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Manufacturing Step-up to Meet Strong Early Orders of 7700 Series ICP-MS

Don Potter
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Since its introduction in July 2009, the 7700 Series has generated a great deal of interest and has enjoyed unprecedented demand; over 140 orders have been placed for the 7700 Series between July and October. In addition, a number of 7500 Series orders have also been placed to fulfill existing purchase contracts.

Building on the market leadership of the 7500 Series, the rapid acceptance of the 7700 Series confirms that high performance combined with ruggedness and ease of use are the most important requirements for ICP-MS buyers.

To satisfy the strong demand for the 7700, the ICP-MS manufacturing team is working at near-full capacity in their state-of-the-art production and test facilities at Agilent’s Tokyo Analytical Division (TAD) in Japan (Figure 1).

Milestone reached: First shipment of 7700 ICP-MS leaves Tokyo

The TAD team took some time out in late July to celebrate the shipment of the first 7700 ICP-MS unit destined for a customer lab (Figure 2).

The 7700s mainframe shown was on its way to support the development of new pharmaceuticals at Schering Plough Research Institute (SPRI) in Union, New Jersey, USA.

Figure 1. Agilent 7700 (and final 7500) Series ICP-MS assembly and final test facility in Tokyo, Japan

Figure 2. Agilent’s Tokyo based ICP-MS team celebrate the shipment of the first 7700 ICP-MS to Schering Plough Research Institute (SPRI) in the U.S.
Accreditation signifies that an environmental testing lab can produce precise, reliable results for its clients – and is often a prerequisite for generating revenue. The management team at Envirolab Services was delighted when they required only two months to gain full accreditation for analyses on their new Agilent 7500 Series ICP-MS – a rare feat that even the assessor considered impossible prior to his visit. With an Octopole Reaction System (ORS) that removes molecular interferences (that might compromise reporting limits for key elements), the instrument continues to deliver accurate results and helps the lab to expand its business.

The Company
Envirolab Services is a high-tech scientific laboratory that serves the environmental sector. Although the lab was founded by Managing Director Tania Notaras only four years ago, it has rapidly grown to about 40 employees and is now a major supplier of environmental analytical services in New South Wales. The lab runs thousands of samples per month in its two-story suburban premises, a facility purpose-built to world class standards in 2008.

In-house ICP-MS for Better Customer Service
Envirolab Services originally used ICP-OES and AA instruments for less demanding analyses. But three years ago, when customers needed results for trace-level metals, the lab was forced to send out samples to a third party for analysis using the more sensitive ICP-MS technique. Turn-around times were long, and eventually the team at Envirolab decided to purchase an in-house ICP-MS to provide better service to clients.

Giovanni Agosti, the lab’s highly experienced Technical Development Manager, explained that they chose the Agilent 7500ce ICP-MS after sending a wide range of difficult test samples to a number of vendors. Agilent provided the best results. Notaras commented that “In making the choice, we also looked at robustness and reliability, and considered the good relationship we have with Agilent. From day one, Agilent has always looked after us.”

Rapid Accreditation
The lab quickly achieved accreditation from the Australian-based and internationally recognized National Association of Testing Authorities (NATA). Agosti remarked, “After the instrument was installed, we were able to get trained and complete validation of all our methods in about two months. That was a great achievement for us. We ran almost 1000 samples during the validation period. Gaining accreditation in two months meant we were able to run paying samples in a very short time.”

Notaras added, “Two months is very quick. The assessor came out but was not planning to give his signature to the accreditation. He did not think it possible to train and validate in only two months. He was pleasantly surprised by our technical expertise and the validation results, and immediately granted accreditation with no restrictions. That is very rare.”

More Accurate Results for Difficult Samples
Seawater is one of Envirolab’s most challenging matrices for ICP-MS analysis for trace metals, in part because it produces polyatomic interferences for metals like arsenic and selenium. The Agilent 7500 Series ICP-MS systems are designed to accurately measure low levels of these metals in high-matrix samples. The unique ORS is a highly efficient collision/reaction cell (CRC) that removes the majority of polyatomic interferences.

Agosti explained that he had used ICP-MS instruments previously, but the Agilent system was the first experience he had with a collision cell. When asked if he thought the ORS improved the analysis of difficult elements/sample matrices, he exclaimed, “Definitely! This instrument is quite amazing. After all the validation using real samples, I know the results I’m getting in the end are the right results. I am confident in this instrument.”

