Agilent ICP-MS Journal

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7900 ICP-MS Special Issue

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The Measure of Confidence

Agilent Technologies

Introducing the New Agilent 7900 Quadrupole ICP-MS: Performance and Technology

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Agilent's new 7900 ICP-MS, introduced in January 2014 at the Winter Plasma Conference in Florida, redefines ICP-MS performance. With matrix tolerance extended to tens of percent total dissolved solids (TDS), a dynamic range up to 11 orders of magnitude, and major improvements in sensitivity and background noise, the range of applications open to ICP-MS has been dramatically increased.

A new version of ICP-MS MassHunter (see article in this issue), new customer training and support services, and innovations in cell technology, detector speed and productivity ensure that the 7900 marks a step change in both analytical capability and ease of use.

It is not unusual for a new analytical instrument to be described as ground-breaking or revolutionary, but it is rare for a new instrument to offer such a major improvement in performance that the description is truly justified.

The new Agilent 7900 ICP-MS is one of those rare cases, as it provides a factor of ten better performance than the outgoing Agilent 7700 Series, which was itself the performance benchmark for the industry:

- 10x higher matrix tolerance
- 10x wider dynamic range
- 10x better signal to noise
- 30x faster TRA acquisition speed

Matrix Tolerance

Agilent's innovative High Matrix Introduction (HMI) technology enables high matrix samples to be run routinely by ICP-MS, and has been field proven in thousands of 7500 and 7700 Series ICP-MS and 8800 ICP-QQQ instruments.

Figure 1. Agilent 7900 ICP-MS

For the Agilent 7900, the HMI technology has been redesigned to give the Ultra HMI (UHMI) option (shown in Figure 2), which provides a factor of 10 high matrix levels to be run (up to 25% TDS, compared to the 2.5% TDS limit for HMI). UHMI therefore offers matrix tolerance a factor of 100 higher than is typical for other ICP-MS systems.

Figure 2. UHMI on the 7900

The high matrix analysis capability of the 7900 with UHMI is illustrated in Figure 3, which shows the long-term stability for the recovery of a 100 ppb spike of several elements spiked into a 25% NaCl brine sample.

Just the fact that this extremely high matrix – too high even for some ICP-OES instruments to handle – can be measured on an ICP-MS is remarkable enough, but the good stability of the spike recoveries over 3.5 hours also demonstrates that the 7900 with UHMI enables reliable routine analysis of trace level analytes in this difficult sample matrix.

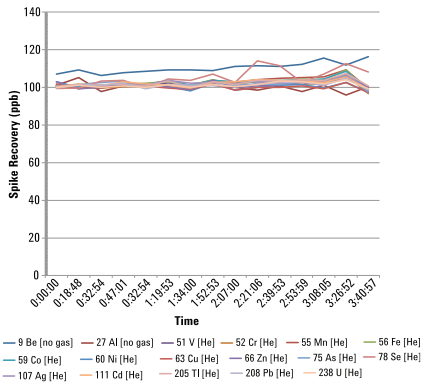

Figure 3. 3.5 hour stability for 100 ppb spike in 25% NaCl

The ability to run very high matrix samples using the same ICP-MS sample introduction hardware as for more typical, lower matrix samples offers a huge benefit in laboratory workload management. Not only does it allow practically any sample to be run using a single consistent instrument configuration, but it also allows the 7900 with UHMI to be used as a backup or replacement for a dedicated high matrix instrument.

Furthermore, the fact that UHMI operation is simply selected through the tune step in the batch means that high matrix sample batches can be run together with lower matrix samples in an unattended sequence.

Sensitivity, Background and Dynamic Range

Commercial quadrupole ICP-MS instruments use an ion (or "pulse") counting electron multiplier detector, which provides high sensitivity and low backgrounds. For higher signals, an analog mode is typically used, extending the dynamic range to around 9 orders of magnitude in the case of the 7700, meaning an upper limit of 100 ppm for a typical element with a background equivalent concentration (BEC) of 0.1 ppt.

