, solution 5) is required to be measured at least once per batch of samples. During this measurement the LFB was analyzed four times. In Figure 6 the calculated recovery rates are shown. All analytes show concentrations within the limits (85–115%) of EPA 200.8.

### Driven by Qtegra ISDS Software

#### Fully Integrated

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

- Prescriptive dilution of samples and calibration standards.
- All quality controls (fortified matrix and blank recoveries or duplicate sample verification) needed by a specific method are monitored continuously during the analysis.
- The LabBook starts your intelligent sequence, with full QA/QC protocol and processes and reports your results.
- Comprehensive, user definable reports allow for 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 marked with a plus sign (Figure 7).

### Conclusion

The Thermo Scientific iCAP Q ICP-MS equipped with an ESI Autosampler and prepFAST Auto-dilution System was validated for use with US EPA Method 200.8. With the rugged iCAP Q ICP-MS and the 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 Q ICP-MS achieved the goal of 52 EPA Method 200.8 analyses per hour.

### High Throughput

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

### Ease of Use

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.

Label	Status	Sample Type	Rack	Vial	prepFAST DF	Standard	Total Dilution Factor
Blank	+	BLK	1	1	1		
Level 1	+	STD	1	2	100	Tune B	
Level 2	+	STD	1	2	10	Tune B	
Level 3	+	STD	1	2	2	Tune B	
Over-range	+	UNKNOWN	1	2	1		
Over-range	+	UNKNOWN	1	2	3.367		
Washout	+	QC	1	1			1

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

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