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Approaches to Improving Ease of Use of ICP-MS

Usability is a primary requirement for many ICP-MS laboratories. In this issue of the Agilent ICP-MS Journal, we introduce the new Agilent 7850, a single quadrupole instrument designed for ease of use in typical ICP-MS applications. With a hardware configuration optimized for accurate analysis of varied samples, the 7850 combines high performance with smart tools to support new users in setting up and running their methods.

An application example from the food industry shows how the IntelliQuant function of ICP-MS MassHunter software can make data review easier and more reliable, speeding up reporting and reducing errors.

Finally, as speciation using HPLC-ICP-MS becomes more widely used and routine, labs require automated, unattended analysis of multiple species in larger sample batches. This approach is enabled by the Agilent Infinity II Multicolumn Thermostat HPLC modules with Quick Change valve head, which can be controlled from ICP-MS MassHunter.

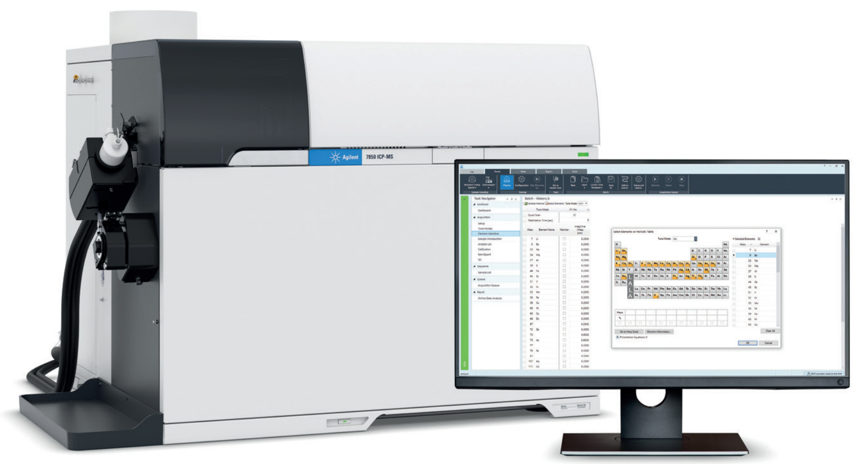

Figure 1. Introducing the Agilent 7850 ICP-MS, designed to improve your analytical workflows.

The New Agilent 7850 Optimizes Analytical Workflows for Typical ICP-MS Applications

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Requirements for ICP-MS in routine applications

Elemental analysis needs vary enormously, but there are some factors that are common across a range of industries and applications. Labs involved in analyses such as environmental monitoring, agriculture and food safety, consumer product testing, pharmaceutical manufacturing quality control, and clinical research often have similar priorities. Analysis is often performed by busy commercial laboratories, turnaround times for results are short, samples may have high and variable matrix levels, and analyte lists are long. Also, costs must be tightly controlled, while ensuring that data quality is not compromised. These industries are also often subject to regulatory oversight, with strict system suitability and quality control requirements.

The extensive list of regulated analytes and the required detection limits make ICP-MS the obvious choice for these types of analyses. But some labs still view ICP-MS as being complicated to set up, difficult to optimize and use, and expensive to maintain. This can mean that labs struggle on with less productive traditional techniques or existing, sub-optimal ICP-MS methods. A 2020 poll revealed the most time-consuming activities that impact on routine labs' workflows, as shown in Figure 1.

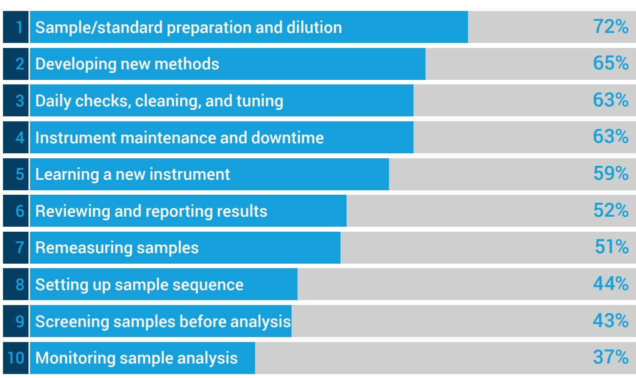

Figure 1. Unproductive and often unnecessary activities – time traps – ranked by users in a poll conducted in September 2020.

