TECHNICAL NOTE 44481

Robust and consistent data acquisition for the analysis of water samples using ICP-MS

Authors: Bhagyesh Surekar,
Cristian Cojocariu and Daniel Kutscher;
Thermo Fisher Scientific, Bremen, Germany

Keywords: Drinking water analysis, ICP-MS, routine maintenance, long term performance, robustness

Goal

This note highlights the robustness of the Thermo Scientific™ iCAP™ RQ ICP-MS for the unattended analysis of drinking waters over six weeks (or 27 days of operation). At the same time, it provides operators such as lab technicians and lab managers useful information for improving maintenance schedules and ultimately improve uptime.

Introduction

Inductively coupled plasma mass spectrometry (ICP-MS) is a widely recognized technique for trace element determinations, providing outstanding detection limits and a wide dynamic range. However, for applications in regulated markets, robust and reliable operation of the instrument is often more important to achieve consistent performance every day, rather than achieving ultimate detection performance. This is the foundation for the ability to provide results back to both internal and external clients in time and achieving return on investment.

Applicable regulated methods for screening of water samples, foodstuffs, or pharmaceutical products require adherence to strict quality control protocols to ensure data quality and accuracy of results. The ultimate objective for analytical testing laboratories is to analyze a variety of sample types in a single method. Drift and deviation of the internal standard as well as failed QC checks caused by the samples lead to additional work, and ultimately cost, as certain samples need to be identified, potentially diluted, and re-analyzed.

To achieve consistent operation over an extended period (and ultimately, the lifetime of the instrument), it is important that the system is designed with robustness in mind. This includes crucial components, such as the sample introduction system, the plasma generator and components of the mass spectrometer, such as ion optical devices, collision reaction cell (CRC), and the detector. At the same time, it is equally important that the system is maintained properly to assure that all individual parts are in perfect condition to fulfill their task. For laboratories running regulated methods to monitor the quality of drinking waters, ensure the absence of toxic elements in waste waters, or analyze soils and sediments, routine maintenance and troubleshooting can be significant sources of downtime, causing inefficiency and loss of productivity. It is therefore of utmost importance that routine maintenance can be executed in an easy and straightforward manner, requiring minimal time. At the same time, consistent intervals for performing maintenance are of high value, as the unproductive time of the instrument can be planned in advance and accounted for in the working plan of the laboratory.

The instruments of the Thermo Scientific™ iCAP™ Qnova™ Series ICP-MS were designed to allow robust and reliable analysis while being easy to maintain. The key components that operators handle most, such as the nebulizer, spray chamber, and interface, can be easily accessed and disassembled for effortless maintenance and minimal downtime.

To enable the productive analysis of the sample matrices typically analyzed in routine laboratories, the instruments are equipped with exchangeable skimmer cone inserts, a proprietary feature, to reduce the impact of differing amounts of total dissolved solids (TDS), which may significantly affect the response of the internal standard, leading to failed samples and hence the need to dilute and re-analyze. The instruments include three types of skimmer cone inserts, each for a different range of applications. In most cases, the High Matrix insert provides the best balance between matrix tolerance and sensitivity for most sample types, so that a wide range of samples can be analyzed without the need to change the configuration of the instrument.

Experimental

For this study, an iCAP RQ ICP-MS was used in conjunction with a Teledyne™ CETAC™ ASX 560 autosampler. The instrument was operated using standard conditions and the exact system configuration is shown in Table 1.

Table 1. System configuration of the iCAP RQ ICP-MS

Nebulizer	Glass concentric, MicroMist
Spray chamber	Quartz cyclonic
Torch	Quartz
Injector	2.5 mm i.d., quartz
Cones	Ni sampler and skimmer cone, High Matrix skimmer cone insert
Peristaltic pump tubing	PVC tube orange/yellow (0.508 mm i.d.) for uptake; Santoprene™ tube gray/gray (1.295 mm i.d.) for rinse
Uptake/wash time	60 s each
Time per sample	208 s

On every day of operation (usually Monday through Friday), the instrument was switched on, and after an automatic warm-up time of 20 minutes, the instrument´s settings and performance were verified. This basic system suitability test comprised a performance report in STD mode (or No Gas mode), followed by a performance report in KED mode, which was the only mode used for the analysis of all elements in this study. It is important to run a performance check also in standard mode (although not used in the actual measurement) to verify correct settings of the plasma conditions (i.e. oxide formation below 2.0%) and general performance of the instrument.

The iCAP RQ ICP-MS was used to analyze an identical sequence containing locally collected tap water. The tap water was acidified to contain 2% HNO_3 for stabilization of the elements in solution and fortified with 100 $ng \cdot mL^{-1}$ of gold to stabilize mercury in solution and avoid memory effects for this element effectively. The analysis was carried out following a quality control protocol similar to the requirements in applicable methods for the analysis of drinking waters (i.e., US EPA Method 200.8 or EN-ISO 17294 as examples). In short, acceptable limits for the internal standard recovery were within 60–130%, and for QC recovery between 80–120%.

