thermo scientific

TECHNICAL NOTE 43225

Increasing the productivity of ICP-OES with the Thermo Scientific Qtegra ISDS Software

Authors

Dr. Elena Chernetsova,
Application Specialist,
Thermo Fisher Scientific,
Bremen, Germany

Keywords

Intelligent Uptake and Rinse, Productivity, Runtime, Throughput, Sprint valve

Introduction

Thermo Scientific™ iCAP™ 7000 Plus Series ICP-OES are field-proven instruments for routine and demanding applications. Designed specifically for high performance combined with low cost of ownership, the iCAP 7000 Plus Series ICP-OES features an echelle optical design with a unique Charge Injection Device (CID) detector that offers the highest levels of analytical performance. The iCAP 7000 Plus Series ICP-OES is capable of performing effective high-speed analysis due to:

- Simultaneous acquisition for all element wavelengths,
- Short exposure times (while retaining sufficient intensity for high accuracy),
- Ability to integrate high and low intensity wavelengths in a single run, and
- Simultaneous background analysis, essential for accurate measurements and short integration times.

Each ICP-OES analysis includes the following stages (Figure 1): uptake (sample flush and stabilization delay), measurement, and wash. Reducing dead time is very important for increasing sample throughput and can be achieved by using different techniques.

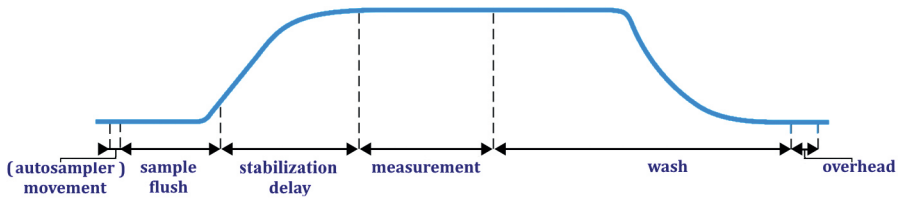

Figure 1. Signal profile during different stages of ICP-OES analysis.

ThermoFisher SCIENTIFIC

In this technical note we describe several approaches to enhance productivity and increase sample throughput using the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software for the iCAP 7000 Plus Series ICP-OES, which facilitate workflows and reduce the costs of analysis. In the following, we discuss the advantages listed here:

- Use of an autosampler for sample introduction,
- Reducing carryover using the "Intelligent Uptake and Rinse" functionality of the software, and
- Using the Sprint Valve for sample introduction.

Instrumentation

The Thermo Scientific iCAP 7600 ICP-OES Duo, equipped with a Sprint Valve for rapid sample introduction, has been used in all experiments, coupled to a Teledyne CETAC™ ASX-560 Random Access Intelligent Autosampler. Method parameters, listed in Table 1, were applied for all analyses (unless stated otherwise).

Table 1. ICP-OES method parameters.

Parameter	Setting
Pump Tubing (Mini Pump)	Sample Tygon™ white/white Drain Tygon™ yellow/blue
Pump Speed	50 rpm (without Sprint Valve) 40 rpm (with Sprint Valve)
Spray Chamber	Glass cyclonic
Nebulizer	Glass concentric
Center Tube	2 mm
Nebulizer Gas Flow	0.5 L·min^{-1}
Auxiliary Gas Flow	0.5 L·min^{-1}
Coolant Gas Flow	12 L·min^{-1}
RF Power	1150 W
Repeats	3
Exposure Time	Axial UV 15 s, Vis Radial 5 s
Wavelengths Measured Axially	Al 167.079 nm, B 249.773 nm, Ba 455.403 nm, Ca 317.933 nm, Cu 324.754 nm, Mg 279.553 nm, Mn 257.610 nm, Ni 221.647 nm, P 177.495 nm, Zn 213.856 nm
Wavelengths Measured Radially	B 249.773 nm, Ba 455.403 nm, Ca 317.933 nm, Cu 324.754 nm, K 766.490 nm, Mg 279.553 nm, Mn 257.610 nm

Sample preparation

Single element stock standards (Fisher Scientific) were used to prepare multi-element solutions at three concentration levels: high, medium, and low (Table 2). The high concentration multi-element solution was prepared in a 100 mL volumetric flask by mixing aliquots of the following single element stock standards, adding 10 mL of 2% (v/v) HNO_3 and diluting the resulting mixture with deionized water up to mark:

- 10000 mg·L^{-1}: P, B, K and Ni,
- 1000 mg·L^{-1}: Al, Ba, Ca, Cu, Mg, Mn, and Zn.