In recent years, engineers responsible for developing Agilent ICP-MS systems have focused on improving the usability of ICP-MS for routine laboratories. These developments have led to the recently released Agilent 7850 ICP-MS. Based on the successful Agilent 7800, but incorporating new performance capabilities and enhanced software functionality, the 7850 embodies ease of use in a high-performance ICP-MS package. The 7850 is focused on simplifying the analysis of varied sample types, routinely achieving the required detection limits, and ensuring accurate, interference-free results across the analyte concentration range. As a result, the 7850 addresses many of the critical time traps highlighted in Figure 1.

Accurate analysis in varied, high matrix samples

Handling high matrix levels and variable samples has long been considered among the more difficult aspects of routine ICP-MS analysis. The traditional matrix limit of 0.2% (2000 ppm) total dissolved solids (TDS) meant that samples often had to be screened to assess matrix levels before analysis. Some labs even ran each sample twice, using different dilutions for majors and traces. With Agilent ICP-MS systems, these limitations have largely been removed through High Matrix Introduction (HMI) technology with automated aerosol dilution. The 7850 benefits from Ultra-HMI (UHMI), which offers a greater level of predefined dilution factors combined with higher carrier gas flows than the HMI system used on the 7800 ICP-MS. UHMI allows a wider range of unknown high matrix samples to be analyzed, while also reducing matrix suppression, enabling varied samples to be run without the need for matrix matched calibrations.

The longstanding issue of errors due to matrix-based polyatomic interferences has also been addressed by the ORS cell – optimized for helium (He) collision mode – fitted to Agilent ICP-MS systems. He mode is the default mode for potentially interfered elements, greatly reducing time-consuming method development. Users no longer need to know which major elements are present in each sample, or to select analyte isotopes that avoid potential overlaps. Routine analysis is also easier, as workflows are more consistent, which makes the system easier to learn and reduces the potential for errors.

Helium mode also simplifies sample and standard preparation. Many labs have traditionally excluded HCl from their samples and standards, to avoid errors due to Cl-based polyatomic overlaps. On the 7850, He mode reliably resolves these Cl-based overlaps, so HCl can be used routinely for sample preparation and stabilization. The addition of HCl eliminates many of the analyte instability issues that labs previously encountered, for example poor linearity of Ag and Sn, and slow wash-in/washout of Hg. Indeed, ICP-MS users are often surprised to learn that Hg, stabilized by the addition of HCl, can be analyzed as part of a multi-element suite.

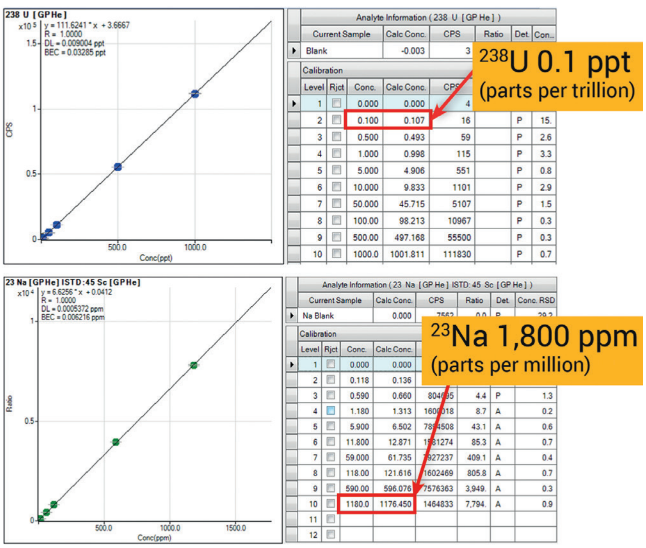

Figure 2. Analyte concentration range from 0.1 ppt to 1180 ppm – a total range of > 10 orders of magnitude.