The calibration solutions used for establishing multi-element calibration curves and the QC standards for all elements are summarized in Table 2.

Table 2. Linearity and QC standards used. All concentrations are given in $\mu g \cdot L^{-1}$.

Elements	Std 1	Std 2	Std 3	Std 4	Std 5	Std 6
Na, Mg, Ca, K	10	50	250	1,000	5,000	25,000
Hg	0.01	0.05	0.25	1	5	25
Be, Al, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Rb, Sr, Mo, Ag, Cd, Sb, Ba, Tl, Pb, Th, U	0.1	0.5	2.5	10	50	250
QC standard						
Na, Mg, K, Ca	1,000					
Hg	1					
Be, Al, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Rb, Sr, Mo, Ag, Cd, Sb, Ba, Tl, Pb, Th, U	10					

This pre-defined sequence was scheduled to run for approximately 10 hours and contained a total of 188 samples (including all blanks, standards, and QC checks) and a total of 160 unknown samples per day.

Corrective actions were executed if the performance report failed or if QC failures, sample failures, or bad calibration curves were observed and included an autotune as a first step of troubleshooting. In accordance with best practices, the steps summarized in Table 3 are generally recommended and were taken as a response to specific incidents.

In addition, the results obtained in the performance report can provide useful indication to identify specific issues during troubleshooting. This is highlighted in Table 4.

Table 3. Regular maintenance of the system. Applicable procedures are summarized in the Operating Manual of the system.

Maintenance action	Frequency	Comment
Visual inspection of all components of the sample introduction system	Weekly	No subsequent action required unless contamination or potential wear out was observed.
Cleaning of glassware (nebulizer, spray chamber, torch and injector)	Upon indication	Elevated concentration levels in the blanks may indicate contamination in the sample introduction system. Drift of the internal standard may indicate a problem with sample delivery. Elevated nebulizer backpressure indicates a potential obstruction of the nebulizer. Alternating a spare set of components can be used to improve uptime.
Cleaning of cones	Upon indication	Indication about required cleaning or exchange of the cones can be obtained from the performance report (i.e. low ^{59}Co sensitivity and $^{59}Co/^{35}Cl^{16}O$ ratio).
Mass calibration	Upon indication	Checked in the performance report and corrective action automatically triggered.
Detector cross calibration	Upon indication	Checked in the performance report and corrective action automatically triggered.

Table 4. Identification of specific issues. Applicable procedures are summarized in the Operating Manual of the system.

Issue/reason for failure of the performance report	Solution
Low sensitivity/high oxides or doubly charged ions	Inspection of the sample introduction system components, especially cones, nebulizer, and spray chamber.
	Check nebulizer supply pressure to eliminate potential blockage as a reason, clean if needed.
	Source Autotune: To adjust torch position relative to the interface, optimize the nebulizer gas flow and first ion lenses in the mass spectrometer.
Poor signal stability leading to high RSD failure	Inspection and potential exchange of the peristaltic pump tubing (shape, color).
	Inspection of all components of the sample introduction system, especially nebulizer and spray chamber.
Low sensitivity and failure of Co/ClO ratio in KED mode	Inspection of all components of the sample introduction system, especially cones (if not done previously).
	Inspection and cleaning of the cones. Autotuning of the KED mode: optimization of helium gas flow and related ion optical components.

Built-in QC testing

Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software contains a full feature set for setting up regular QC checks. Available tests include blank tests (i.e., ICB and CCB) for verification of contract required detection limits, calibration tests (ICV/CCV), paired sample tests (duplicates and serial dilutions), spike tests (matrix spike, fortified blanks), as well as continuous tests (internal standard recovery, relative stability verification). These tests can be customized with respect to the selection of specific analytes and acceptance criteria (applicable warning and failure limits for each analyte). Results are displayed using customizable color coding, also in all subsequent views used for data evaluation. In case of a QC warning or failure, corrective actions can also be automatically triggered by the Qtegra ISDS Software, including the option to rinse and repeat the test (in which case a new line will be automatically added to the sequence), full re-calibration and an automatic repeat of all samples analyzed after the last successful QC check, and also aborting the remaining sequence in order to avoid an unnecessary consumption of both argon gas and samples. Due to the available functionality, the sequences were run completely unattended and abortion of LabBooks caused by failed QC checks was accomplished automatically by Qtegra ISDS Software alone. Samples that were not acquired because of an interruption of the planned sequence were rescheduled for analysis on the next day.

Qtegra ISDS Software also monitors a wide range of hardware parameters during a measurement and stores them on a per sample basis in the same data file, the LabBook. The monitored parameters include a wide range of utilities, such as readbacks of plasma power, nebulizer flow and backpressure, as well as set and read back voltages of all ion lenses in the system. Therefore, a complete record of the condition of the instrument is generated in every analysis, enabling effective troubleshooting of potential issues related to hardware malfunction.

Results and discussion

Over the entire period covered in this study (six weeks or 27 days of operation), the performance of the iCAP RQ ICP-MS exceeded the minimum performance specifications significantly. The average performance is highlighted together with applicable limits in Table 5.