The medium and low concentration stock solutions were prepared by diluting the high stock solution with 0.2% (v/v) HNO_3 to achieve the concentrations listed in Table 2.

Table 2. Concentrations of solutions in 0.2% (v/v) HNO_3. All values are in mg·L^{-1}.

Element	High	Medium	Low
P	100	10	1
B	100	10	1
K, Ni	50	5	0.5
Al, Cu, Mn	10	1	0.1
Ba, Ca, Mg, Zn	2	0.2	0.02

As a blank sample, 0.2% HNO_3 was used for all measurements.

Use of an autosampler

Using autosamplers for ICP-OES analysis reduces user intervention and is the first step towards increasing sample throughput. Autosamplers allow implementing unattended analysis with higher reproducibility of measurements, with precise uptake and wash cycles applied to each sample.

Method

Sample uptake was preformed via a Teledyne CETAC ASX-560 Autosampler. All parameters relating to the autosampler can be set directly in the Qtegra ISDS Software (Figure 2). Timings and layout can be modified directly in the respective LabBooks, with flexible rack configurations available.

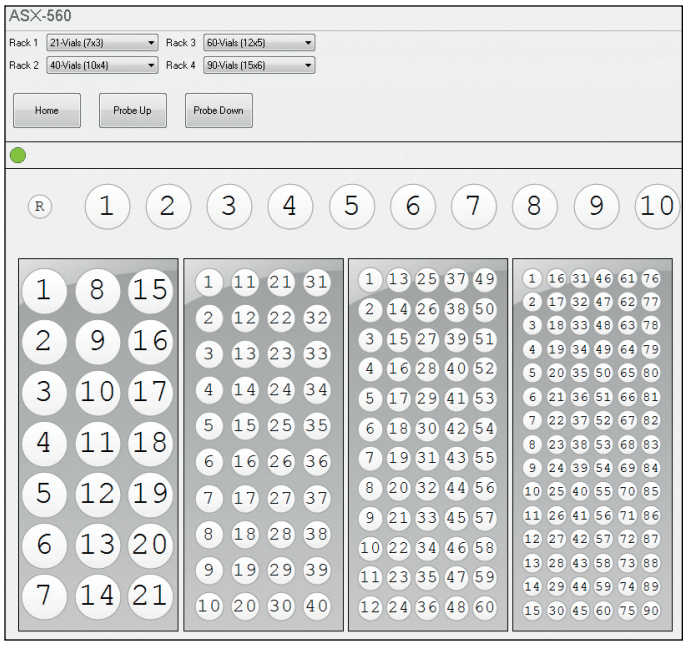

Figure 2. ASX-560 Autosampler view on the Dashboard of Qtegra ISDS Software.

Using Qtegra ISDS Software, it is possible to apply three different modes of analysis (Figure 3): *Normal*, *Speed*, and *Sprint*. These three different Analysis Modes relate to the sequencing of the plasma views.

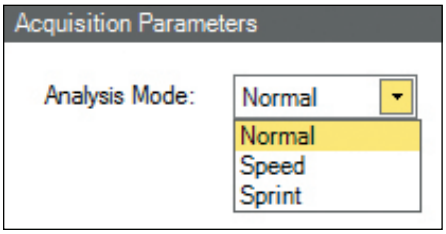

Figure 3. Analysis Mode drop-down menu in Qtegra ISDS Software.

In *Normal* Analysis Mode the analysis is carried out per replicate, while *Speed* Analysis Mode allows a faster analysis by minimizing the number of transitions between different plasma views and slits. This is clarified in the following example. When the visible range wavelengths are measured radially and the UV wavelengths are measured axially, like in this study, the measurements are carried out in *Normal* and *Speed* Analysis Modes in the following sequences, where R is Replicate and → represents the transition between plasma views.