One of the reasons labs switch to ICP-MS to replace existing metals analysis techniques is its ability to measure majors and trace elements in a single run. But covering the full range of concentrations required in many regulated methods is not always easy. Some ICP-MS systems require custom reaction cell voltages to be set up to attenuate the ion transmission for the major elements. Others require lens detuning or selection of a low-gain detector mode. The 7850 takes a simpler, more robust approach, by providing a full 10 orders analytical range at the detector. Starting from trace level standards at 0.1 ng/L (ppt), the 7850 can measure a fully ionized, monoisotopic element like Na at more than 1000 mg/L (ppm). The wide analytical range, illustrated in Figure 2, means that consistent methods can be used for a range of sample types, while over-range results – and the reruns that follow – are practically eliminated.

Simple, streamlined 7850 ICP-MS workflows

The latest revision of Agilent ICP-MS MassHunter, version 5.1, includes new features that make ICP-MS setup and routine operation easier than ever. Improved Method Wizard functionality ensures that method setup is fast and error-free. Pre- and post-run performance checks monitor system performance and recommend when any intervention is needed. And early maintenance feedback (EMF) schedules routine maintenance tasks according to your lab's sample types and workload. EMF links with a new Help and Learning Center, which puts Agilent ICP-MS expertise at your fingertips. With videos of common user tasks, tutorials, and interactive troubleshooting guides, it's like having an expert permanently in your lab.

The 7850 with ICP-MS MassHunter combines with the optional, browser-based ICP-Go user interface for simpler routine analysis. The 7850 is also compatible with ICP-MS Analyzers for turnkey setup of regulated methods.

More information on the Agilent 7850 ICP-MS

Brochure: Agilent 7850 ICP-MS, 5994-2302EN

Flyer: Free your workflow from common time traps, 5994-2758EN

Application note: Fast, Accurate Analysis of 28 Elements in Water using ISO Method 17294-2, 5994-2804EN

Application note: Routine Analysis of Soils using ICP-MS and Discrete Sampling, 5994-2933EN

Technical overview: Agilent IntelliQuant for ICP-MS, 5994-2796EN

Flyer: Smart Self-Health Checks for ICP-MS Instruments, 5994-2780EN

ICP-MS IntelliQuant Extends the Information Available in Routine Food Analysis

Jenny Nelson and Ed McCurdy, Agilent Technologies, Inc.

Routine elemental analysis of food

The elemental content of foods is routinely monitored to check the level of essential nutrient elements and potentially harmful elements such as heavy metals. Elemental concentrations can also be used to help in the fight against food fraud, such as by confirming region of origin, identifying mislabeling, or detecting adulteration. Whatever the objective of the analysis, ICP-MS is often the technique of choice for elemental analysis of foods. ICP-MS offers fast multi-element analysis, low detection limits, wide dynamic range, and high sample throughput. ICP-MS can also be easily coupled to a chromatographic device such as HPLC, providing the speciated analysis often required in food regulations.

Agilent single quadrupole ICP-MS instruments are ideal for routine food analysis, which typically requires good matrix tolerance, effective control of spectral interferences, and analysis over a wide concentration range. Food samples often contain high and variable matrix levels, which can lead to signal drift and suppression if the ICP plasma is not well optimized. Agilent ICP-MS systems operate under robust plasma (low CeO) conditions as standard, providing exceptional matrix tolerance. High Matrix Introduction (HMI/UHMI) technology further improves plasma robustness for the analysis of particularly high, percent level sample matrices. Another benefit of HMI/UHMI is that varied, high matrix sample digests can be run against a simple, synthetic calibration, without requiring matrix-matching.

The Agilent ORS^4 collision/reaction cell is optimized for effective control of common polyatomic interferences using helium (He) collision mode with Kinetic Energy Discrimination (KED). The optimized He mode avoids the need to use reactive cell gases for elements such as Se (*1*). For more unusual spectral interferences, such as doubly charged ion overlaps that cannot be resolved using He mode, Agilent half-mass mode automatically corrects the contribution, ensuring accurate analysis.

ICP-MS MassHunter IntelliQuant provides a complete elemental profile of each sample

ICP-MS MassHunter software includes several features that help laboratories run their ICP-MS analysis more efficiently, improve accuracy and certainty, and avoid re-analysis. From version 4.6, ICP-MS MassHunter includes the IntelliQuant function, which provides greater insight into each sample's composition from a two second Quick Scan acquired in He mode. No additional sample, standard, or QC analysis is required.