Table 5. Applicable performance criteria for the iCAP RQ ICP-MS and average performance during study

Standard mode		
Parameter	**Specification**	**Average performance**
^{7}Li [cps/(µg·L^{-1})]	55,000	118,249
^{59}Co [cps/(µg·L^{-1})]	100,000	147,067
^{115}In [cps/(µg·L^{-1})]	240,000	335,415
^{238}U [cps/(µg·L^{-1})]	330,000	565,609
Oxide ratio [%] ($^{140}Ce^{16}O/^{140}Ce$)	2.0	1.7
Doubly charged ratio [%] ($^{137}Ba^{++}/^{137}Ba$)	3.0	2.8
KED mode		
^{59}Co [cps/(µg·L^{-1})]	30,000	54,367
Interference suppression factor ($^{59}Co^{+}/^{35}Cl^{16}O^{+}$)	>18	>25.6

Even on the day with the lowest sensitivity obtained, the performance of the system still exceeded specifications between 11 and 44%. The mass calibration was stable for the entire duration of the study as indicated by the daily performance reports.

The planned sequence ran without any issues and deviations on almost all days of this study. On two days, interruptions to the planned sequence were caused by issues with the manually prepared QC check solutions (i.e., solution vial empty). On one day, an increased drift of the internal standard lead to an automatic abortion of the sequence through the QC management tools in the Qtegra ISDS Software. However, the constant monitoring of instrument related settings easily facilitated subsequent troubleshooting. In this case, the increased drift of the internal standard correlated with an increased nebulizer supply pressure (up to 40% increase during the run), so that the nebulizer could be identified quickly as the reason for failure, and subsequent cleaning followed by an autotune completely resolved the issue. In addition, as a performance report was run prior to troubleshooting on the next day, confirmation of the issue and the root cause was readily achieved and subsequent failures or downtime effectively avoided.

thermo scientific

A full overview of all QC samples that were successfully acquired (>200) is shown in Figure 1. As can be seen from the data, the average recovery was well within the regulatory limits. The standard deviation for individual elements across all QC samples was within ±6%.

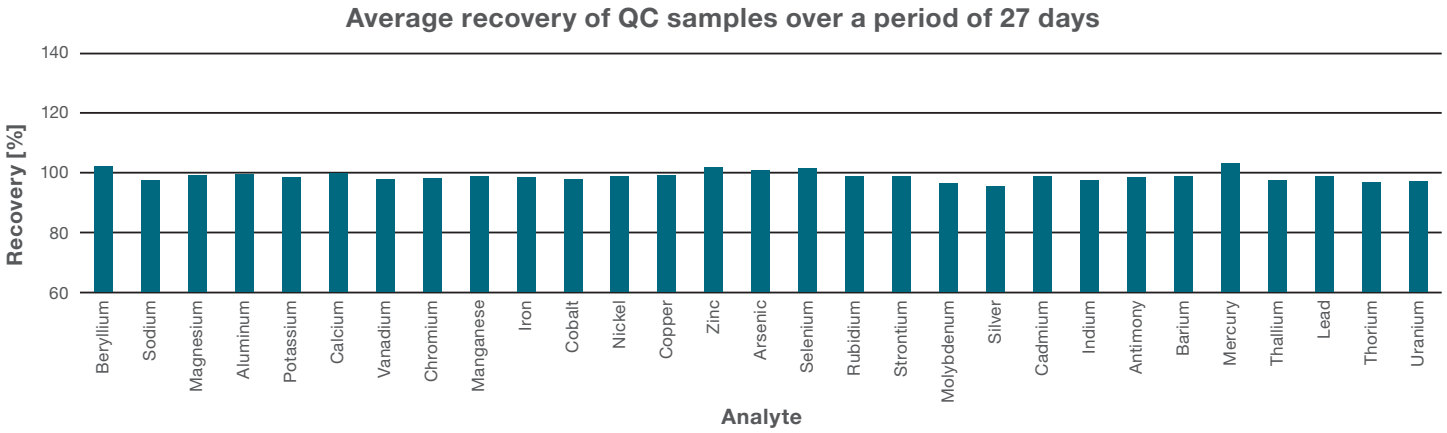

Figure 1. Average QC recovery observed in more than 200 QC samples acquired over 27 different days

Conclusion

Over several weeks, a predefined sequence containing 160 invoiceable samples in a total batch of 188 samples was run during 27 operating days on an iCAP RQ ICP-MS. In total, close to 4,000 individual samples were acquired in this period. Over the entire time, the system's performance exceeded specifications significantly, and allowed required detection limits and consistent response to the required QC checks to be easily achieved. The built-in feature set for automated QC checks in the Qtegra ISDS Software together with its leading data visualization allowed for easy creation of sequences for unattended daily operation and help with interpretation of the data. This highlights the potential of the iCAP RQ ICP-MS to cope with the key requirements in a busy laboratory performing analytical testing of all kinds of water samples, namely to allow for robust and reliable analysis of a high number of samples over an extended period.

Find out more at **thermofisher.com/ICP-MS**

ThermoFisher SCIENTIFIC