Various approaches can be used to allow higher concentration analytes to be measured by ICP-MS, including attenuating the detector gain, or selectively reducing ion transmission using user-set lens, cell or quadrupole voltages. But these approaches require that the high concentration elements are known in advance, so the user can set appropriate acquisition conditions. This is one of the key reasons why many commercial labs retain a separate analytical technique for the determination of the major elements, even if trace element analysis is performed routinely using an ICP-MS.

The Agilent 7900 ICP-MS uses a new Orthogonal Detector System (ODS), shown in Figure 4, to provide a novel solution for the measurement of major elements in many sample types. With the ODS, the upper limit of the analog detector mode has been extended to 10^9 cps (10 GHz) giving up to 11 orders dynamic range for a background of 0.1cps. In practice this means that an element at 1000's of ppm in solution will be within the range of the detector, practically eliminating the time and cost of repeat analysis due to over-range results.

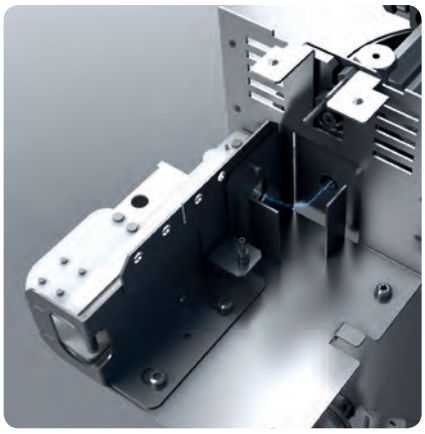

Figure 4. New ODS detector

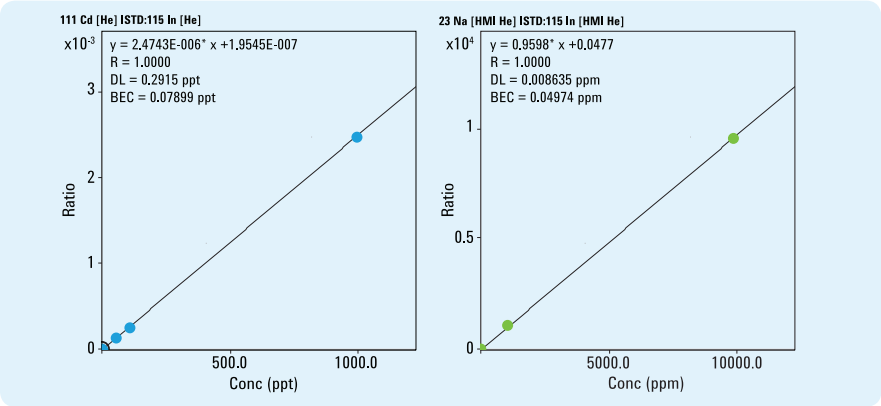

Figure 5. Cd and Na calibrations covering range from 0.1 ppt (Cd BEC) to 10,000 ppm (Na top standard)

The ODS, together with a redesigned interface, ion lens and expansion stage vacuum system, also reduces the instrumental background, typically to < 1cps, and increases sensitivity, ensuring superior measurement of trace level analytes.

The extended range of analysis is illustrated in the two calibration plots shown in Figure 5. On the left, the Cd calibration demonstrates the low background (Cd BEC is <0.1 ppt) and, on the right, the Na calibration demonstrates the extended upper measurement limit (top standard for Na is 10,000 ppm (1%).

Productivity Enhancements

ICP-MS, once considered a research technique, is increasingly favored by busy commercial laboratories who value its combination of speed, low detection limits and wide elemental coverage. Commercial contract laboratories typically run several hundred samples per day on each instrument, and so a reduction in measurement time has a big impact on the profitability and competitiveness of their analytical service.

Discrete Sampling (DS), where the sample fills a loop and is then injected into a carrier flow for transport to the nebulizer, is widely used to increase sample throughput. DS gives reduced uptake and rinse times and provides a better analytical duty cycle because data acquisition overlaps the uptake and stabilization steps for the next sample.

As an accessory for the 7900 ICP-MS, Agilent's third generation Integrated Sample Introduction System (ISIS 3), shown in Figure 6, provides highly efficient DS functionality, due to the very short distance between the 7-port injection valve and the nebulizer. This close-coupled layout, together with a high speed piston pump for sample uptake, ensures that sample delivery is optimized. The 7-port valve includes a port for on-line internal standard (ISTD) addition, and the inclusion of a 3-way valve to switch between ISTD and Tune solutions means that Startup and full autotuning functions are supported with ISIS 3.