Normal:
R1 (Vis) → R1 (UV) → R2 (Vis) → R2 (UV) → R3 (Vis) → R3 (UV)

Speed:
R1 (Vis) R2 (Vis) R3 (Vis) → R1 (UV) R2 (UV) R3 (UV)

Sprint Analysis Mode operates similarly to *Speed* Analysis Mode, but removes the pre-exposure time. The pre-exposure sets individual read out times for each pixel depending on the signal intensity and allows for more precise analysis without saturation of pixels. *Sprint* Analysis Mode is intended for trend analysis and where sample numbers are very high, therefore in this note we focus on *Normal* and *Speed* Analysis Modes only.

To demonstrate the general capabilities of using Qtegra ISDS Software with an autosampler for routine applications, 50 aliquots of the medium multi-element solution were analyzed, using the *Normal* Analysis Mode. Wash and uptake times have been set to 40 seconds in order to minimize memory effects and provide stable measurements over a prolonged period of time. These timings were preliminarily tested and provided minimal carryover between samples from this sequence. The same experiment was repeated in *Speed* Analysis Mode.

Results

The intensities of P 177.495 nm (Axial Measure Mode) and Mg 279.553 nm (Radial Measure Mode) for the 50 analyzed aliquots of a medium concentration multi-element solution are shown in Figure 4. All of the elements analyzed exhibited similar behavior to the two elements shown.

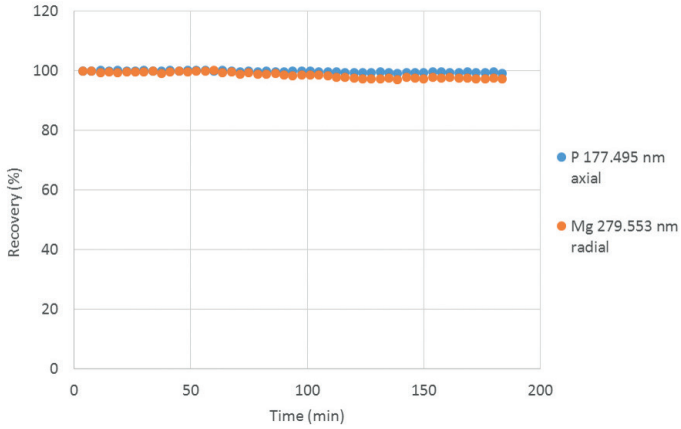

Figure 4. Recoveries of P 177.495 nm (axial) and of Mg 279.553 nm (radial) for 50 samples, analyzed in *Normal* Analysis Mode.

Using a triplicate analysis for 50 samples gives a total analysis time of 3 hours and 8 minutes when using *Normal* Analysis Mode. This equates to an analysis time of 3 minutes 45 seconds per sample.

Analyzing the same sequence of samples in the *Speed* Analysis Mode proves an increased sample throughput (Figure 5). In this mode, analysis per sample took 20 seconds less than in the *Normal* Analysis Mode, with a total analysis time for 50 samples of 2 hours and 52 minutes.

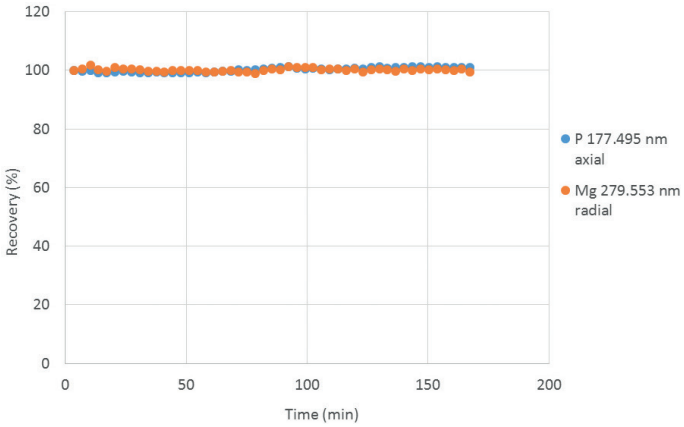

Figure 5. Recoveries of P 177.495 nm (axial) and of Mg 279.553 nm (radial) for 50 samples, analyzed in *Speed* Analysis Mode.