Growing Business
Notaras and Agosti described how the 7500ce has allowed them to take on new types of work related to landfills and groundwater, which has increased their revenue in both ICP-MS and other analyses. “We’re running a lot more samples now, so the instrument is paying itself off. We have been able to build up our business with this instrument,” said Notaras.

When asked if there were any surprises with the instrument, Agosti replied, “I have been positively surprised at how well the collision cell works. We can now accurately analyze arsenic and selenium at low levels in complex matrices.”

Notaras added, “We often get better results than our competitors. Some clients want to compare, so they split samples. Because of the ORS, we always come out ahead.”

www.agilent.com/chem/icpms
Characterization of Nanoparticulates using Field Flow Fractionation-ICP-MS

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Introduction
Recent interest in the analysis of nanoparticulates in the environment has largely centered on the potential impacts of manmade particles. However, natural nanoparticles are ubiquitous in aquatic environments. The Great Salt Lake (GSL) is a useful field laboratory in which to test methods for analysis of nanoparticles. Being a hypersaline terminal lake, the GSL has high concentrations of dissolved solids and it is super-saturated with respect to many phases containing major and trace elements. The flow of higher density water (1160 kg/m³) from the north arm to the less dense south arm (1100 kg/m³), yields a persistent layered system with an oxic brine upper layer and an anoxic brine deeper layer, yielding strongly contrasting geochemical conditions over a 9 m vertical distance.

The GSL hosts a large avian population that is potentially affected by trace metals and metalloids such as Se, As and Hg, and natural nanoparticulates may act as a gateway for introducing trace elements into the food chain of the lake. Laboratory experiments have proven that mineral nanoparticles may be directly ingested by mixotrophic phytoplankton, which constitute the base of the GSL food chain.

This work examines the size distribution and elemental composition of nanoparticulates from oxic and anoxic samples in the GSL, with the working hypothesis that the size and elemental distributions differ strongly among the oxic and anoxic layers.

Methods
Flow-field flow fractionation (FIFFF) is a minimally invasive technique that uses hydrodynamic principles and a thin rectangular channel to separate particles by size. Asymmetric flow-field flow fractionation (AF4) uses a trapezoidal channel which reduces sample dilution relative to symmetric channels and provides exceptional nanoparticle size resolution. To determine the trace elemental distribution in fractionated particulates, the AF4 was coupled with an Agilent 7500ce ICP-MS with an Octopole Reaction System (ORS) cell. The 7500ce was calibrated using a mixed standard (plus 1 mg/L Cs internal standard) added to the AF4 carrier solution. Adding an internal standard to the carrier rather than the sample avoids discrepancies due to variations in solution flow to the detector during fractionation. To increase the sensitivity of trace element analysis, the ORS was employed.

Figure 1. Fractograms of GSL deep brine water sample (site 3510 at 7.5 m in depth; sample collected on 10/26/07). Size range: 0.9 - 7.5 nm.
elements such as Se and As, methanol was added to the carrier solution (3% v:v). A total of 16 elements (Al, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Mo, Sb, Au, Hg, Pb, U, Cs) were measured by AF4-ICP-MS.

**Results**

Trace elements such as Mn, Pb, Zn, Cu, U, Ni and Co were associated with nanoparticles in the 1 - 2 nm size range (Figure 1, blue and green rectangles), as well as the 5 - 7.5 nm size range (Figure 1, red rectangles). These nanoparticle size fractions and elemental associations with Mn, Pb, Zn, Cu, U, Ni and Co were observed in the deep brine sample (Figure 1), but also in the shallow brine. Figure 1 shows a continuum in signal intensities for these elements, increasing with increasing depth (increasing degree of O₂ depletion and sulfide presence), suggesting the formation of sulfides. Aluminum showed a peak only in the 5 - 7.5 nm size range (not in the 1 - 2 nm size range) with this peak being somewhat larger in the deep brine samples relative to the shallow brine samples (Figure 1). Mercury was significant in the 1 - 2 nm size in the anoxic deep brine sample (Figure 1), but was not present in the shallow brine samples. In contrast, arsenic was present in the 1 - 2 nm size range, but was most significant at shallow depth and much less significant in deeper samples. It should be noted that the raw signal intensities in Figure 1 are not corrected for influences of ionization efficiency; these influences are corrected in the integrated elemental masses described below.