Figure 6. New ISIS 3 accessory

In high-throughput laboratories where a saving of a few seconds per sample is significant, the Agilent 7900's new Octopole Reaction System (ORS^4) also contriuburtes to improved productivity. With a new gas controller providing cell gas switch times of as little as 2 seconds, a multi-mode (optimum cell gas mode for every analyte) acquisition for a regulated method such as EPA 6020 can now be completed in less then 60 seconds per sample.

More Information

To learn more about the 7900, including access to a brief video animation that provides an overview of our new instrument, go to:
agilent.com/chem/7900icpms

ICP-MS MassHunter 4.1: The Simplest, Most Powerful and Flexible ICP-MS Software Ever!

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[1]ICP-MS Software Product Manager, Agilent Technologies, US. [2]ICP-MS Software R&D Manager, Agilent Technologies, Japan

The latest version of ICP-MS MassHunter software (version 4.1) was developed in conjunction with the new Agilent 7900 quadrupole ICP-MS, and provides complete control of configuration, method setup, data acquisition, data processing and reporting.

ICP-MS MassHunter 4.1 is also compatible with the Agilent 7700 Series ICP-MS and 8800 ICP-QQQ instruments, with the required version being defined by the "option" selected when the software license or PC bundle is ordered (#001, #002, or #003 for the 7700, 8800 and 7900 respectively).

ICP-MS MassHunter 4.1 Workstation runs on the Windows 7 Professional (64bit) operating system. The base version includes all the functionality required for most typical applications. Advanced functions can be added as options.

Examples of advanced options include Time Resolved Analysis, Isotope Analysis, Intelligent Sequencing, Chromatographic Analysis, and User Access Control.

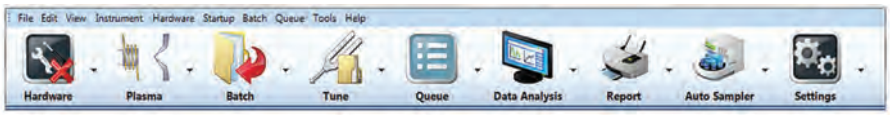

Figure 1. Top-level dashboard

New Features of ICP-MS MassHunter 4.1

ICP-MS MassHunter 4.1 maintains all the power and functionality of the previous software version, but with an emphasis on simpler, more intuitive and more flexible operation.

The new ICP-MS Top pane features a completely redesigned dashboard style user interface, which assigns all top level functions to large, easy to understand "gadgets" (Figure 1). The gadget icons are arranged in the order in which they are commonly accessed, and combine related functions into a single logical group, such as the plasma ignition parameters accessed through the Plasma gadget (Figure 2). Important but less frequently used functions are logically organized together under the "Settings" gadget.

Other new functions and features include:

- User configurable "Startup" and Autotune
- Standard QC Action on Failure functions
- Status Viewer shows quick overview of instrument hardware and operational status
- "Re-queue" permits a batch to be easily edited and resubmitted to the Queue for re-analysis
- Simplified, flexible User Tune and Global Tune functions
- Intelligent Method Wizard with interactive method creation
- Method Wizard with fully automated method creation*
- Support for ISIS-3*
- Support for Fast Transient Signal monitoring*
- Support for Ultra HMI (UHMI)*
- Simple, new drag and drop custom report template designer that does not require Excel
- Support for mobile devices via the Agilent ICP-MS Mobile app for iOS and Android mobile devices

** 7900 mainframe only*

Intelligent Method Wizard

The new Method Wizard (Figure 3) allows new users or experienced users who need to develop new methods to quickly create a well optimized method based on answering a few simple questions about the application, or introducing a typical sample.

The Wizard automatically selects the most suitable isotope for each analyte and internal standard, and selects appropriate integration times, plasma conditions and tune modes, based on your hardware configuration and method performance priorities – all with a few simple mouse clicks.