Reduction of carryover using the flexibility of Intelligent Uptake and Rinse

ICP-OES productivity depends not only on the runtime per single sample. It also highly depends on the efficiency of the rinse, which is implemented to prevent carryover. If carryover from one sample to the next takes place, this can lead to incorrect data, losses of samples and increased analysis costs due to the necessity to repeat experiments. With the improved sensitivity and dynamic range of the iCAP 7000 Plus Series ICP-OES, sample introduction and washout become a limiting factor in ICP-OES analysis. A solution to reduce carryover is the "Intelligent Uptake and Rinse" functionality of Qtegra ISDS Software. It automatically adjusts the time used for sample uptake and rinse, depending on the stability and level of a signal, which is continually monitored.

Before starting the whole analysis sequence with the Intelligent Uptake and Rinse, the analyst needs to define the proper threshold limits. This can be done by estimating the expected signal and background intensities from the element subarray windows of Qtegra ISDS Software. The threshold limits have to be entered into the Intelligent Uptake and Rinse section of a LabBook.

The acquisition starts when the signal intensity is above the specified threshold value. After data acquisition the system is being rinsed and whilst the wash is taking place, the signal is monitored continuously. When the monitored signal falls below the specified threshold, the autosampler moves to the next sample.

Method

The following experiment has been designed to demonstrate the Intelligent Uptake and Rinse for minimizing carryover from one sample to another. In this experiment low, medium, and high concentration multi-element solutions were analyzed, representing a concentration range of three orders of magnitude. The parameters for Intelligent Uptake and Rinse have been optimized using P 177.495 nm in Axial Measure Mode (Figure 6).

A sequence of 15 samples at three different levels of concentrations was analyzed in batches of the following order: First, a medium concentration sample was analyzed, then a low concentration sample, and finally a high concentration sample (three repeated measurements were carried out for each sample run). The analysis of this sequence was repeated 5 times. After this, a blank 0.2% HNO_3 sample was analyzed 10 times, and the intensities of these 10 replicates were monitored. To compare the benefits of the Intelligent Uptake and Rinse function to an analysis without using this function, the same sequence was run in the *Speed* Analysis Mode, using the method parameters as described in the section *Use of an Autosampler* without applying the Intelligent Uptake and Rinse.

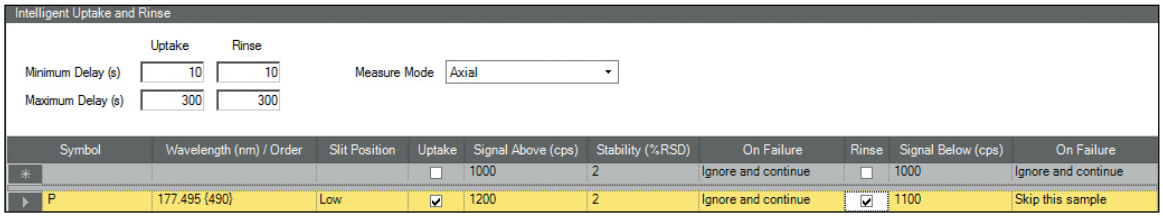

Figure 6. Setting up Intelligent Uptake and Rinse parameters for P 177.495 nm in Axial Measure Mode.

Results

Two very different patterns in the results can be seen when comparing the signal intensities in blank samples, analyzed immediately after the introduction of the high concentration multi-element solution. Without the Intelligent Uptake and Rinse, the decrease in the intensities for the blank samples to base line is very slow (Figure 7). However, when the Intelligent Uptake and Rinse is used, the baseline concentration is achieved instantly.