IntelliQuant results are calculated for all elements, not just those analytes included in the quantitative method. For example, US FDA EAM method 4.7 specifies the analysis of 12 elements, arsenic, cadmium, chromium, copper, lead, manganese, mercury, molybdenum, nickel, selenium, thallium, and zinc, in food samples. But IntelliQuant calculates semiquantitative concentrations for up to 78 elements, providing valuable additional information on sample composition.

IntelliQuant data can be viewed in a table of results or as a periodic table heat map. The periodic table view gives an easily interpreted display of the concentration ranges of all elements in each sample; darker colors indicate higher concentrations. Figure 1 shows the heat map for a dark chocolate sample analyzed using an Agilent 7800 ICP-MS (*2*).

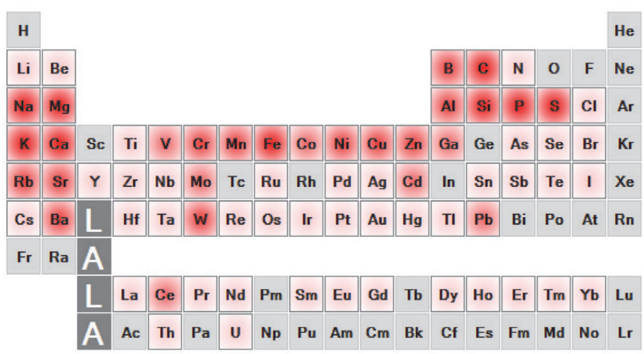

Figure 1. Periodic table heat map view of ICP-MS IntelliQuant data acquired for dark chocolate.

The heat map updates to show the currently selected sample as the user scrolls through the results table, so reviewing and comparing results is quick and easy. Identifying contaminants is also straightforward.

The use of IntelliQuant data to extend the information available from a routine ICP-MS analysis is illustrated in Figure 2. This figure shows a section of the periodic table heat maps for several different digested foodstuffs, focusing on the levels of tungsten (W). Tungsten is not included in EAM 4.7, so was not measured quantitatively, but the heat maps in Figure 2 show a relatively high level of W in the dark chocolate sample. The concentration of W was determined semiquantitatively as 1.2 mg/kg (ppm) in the original chocolate sample.

A relatively high level of titanium (Ti) is also noticeable in the donut and gummy bear confectionary samples. The raised levels are probably due to titanium dioxide food additive, widely used as a whitener in frosting.

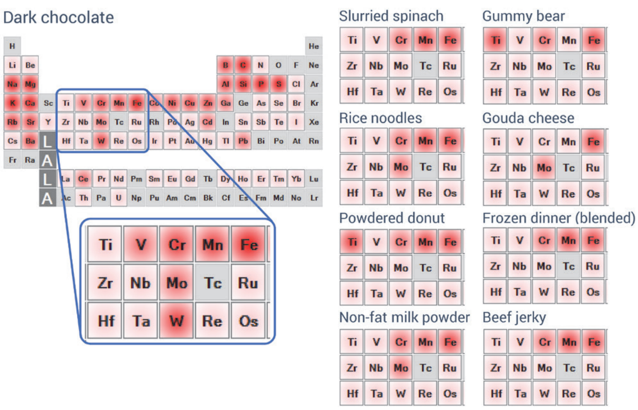

Figure 2. Periodic table heat maps for various digested foodstuffs, showing easy identification of the relatively high level of W in dark chocolate and Ti in donut and gummy bear.

The IntelliQuant full mass Quick Scan acquisition is collected in He collision cell mode by default, so common polyatomic interferences are removed effectively. This means that IntelliQuant results offer a reliable way to confirm the presence of additional or unexpected analytes using an isotope abundance template match. Figure 3 shows a section of the full mass Quick Scan spectrum acquired for the dark chocolate sample; natural isotope abundance templates are selected for W and lead (Pb).

The measured mass spectrum closely matches the theoretical isotope abundances giving added confidence in the data, which is useful when confirming queried results.