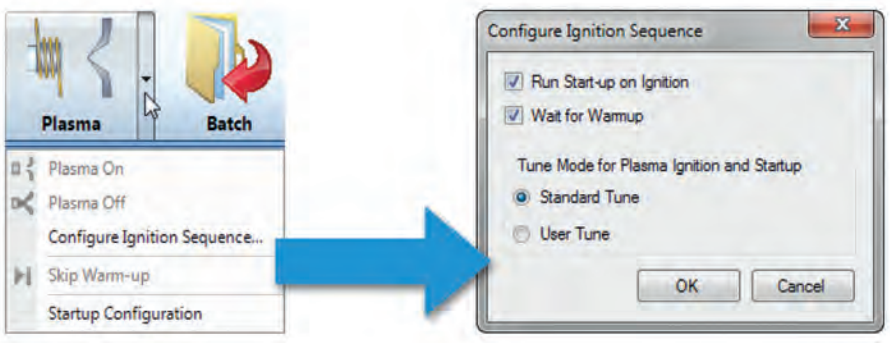

Figure 2. Plasma gadget pull down includes access to all plasma related functions. Simply double-clicking the gadget will ignite or extinguish the plasma.

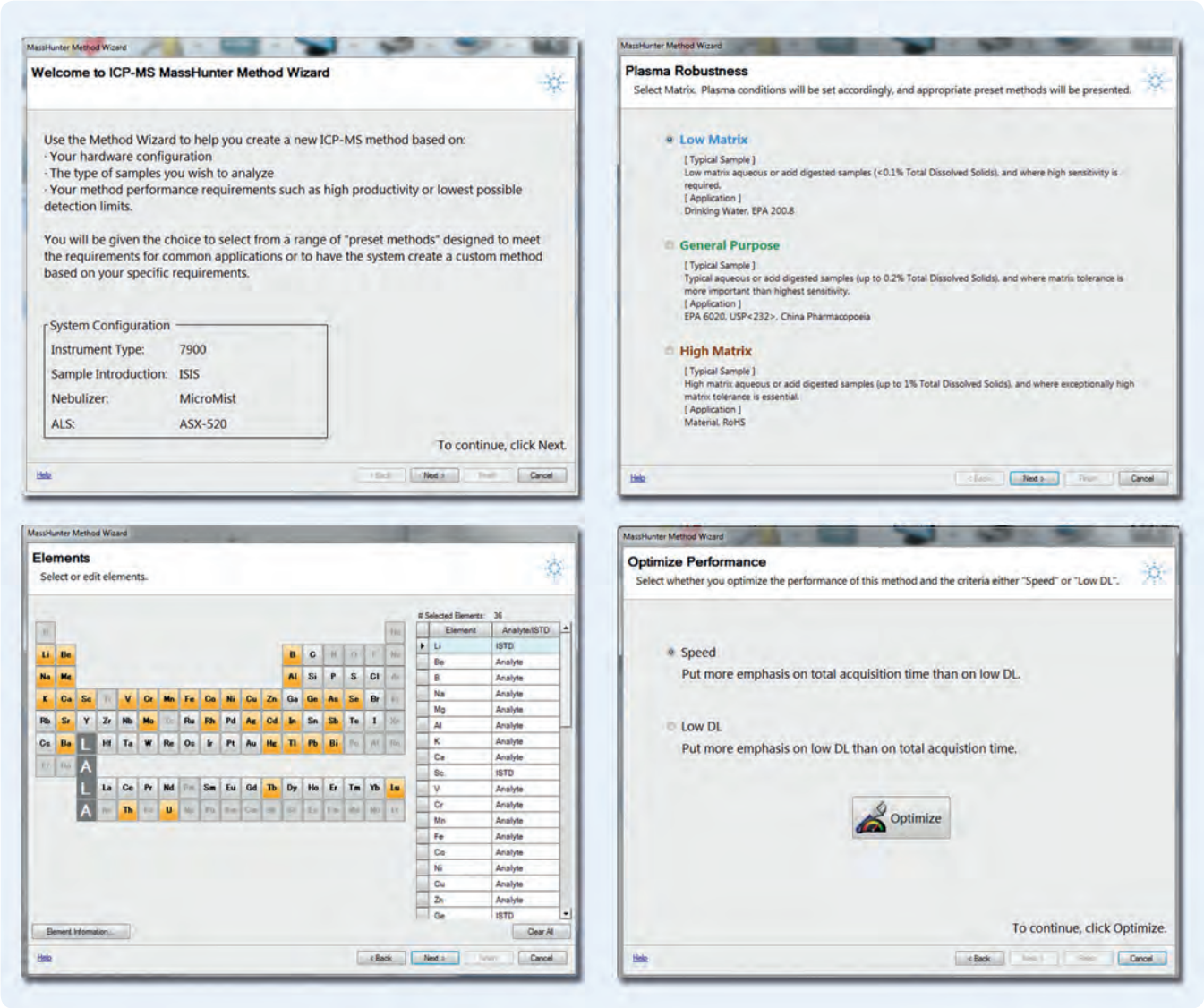

Figure 3. Method Wizard user interface and workflow

Agilent ICP-MS Mobile App

The new (early-release) ICP-MS Mobile app (Figure 4) is being made available free of charge to Agilent ICP-MS users with Apple iOS and Android Mobile devices, and will be downloadable from the Apple App store and Google Play store.

The ICP-MS Mobile app allows users to connect to their web-connected Agilent ICP-MS instrument running MassHunter 4.1, and view current instrument status including hardware, batch and queue status as well as any error conditions. The Plasma can be ignited or extinguished remotely, and the Queue can be paused and restarted. Plotting functions permit remote viewing of MassHunter internal standard or quality control charts for the currently running batch.

Figure 4. ICP-MS Mobile app for Agilent ICP-MS instruments running MassHunter 4.1.

Learn More

For more information on what ICP-MS MassHunter 4.1 can provide to your laboratory, go to **agilent.com/chem/icpmsMassHunter**

UHMI Technology Enables the Analysis of Very High Salt Matrix Samples by Agilent 7900 ICP-MS

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[1]Eurofins Analytico, NL
[2]Agilent Technologies, UK

The development of new ICP-MS instrumentation and technologies at Agilent is strongly influenced by user requirements. As well as user input being considered during the specification of new instruments, Agilent also works with partner labs to "road-test" upcoming hardware and software developments in a real-world production environment.