Figure 2 shows concentrations (per L solution) of particulate-associated elements as a function of depth and size range. For >450 nm microparticles, the concentrations of nearly all associated elements increased with depth (Fig. 2 top). The same observation held for the 10 – 250 nm nanoparticles (except Co, As, and Sb) (Fig. 2 middle). In contrast to the >10 nm size range, the majority of the trace element concentrations in the 0.9 - 7.5 nm size range did not increase with depth (Fig. 2 bottom), except for Al, Mo and Hg. The majority of trace element mass associated with particulates was in the >450 nm size range, as shown by comparing the measured concentrations (per L solution) between the 3 particulate size ranges (Figure 2). The trace element mass associated with the 10 - 250 nm size range was much lower (multiple orders of magnitude for some elements) relative to the > 450 nm size range. The same was true for the 0.9 - 7.5 nm size range, but with greater concentrations of Ni, Zn, Au and Pb in the 0.9 - 7.5 nm size range relative to the 10 – 250 nm size range.

**Conclusions**

The results demonstrate that AF4-ICP-MS with ORS is a simple but powerful tool for determining the trace elemental composition of nanoparticulates in complex aqueous environments. Associating nanoparticle size fractions and elemental composition between the shallow oxic and deep anoxic brine layers of the Great Salt Lake is expected to help understand the pathways of toxic and other elements into the food chain.
During the past 5-6 decades, there had been many advances in Isotope Dilution Mass Spectrometry. Its wider acceptance for routine use, however, has been hindered by technical complexities, especially for accurate multi-species analysis. One group of enabling metrologies and tools called Isotope Dilution Enabled Automation overcomes many of these obstacles.

The IDEA-MS concept includes a unique Calibration Curve Free Quantitation (CCFQ™ *) package incorporating a mathematical foundation that utilizes additional degrees of freedom to achieve unprecedented accuracy, multi-analyte, multi-species capability and shorter, simplified application. The CCFQ software also includes a Spike-to-Analyte optimization tool that allows the user to select the appropriate error propagation factor and corresponding amount of spike to help deliver or maintain the desired level of accuracy for each element. For sample processing, the key automation tool is the i-Spike™ sorbent-cartridge that is used for in-flow spiking and equilibration, facilitating rapid analytical cycles to address the demands of high-throughput workflows.

CCFQ-SIDMS is now accepted worldwide as the most accurate metrology for delivering definitive and legally-defensible multi-species results. Recognizing the utility of IDMS/SIDMS, the US EPA has published them, after global scientific scrutiny and stellar performance, as a national Resource Conservation and Recovery Act (RCRA) method, Method 6800, which is dedicated to the ID metrology. Method 6800 is now accepted as the gold standard for speciation.

AIT’s 3Hg-SPC™ Kit, for example, enables simultaneous definitive measurement of 3 mercury species, while using the same extracted sample for Non-Extractable (NE) Hg, including metallic mercury (Hg⁰). In the near future, AIT will expand the method to include Hg-species among the NE group (Figure 1). This particular application is of great interest to oil refiners because accurate Hg-species information enables process optimization to minimize catalyst poisoning in the refining process, thus maximizing profits.

The same method and 3Hg-SPC Kit is utilized by Centers for Disease Control to accurately test blood samples for high-priority industrial metals. Among these, accurate speciation of Hg is a critical requirement (Figure 2).

The problems for which IDEA metrology is providing solutions range from environmental monitoring to biochemi-marker™ discovery and quantitative measurements in biological samples. Biochemi-markers will soon help physicians confirm diagnoses of diseases earlier and more accurately. With IDEA (Figure 3), scientists are generating previously unavailable information about diseases such as autism and cancer.

IDEA, CCFQ, i-Spike, SPC and biochemi-marker are trademarks of Applied Isotope Technologies, Inc.
Well Equipped ICP-MS User Training Lab Opens in Americas

Lisa Trainor
ICP-MS Sales and Support, USA

Opened in Sept 2009, the ICP-MS Training Facility was established to provide a new approach to ICP-MS training for analysts who are either new to the technique, unfamiliar with the Agilent system or who require additional training.