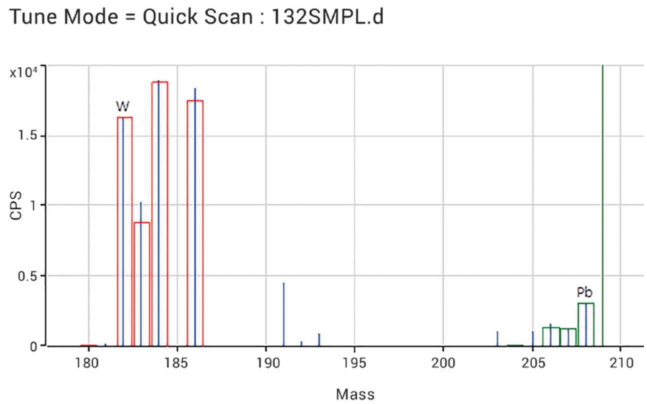

Figure 3. Section of full mass range Quick Scan for dark chocolate sample, illustrating confirmation of W and Pb by isotope template match. Other peaks shown are from Ir and Bi internal standards.

Conclusion

ICP-MS is often the technique of choice for elemental analysis in the food industry. ICP-MS is unique in providing good tolerance to high and variable matrices, effective control of polyatomic interferences, and the ability to measure from ultratrace to major element concentrations. But the scope of information available from the technique is often not fully utilized.

By combining He mode full mass Quick Scan acquisition with IntelliQuant automated semiquantitative calibration, ICP-MS MassHunter software now allows users to extract more information from each sample. With only two seconds extra acquisition time and with no need for additional calibration or sample analysis, IntelliQuant provides semiquantitative results for practically every element in every sample. The additional sample information is easy to review and interpret and can improve the user's confidence in their data quality, reducing the need for sample reruns.

More information

1. Enhanced Helium Collision Mode with Agilent ORS^4 Cell, Agilent publication, 5994-1171EN
2. Jenny Nelson *et al.* Agilent publication, 5994-2839EN

Automated Multi-Element Speciation Analysis Using HPLC-ICP-MS with a Quick Change Valve Head

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Speciation analysis using HPLC-ICP-MS

ICP-MS is widely accepted as the technique of choice for trace element analysis across many industries and sample types. But total elemental analysis doesn't always provide the full picture. The mobility, bioavailability, and toxicity of several elements, including As, Hg, Cr, Pb, Sn, Br, I, Se, and Sb, depends on the element's chemical form or species. To enable these different chemical forms to be separated before analysis, a chromatographic technique, such as HPLC, is coupled to the ICP-MS (*1*).

HPLC-ICP-MS methods are normally used to detect species that can be separated on a single column, which means they are usually limited to measuring the different forms of a single element. However, labs often need to analyze species of several elements in each sample, such as monitoring inorganic As, organotin, and methyl mercury in seafoods. Multi-element species can be run using the same column and mobile phase (*2*), but this may lead to compromised conditions and degraded performance. So labs typically run a separate sequence for each element, manually changing the column and mobile phase between runs. This approach affects turnaround time and productivity.

Quick change column selector valve heads

Agilent's Infinity II HPLC system Multicolumn Thermostat (MCT) column compartments, shown in Figure 1, can be fitted with a Quick Change valve head to switch between 2, 4, 6, or 8 column positions. A solvent selection valve is also available, enabling switching between up to 12 different mobile phases. The complete HPLC-ICP-MS system is controlled using the Agilent ICP-MS MassHunter software, enabling automated analysis using multiple columns and methods, each run with the optimum mobile phase. Automating a sequence to run multiple speciation methods increases productivity and adds flexibility, while also allowing unattended overnight operation.

Figure 1. Agilent 1260 HPLC Multicolumn Thermostat with Quick Change valve head.

The work described here demonstrates the flexibility and reproducibility of an unattended HPLC-ICP-MS analysis using three separate methods to determine inorganic arsenic, methylmercury, and bromine and iodine species.

Experimental

An Agilent 1260 Infinity II HPLC system with a quaternary pump and InfinityLab Quick Change 4-column-selector valve head (p/n G4237A) was used. The quaternary pump can control up to four mobile phases, so no solvent selection valve was required for the three methods (four elements; seven species) used in this study. The HPLC was coupled to an Agilent 7900 ICP-MS using the Agilent LC connection kit (p/n G1833-65200), as shown in Figure 2. The HPLC methods used (see Table 1) were based on previous studies; see reference 3 for details.