One Agilent ICP-MS partner lab is Eurofins Analytico in Barneveld, Netherlands, where the range of difficult sample types routinely analyzed ensures that new ICP-MS systems can be thoroughly tested prior to general release. The pre-release testing of the new Agilent 7900 ICP-MS at Eurofins focused on the enhanced high matrix capability provided by the Ultra High Matrix Introduction (UHMI) system, and included the analysis of very high salt solutions, up to 25% NaCl.

The matrix tolerance of ICP-MS instruments has traditionally been limited by two factors:

- Reduced analyte signal due to ionization suppression
- Long-term drift due to clogging of the interface cones

Signal suppression occurs when a high matrix load absorbs so much of the plasma's energy that there is insufficient residual energy to ionize the analyte atoms. This is a particular problem for poorly ionized analytes (As, Se, Cd, Hg, etc.) in samples that contain high levels of easily ionized elements (Na, K, etc.), which contribute a large population of free electrons that suppress the ionization of poorly ionized analytes.

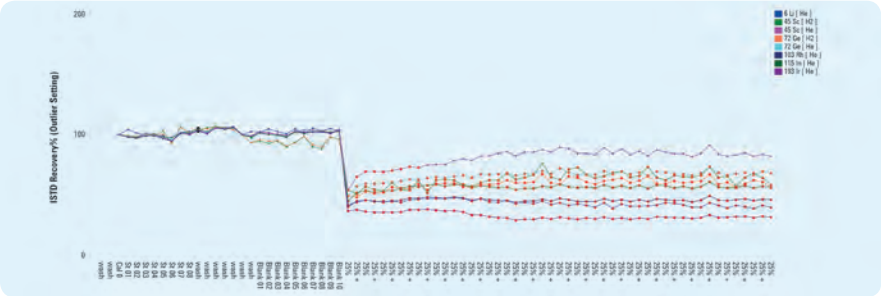

Figure 1. Internal Standard signal in 50 samples of 25% NaCl matrix

Similarly, clogging of the interface cone orifices occurs when the plasma has insufficient energy to completely decompose the sample matrix, leading to the residual matrix condensing (depositing) on the cones. The matrix tolerance of an ICP-MS is improved by operating the plasma under robust conditions (low CeO/Ce ratio), and Agilent's HMI technology extends matrix tolerance to 2.5% total dissolved solids (TDS) by diluting the sample aerosol as it passes from the spray chamber to the torch, so reducing the matrix and aerosol load.