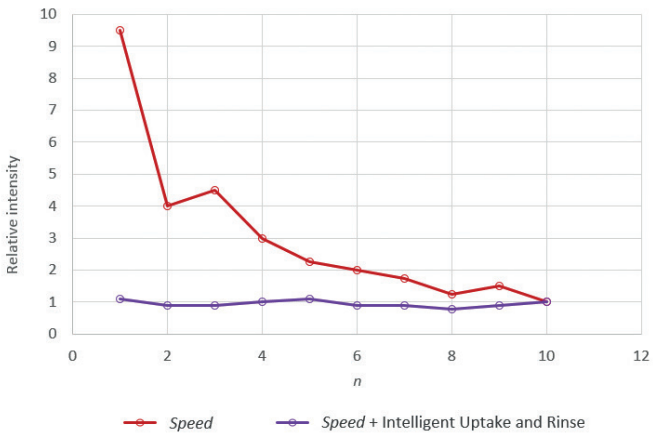

Figure 7. Relative intensities of P 177.495 nm (axial) in replicate analyses of one blank sample, after running 15 samples with P levels at 3 orders of magnitude without and with Intelligent Uptake and Rinse. n is the number of blank replicate analyses after running a highly concentrated P-containing sample.

The Intelligent Uptake and Rinse feature of the Qtegra ISDS Software helps to reduce the costs of analyzing your sample due to the flexible and intelligent on-the-run selection of adequate uptake and rinse times. It is possible to monitor multiple element wavelengths simultaneously using this feature. Moreover, by Intelligent Uptake and Rinse it is possible to monitor the element wavelengths, e.g., for the sample matrix constituents, without recording their intensities into the final LabBook file. This helps in adjusting the instrument conditions on the run and saving hard drive space. With the Intelligent Uptake and Rinse feature valuable samples with different concentration levels of elements are saved due to their successful analysis on the first attempt, and the overall productivity of analysis is increased.

Ultrafast analysis using the Sprint Valve

Using the integrated Sprint Valve of the iCAP 7600 ICP-OES enhances analysis productivity due to minimized dead time for your application. The Sprint Valve is a 6-port injection valve with a customizable sample loop, compatible with many autosamplers, including the following models from Teledyne CETAC:

- ASX-560,
- XLR-8 extended rack,
- ASX-1400, and
- ASX-1600.

When the valve is in load position (Figure 8, upper part), the loop is filled with the sample by a vacuum pump. At the same time, the carrier/rinse solution is transported through the bubbler T-piece and through the valve ports 5 and 6 to the nebulizer. Having air bubbles between solution portions prevents analytes from the sample diffusing into the carrier solution and reduces the time between the runs.

When the valve switches to the inject position (Figure 8, lower part), the sample is pushed into the nebulizer for analysis via the carrier/rinse solution by the peristaltic pump. At the same time, the autosampler probe is moved to the rinse station and flushed with rinse solution using the vacuum pump.

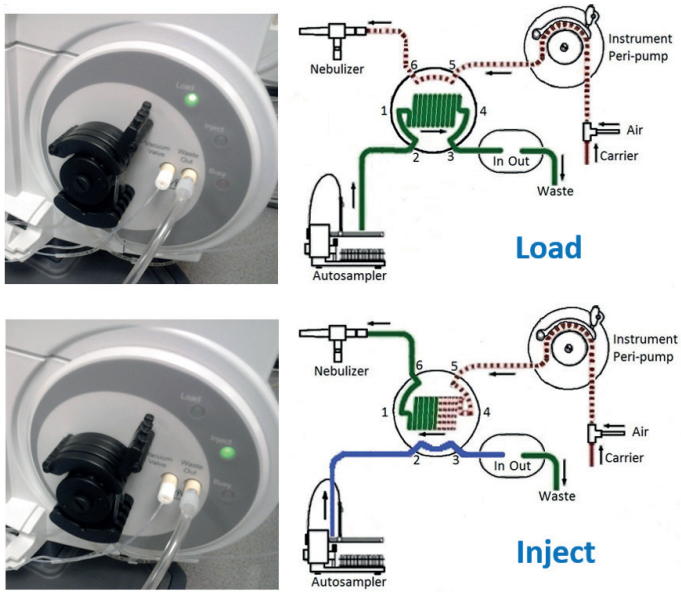

Figure 8. Sprint Valve in Load (upper part) and Inject (lower part) positions.