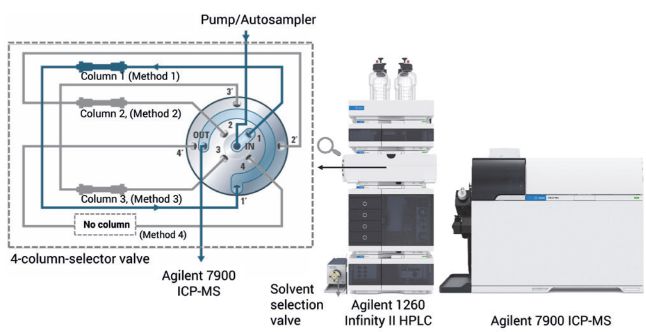


Figure 2. Agilent 1260 HPLC coupled to Agilent 7900 ICP-MS.

Table 1. Columns and mobile phases used for the three methods.

Method	Mobile Phase	Column
1. As(III) and As(V)	2.0 mM PBS/0.2 mM EDTA/10 mM CH_3COONa/3.0 mM $NaNO_3$/2% EtOH, pH 11.0 adjusted with NaOH	Anion exchange, Agilent p/n G3288-80000
2. CH_3Hg	2% MeOH/0.5g/L L-Cysteine, pH 2.3 adjusted with HCl	Zorbax Eclipse Plus C18, Agilent p/n 959941-902
3. Bromine and Iodine	5.0 mM NaH_2PO_4/15.0 mM Na_2SO_4/5.0 mM EDTA	Anion exchange, Agilent p/n G3268-80001

To demonstrate the applicability of automated column switching for routine unattended analysis, the three speciation methods were run with a different sequence order over three days, as shown in Figure 3. Method performance for linearity, recovery, and precision over the three days' analysis are provided in reference 3.

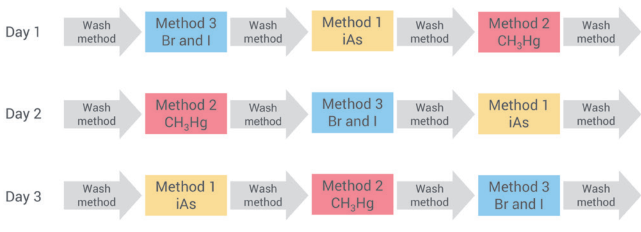
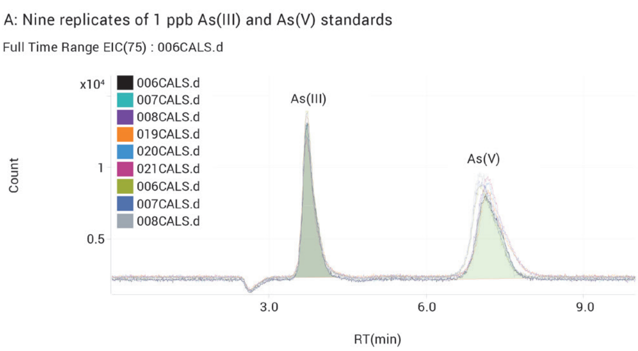
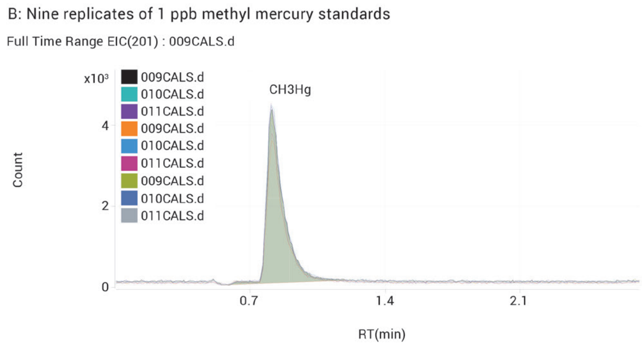


Figure 3. Experimental flowchart for the automated multi-method HPLC-ICP-MS sequences run over three days.