The Agilent 7900's optional UHMI (described in the 7900 introduction article in this issue) extends the matrix tolerance by up to a further factor of ten, allowing samples up to 25% TDS to be measured. UHMI is a dilution technology, but it avoids the downsides of conventional liquid dilution (time, cost of reagents, potential for errors and contamination, and waste disposal costs) by diluting the sample aerosol using clean, argon gas. This improves plasma robustness, and the dilution factor can easily be calibrated and reproduced reliably by automatic adjustment of the nebulizer and dilution gas flows.

Figure 1 shows the internal standard signals for an aqueous calibration followed by fifty 25% NaCl solutions, run at UHMI-100. While the signal level in the NaCl matrix is reduced by about ½ compared to the aqueous standards (mainly due to sample transport, nebulization and spray chamber effects), the ISTD signals are all reduced by about the same amount (no mass bias) and remain stable throughout the 4 hours of 25% NaCl analysis, confirming that no interface clogging occurred.

This level of matrix tolerance is unprecedented for ICP-MS, but the most remarkable aspect of the performance of the new 7900 with UHMI is in the consistency of results in widely variable sample matrices.

Figure 2 shows the measurement of a 50 ppb trace element spike (25 ppb for As, 1 ppb for Hg) in a NaCl matrix, calibrated against non-matrix matched standards. The same spike was added to samples containing a range of matrix levels from zero to 25% NaCl. The quantitative results for the spiked elements are accurate (the actual spike concentration is shown as the first point) and consistent for all matrix levels, an astonishing achievement. Even the Hg spike at 1 ppb was measured accurately (average recovery of 93%) and consistently (RSD of 6.3% between the eight different NaCl matrix levels).

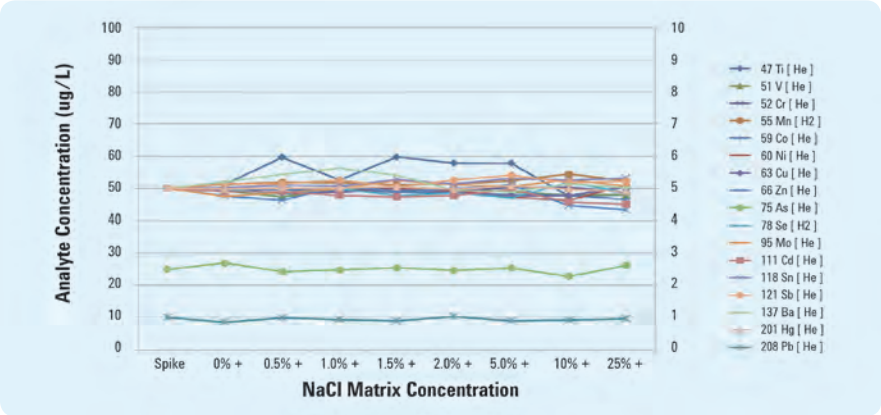

Figure 2. Spike recovery in variable NaCl matrix

Single Particle Analysis of Nanomaterials using Agilent 7900 ICP-MS

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Agilent Technologies, France

Introduction

There is an urgent need to develop analytical methods that are suitable for the evaluation of nanoparticles (NPs) because of concerns about their safety and impact on health.

An interesting approach for the characterization of NPs has been developed by Degueldre et al. using ICP-MS [1]. If samples containing NPs are introduced at a low number of particles in solution, analysis in continuous time resolved analysis (TRA) mode makes it possible to measure the signal for a single particle as it is vaporized and atomized in the plasma. Each measured data point can then be correlated to the size and mass fraction of a unique NP. This method is called Single Particle (SP-) ICP-MS analysis.

Experimental

Standard and Sample Prep

Silver NP solutions with different NP sizes were purchased from Sigma Aldrich.

The NP samples were diluted with water in polypropylene vials. Sample dilution was performed on the day of the analysis in order to avoid sample degradation. Before dilution of the samples and prior to their analysis, all solutions were placed in an ultrasonic bath for 10 mins to ensure sample homogeneity.