Method

Sprint Valve settings were optimized in the Qtegra Configurator for the medium concentration multi-element solution. The chosen values and their graphical explanation are shown in Figures 9 and 10. Using these settings, 50 aliquots of the medium multi-element solution were analyzed one after another. Uptake time included loop evacuation delay, loop rinse delay, loop load time, and equalization delay, which took 10 seconds altogether. Keeping in mind the additional time of about 5 seconds that is needed for the sample to reach the plasma and for further plasma stabilization, an overall uptake time of 15 seconds was set up in the autosampler settings in Qtegra ISDS Software.

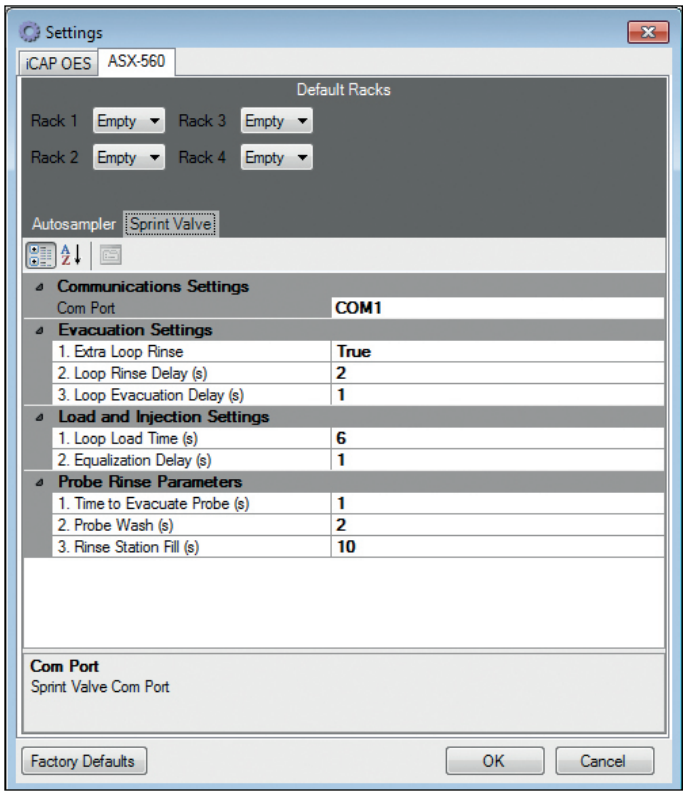

Figure 9. Sprint Valve settings in the Qtegra Configurator window.

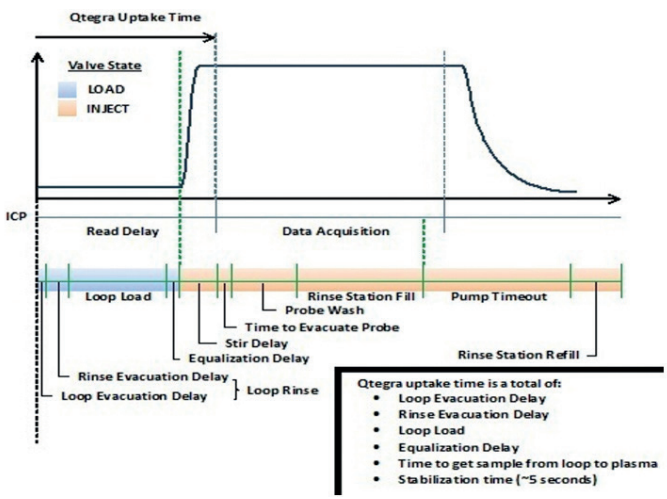

Figure 10. Graphical explanation of the Sprint Valve settings.

The *Speed* Analysis Mode was used for the measurements. For a sample loop of 4 mL, pump speed and flush pump speed were set to 40 rpm both.