Within-day and between-day stability

Figure 4 shows overlaid chromatograms from three days' analysis of single or sub-ppb level calibration standards for iAs, CH_3Hg, IO_3^-, I^-, BrO_3^-, and Br^-. For each day's analysis, automated switching of the column and mobile phase ensured that all species were separated and measured using optimum HPLC conditions.





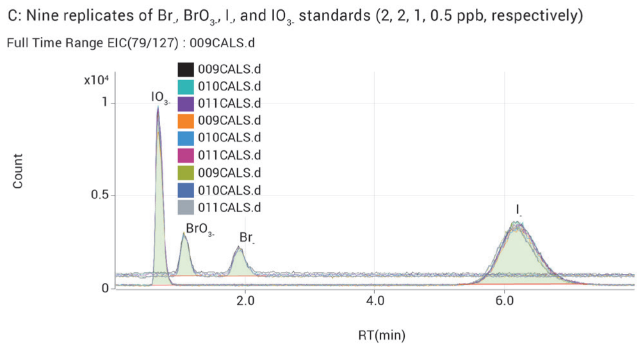


Figure 4. Overlaid chromatograms from replicate injections of calibration standards for A: As(III) and As(V), B: CH_3Hg, and C: Br and I species, n=9.

Conclusion

The use of HPLC-ICP-MS for elemental speciation can now be extended to allow multiple methods to be analyzed in a single unattended run. Automated analysis is supported using a Quick Change multicolumn switching valve head, combined with mobile phase selection using a solvent selection valve. The complete HPLC-ICP-MS system is set up and controlled from the ICP-MS MassHunter software, giving an easy-to-use integrated system for routine use, while also having the flexibility to support research applications.

References

1. Handbook of Hyphenated ICP-MS Applications, 2nd Edition, Agilent publication, 5989-9473EN
2. Wahlen, R. and Catterick, T., *Rapid Commun. Mass Spectrom*. **2004**; 18: 211–217
3. Automated Sequencing of Elemental Speciation Methods Using HPLC-ICP-MS with a Quick Change Valve Head, Agilent publication, 5994-2943EN

Congratulations to atomic spectrometry contest winners

In 2020, the most unusual year that most of us can remember, the North America Atomic Spectrometry team at Agilent helped to introduce some positive news by running an atomic spectrometry contest. The contest was simple: entrants were asked to propose a research project they wanted to complete and explain how a new Agilent ICP instrument would enable them to achieve their research goals. Three Grand Prizes of a one-year loan of an Agilent ICP instrument were on offer. The contest was popular, with about 140 ICP-MS-focused entries covering an eclectic mix of interesting and original topics. The judging panel of three Agilent ICP-MS experts reviewed each entry based on multiple criteria including how well the need for the ICP-MS was explained, the clarity of the research proposal, and the uniqueness of the intended work. Each of the entries had great potential, which resulted in robust debate among the judges; however, after a week-long discussion, two ICP-MS proposals emerged as clear winners:

- The Agilent 8900 ICP-MS/MS was awarded to Jana Mihalic from Johns Hopkins, Baltimore; A multi-disciplinary proposal encompassing research across multiple departments from life science, agriculture, and anthropology, united in the aim of improving the quality of life.
- The Agilent 7900 ICP-MS was awarded to Vicki Colvin from Brown University, Rhode Island; Development of living filters containing microorganisms for the removal of toxic contaminants in drinking water.

We thank all the participants for taking part in the contest and we look forward to seeing the outcomes from the winning proposals.

Latest Agilent ICP-MS publications

- **Application note:** Determination of Critical Elements in Foods in Accordance with US FDA EAM 4.7 ICP-MS Method, 5994-2839EN
- **Application note:** Automated Sequencing of Elemental Speciation Methods Using HPLC-ICP-MS with a Quick Change Valve Head, 5994-2943EN
- **Application note:** Analysis of Ultratrace Impurities in High Silicon Matrix Samples by ICP-QQQ, 5994-2890EN
- **Application brief:** Analysis of Trace Elements in Water Samples per ISO 17294-2, 5994-2803EN
- **Application brief:** Routine Analysis of Soil Samples using ICP-MS, 5994-2828EN
- **Guide:** Agilent 7800/7850/7900/8900 ICP-MS Supplies, 5991-7990EN

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