Instrumentation

Measurements of NPs were performed using the Agilent 7900 ICP-MS. The samples were introduced directly into the ICP-MS system using the standard peristaltic pump and ASX-520 autosampler. Analyses were performed in TRA mode. The general settings of the 7900 system are detailed in Table 1.

Plasma power	1550 W
Carrier gas	1.05 L/min
Make up gas	0.10 L/min
Sample depth	8 mm
Integration time	3 ms or 100 µs (see text)
Acquisition time	60 s

Table 1. 7900 ICP-MS operating parameters

Data Analysis

A dedicated spread sheet developed by the National Institute of Food Safety in the Netherlands (RIKILT) was used for data conversion. The SP-ICP-MS analysis and custom spreadsheet calculations are able to determine the number of particles present in the sample, their size distribution, the median size of the NPs population and the mass concentration for the element.

Results and Discussion

Figure 1 shows a TRA acquisition of a solution of 40 nm Ag NPs measured using the 7900 ICP-MS with 3 ms integration time.

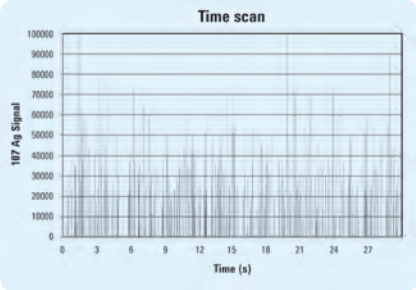


Figure 1. Measurement of 40 nm silver NPs acquired using SP-ICP-MS mode

From this raw data, the background signal was eliminated by the custom spreadsheet, and the remaining intensities were converted into particle size to give the distribution pattern shown in Figure 2.

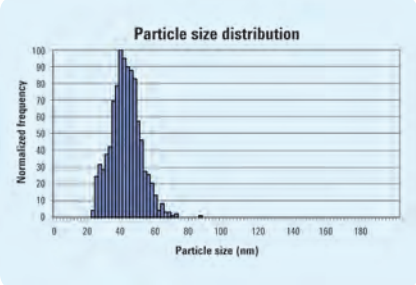


Figure 2. Particle size distribution for a 40 nm Ag NP

From the size distribution plot, the median size was calculated at 40 nm. This measured size is in good agreement with the 40 ± 4 nm value provided by the supplier, which was determined using Transmission Electron Microscopy (TEM) analysis. The particle number in the diluted sample was estimated to be 3.4 x 10^7 particles/L and the mass concentration for Ag was 13 ng/L.

Summary results for the analysis of three different Ag NP sizes (Table 2) confirm that in all cases the 7900 results agree with the specification values provided by the NP manufacturer's TEM values.

Supplier's specification (nm)	40 ± 4	60 ± 4	100 ± 8
Experimental size (nm)	40	55	103
Number of particles (particles/L)	3.4 x 10^7	1.5 x 10^7	5.2 x 10^6
Element concentration (ng/L)	13	14	424

Table 2. Results for the analysis of Ag NPs

Future Developments

The new 7900 ICP-MS allows the measurement of NPs with a fast integration time of 100 µs. This enables the measurement of individual NP peak signals (Figure 3).

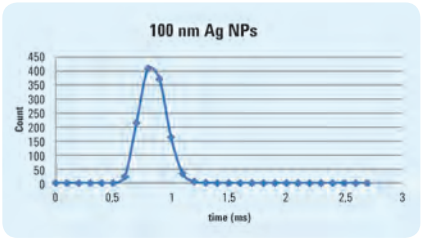


Figure 3. Measurement of one single 100 nm Ag NP with 100 µs integration

Conclusions

The Agilent 7900 ICP-MS method has been successfully applied to the analysis of silver NPs providing size distribution, median size, number of particles and the elemental concentration of a given NP sample.

Acknowledgements

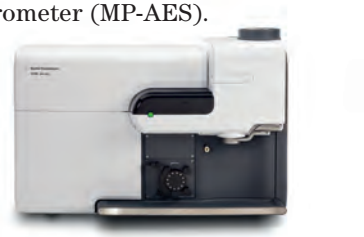
We would like to acknowledge the contribution of RIKILT, who provided the Microsoft® Excel® worksheet that was used to convert the raw SP-ICP-MS analysis data.