For testing carryover, 10 blank samples were analyzed after running a sequence of 50 medium multi-element solution aliquots. In order to compare the results with the other methods described in this note, similar sequences of analysis were run with an autosampler without the Sprint Valve using the *Normal* Analysis Mode and using the *Speed* Analysis Mode with the Intelligent Uptake and Rinse (in this case the thresholds of Intelligent Uptake and Rinse were set for B 249.773 nm). Carryover of boron has been compared in all three modes.

Results

Analyzing 50 aliquots of the medium multi-element solution with the Sprint Valve took 2 hours 2 minutes. This equated a time per run of 2 minutes 26 seconds and was the minimum achieved during all studies described in this note.

For evaluation of the carryover, boron signal intensities at B 249.773 nm were monitored in the blank samples, because boron is known to be one of the most “sticky” elements for ICP-OES, exhibiting a high carryover. The relative intensities of boron signals in 10 blank samples are summarized in Figure 11 for the following three datasets received using an autosampler: using the *Normal* Analysis Mode, using the *Speed* Analysis Mode with the Intelligent Uptake and Rinse, and using the *Speed* Analysis Mode with the Sprint Valve.

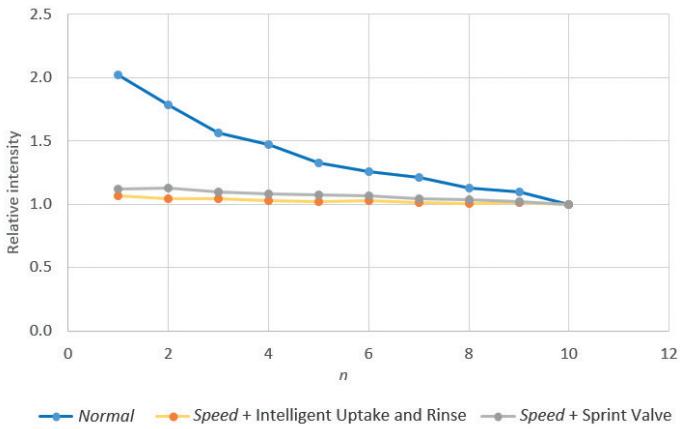

Figure 11. Relative intensities of B 249.773 nm (axial) in replicate analyses of one blank sample, after running 50 medium multi-element solution aliquots with 10 mg·L^{-1} B in the three different modes: *Normal* Analysis Mode, *Speed* Analysis Mode with Intelligent Uptake and Rinse, and *Speed* Analysis Mode with Sprint Valve. n is the number of blank replicate analyses after running the B-containing sample.

In the acquisition experiment using *Normal* Analysis Mode the carryover of boron was relatively high. This explains, that longer uptake and wash times are usually needed to analyze solutions of elements with their concentrations at ppm level. Using *Speed* Analysis Mode with the Intelligent Uptake and Rinse or *Speed* Analysis Mode with the Sprint Valve provided minimal carryover even for such a sticky element like boron with a flexible variation of sample runtime for concentrations at three different orders of magnitude. For analyzing a set of samples with concentrations at the same level, using the Sprint Valve was identified as the best solution, providing the fastest analysis time and no carryover.

Conclusion

To increase analytical productivity, Qtegra ISDS Software has a variety of features, depending on specific application, type and number of samples. Using the iCAP 7000 Plus Series ICP-OES with Qtegra ISDS Software features, it is possible to increase throughput of your analysis by 30% or more, as compared to a common ICP-OES analysis.

The maximal decrease in analysis time per single run was achieved introducing the sample *via* the Sprint Valve. Using Intelligent Uptake and Rinse is highly recommended for multiple samples with unknown levels of concentrations. It allows to flexibly reduce sample uptake to a minimum and to ensure adequate rinsing regardless of the composition of a previous sample without compromising performance. For both features introduced in this study (Intelligent Uptake and Rinse, Sprint Valve) carryover was minimized as compared to common sample analysis *via* autosampler introduction and analysis per replicate (*Normal* Analysis Mode). These approaches, implemented using the iCAP 7000 Plus Series ICP-OES with Qtegra ISDS Software, eliminate the need to re-run samples and reduce the cost of analysis, and bring your analysis workflows to a new level of robustness and confidence.

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

ThermoFisher SCIENTIFIC