Training for Analysts Based in the Americas and Beyond
Located in the historic district of St Charles in Missouri, USA, the ICP-MS Training Facility is only 16 km (10 miles) from St Louis’ International Airport. With such easy communication links, analysts from outside of the Americas are welcome to join any of the range of training courses. Currently, there are 5 different ICP-MS training courses offered at the ICP-MS Training Facility.

<table>
<thead>
<tr>
<th>Course Title</th>
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<td>Environmental Applications</td>
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New Teaching Approach Enhances Learning
The advanced training facilities have been carefully designed so that the two Agilent 7500 ICP-MS systems and attendee desks and seating are located in the same room (Figure 1). Use is also made of two 100 in. screens that are used to deliver training materials and to relay the ICP-MS software suite. Wireless keyboards are also utilized to give students the chance to operate the ICP-MS from their seats. And with a maximum of 10 students per course, everyone is sure to have ample occasion for hands-on learning and the time to ask the course-leader about specific points. Feedback from the inaugural course, led by Dr. Tom Rettburg, was overwhelmingly positive (Figure 2).

Expert Team of Trainers
The team of course leaders combine training and development experience with many years hands-on operation of ICP-MS systems in different industries. With nearly 20 years direct experience with ICP-MS, Tom Rettberg’s areas of applications specialization include EPA environmental analysis, high purity semicon materials analysis, solid sample analysis, organic materials analysis, isotope and earth science applications. Thong Quang Ta has been delivering applications support, consulting, and training for Agilent ICP-MS users in North America since 2005 with previous experience in the semiconductor, environmental and clinical applications. Philip Schmidt, who created the training facility, brings specialized applications experience from silicon wafer manufacturing together with general knowledge built up over 8 years as an Applications Engineer. The founder of the ICP-MS Training Facility and ICP-MS expert, Abe Gutierrez, leads classes in Spanish.

To find out more on the courses held at the ICP-MS Training Facility, contact Lisa Trainor: lisa@agilentpcms.com or tel: US 972-977-4684

Figure 1. Combining laboratory and lecture facilities in cutting edge training premises specifically for Agilent 7500 ICP-MS users.

Figure 2. Participants of the first training course held at the Agilent Training Facility in September 2009. Trainer Dr Tom Rettberg is on the right of the photo.
ICP-MS, thereby making such qualifier ions available for evaluation of data quality.

In the second Webinar, they will present data demonstrating the use of qualifier ions to improve ICP-MS data quality in complex sample matrices, by confirming the measured result from the primary isotopes.

Title: Part 1: He Mode Interference Removal for Spectral Clarity and Multi-Isotope Confirmation

Date: Wed, December 16, 2009
Time: 08:00 PT (USA)
11:00 ET (USA)
16:00 GMT (UK)
17:00 CET (Central Europe)

Part 2: Using Qualifier Isotopes to Validate Multi-Element ICP-MS Data in Complex Sample Matrices

Date: Wed, February 10, 2010
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Agilent’s Ed McCurdy, ICP-MS Product Marketing, and Steve Wilbur, ICP-MS Environmental Marketing, will explore the capability of the 7700x ORS3 helium (He) mode to remove the polyatomic interferences from all isotopes of the target elements, so allowing the user to measure additional isotopes for many analytes. This approach is widely used in organic mass spectrometry, where these secondary ions are referred to as 'qualifier' ions.

In the first Webinar of this series, Ed and Steve will assess the fundamental performance of He mode in removing spectral interferences in ICP-MS, thereby making such qualifier ions available for evaluation of data quality.

Agilent has an on-going program of 7700 Series ICP-MS seminars and User Group Meetings. Be sure to check with your local Agilent office or www.agilent.com/chem/events web page for details of events in your area. Example events include:

Agilent Environmental Seminars, Jan 19 and 21, 2010, Spain
ICP-MS User Meeting, Jan 27-28, 2010, Finland
ICP-MS User Meeting, Feb 11, 2010, Denmark

To register go to www.spectroscopynow.com and look for the “Webinars” link.

Or use the direct link: www.spectroscopynow.com/coi/cda/detail.cda?id=22519&type=Feature&chId=1&page=1

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