Reference

1. Degueldre S., Favarger P.-Y., Bitea C., (2004) Anal. Chim. Acta, 518: 137-142

Agilent Strengthens Spectroscopy Portfolio with New ICP-MS and MP-AES

As well as the launch of the 7900 ICP-MS featured in this Journal special issue, the 2014 Winter Plasma Conference (WPC) also saw the introduction of the Agilent 4200 Microwave Plasma-Atomic Emission Spectrometer (MP-AES).



Agilent's new 4200 MP-AES

Both new instruments feature enhancements in performance and functionality that make them ideal for a wider range of challenging applications and more accessible to a broader spectrum of laboratory personnel.

"At Agilent we continue to introduce 'new to market' technologies across our platforms, enabling our customers to expand their labs' capabilities and simplify their workflows," said Philip Binns, Agilent Vice President of spectroscopy products. "The introduction of the 4200 MP-AES and 7900 ICP-MS further demonstrates our commitment to providing our customers with the best tools to meet their application needs, and confirms our position as the leader in elemental analysis."

Find out more about the 4200 at: **agilent.com/chem/RunsOnAir**

Learn All About the New 7900 ICP-MS at Our Free Webinar

Title: Raise Your Expectations: Learn How the New Agilent 7900 ICP-MS Redefines ICP-MS Performance
Date: February 26, 2014 at 8:00am PST/ 11:00am EST/4:00pm GMT/ 5:00pm CET
Presenter: Ed McCurdy, Agilent ICP-MS Product Marketing

Register now at:
spectroscopyonline.com/performance

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5991-3903EN

Agilent's Headlines from WPC 2014

On Monday, January 6th, Agilent unveiled two exciting new products at the Winter Conference on Plasma Spectrochemistry, Amelia Island, Florida, US. The 7900 ICP-MS and 4200 MP-AES were introduced to several hundred thought leaders in the fields of atomic spectroscopy and elemental analysis.

"We have received many positive comments on the performance gains of the 7900 and our newly redesigned MassHunter software", said Ken Suzuki, Agilent ICP-MS Marketing Director. "The 7900's ultra-high matrix tolerance, wider dynamic range and improved signal-to-noise performance appears to be resonating with users in key markets, including customers in demanding, high throughput laboratories performing routine environmental and clinical analysis." The 7900 Lunch Seminar was full to capacity as delegates gathered to hear details about the new ICP-MS.

Additionally, interest in the 8800 ICP-QQQ continues to grow rapidly as a capacity crowd attended the 8800 Lunch Seminar. Also at the WPC, Agilent hosted the first 8800 User Meeting, giving users an opportunity to share their experiences of this pioneering technique.

"We had an impressive turnout of more than 40 attendees at the 4200 MP-AES Lunch Seminar on Tuesday, all keen to learn about the new instrument and its capabilities. Feedback was extremely positive. Attendees were impressed to see advancements in performance offered by the 4200 while retaining the safety and cost-effective elemental analysis inherent in the MP-AES technique," said Agilent's Keith Bratchford, General Manager, Optical Spectroscopy Products.

Agilent specialists presented a total of 16 oral and poster presentations throughout the conference scientific sessions. There was also a "sell-out" Customer Appreciation evening event for users of Agilent products that was enjoyed by all! For more information on all of Agilent's spectroscopy products, please visit **agilent.com/chem/atomic**

Agilent ICP-MS Publications

To view and download the latest ICP-MS literature, go to **www.agilent.com/chem/icpms** and look under **"Literature Library"**

- **Brochure:** Agilent 7900 ICP-MS: Raise Your Expectations with the Next Generation ICP-MS, 5991-3719EN
- **Application note:** Speciation of Inorganic Arsenic in Baby Rice Cereals Using HPLC-ICP-MS, 5991-2568EN
- **Application note:** Method Validation for 16 Trace Element Determinations in Polypropylene and High Density Polyethylene by ICP-MS, 5991-3536EN
- **Application note:** Multi-element analysis of crude oil samples by ICP-MS, 5991-3538EN

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