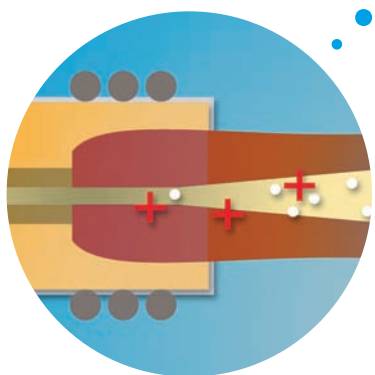


# Agilent ICP-MS Journal

August 2008 – Issue 35



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**Agilent Technologies**

# Comparison of Cell Modes for the Measurement of Interfered Analytes in a Complex Matrix

Ed McCurdy, Glenn Woods

Agilent Technologies UK Ltd

## Introduction

Much of the published work relating to collision/reaction cell (CRC) technology has focused on single-element reaction process methods, most of which are of little relevance to more routine laboratories, which use ICP-MS as a commercial tool for multi-element analysis in complex and variable matrices. The 3 most important potential limitations of any cell method are rarely addressed in the published work, namely:

1. Failure to remove unreactive interferences
2. Creation of new, cell-formed reaction product ions, and
3. Loss of analyte sensitivity by reaction.

The comparative tests were designed to investigate the true effect of these potential issues, and to answer the question of which mode, collision or reaction, is more effective for multi-element analysis in a complex sample matrix. The complex matrices comprised 5% HNO<sub>3</sub>, 5% HCl, 1% H<sub>2</sub>SO<sub>4</sub>, 1% Acetic Acid, 200ppm Na, 200ppm Ca and 500ppm P. Each of these matrix components was prepared individually as a single matrix solution, and a final, combined mixed matrix solution sample was prepared, containing all of the individual matrix components.

## Analytes & Matrix Components

A full list of the elements measured and the isotopes used is given on page 35 of the Agilent ICP-MS Primer [1] along with a list of the potential polyatomic interferences which might arise in the complex matrix in which the analytes were to be measured. At least 1 polyatomic interference could occur on every isotope of every element in the mass range 45 to 80, also the various polyatomics which overlap each analyte mass typically arise from different matrix components.

## Tuning and Data Acquisition

The 7500cx was tuned in each of the 3 modes (No-Gas, He and H<sub>2</sub>), using the standard Agilent-recommended robust tuning conditions. The sample sequence was prepared, with a 2-point multi-element external calibration, stabilized with 0.1% HNO<sub>3</sub> (the zero matrix calibration reference). All the remaining sample matrices were then measured against this external calibration. No internal standards were added. Each sample was measured in all 3 gas modes, from a single visit to each vial. The standard Agilent multi-step rinse program was used, in conjunction with pre-emptive rinse.

## Results and Discussion

The measured or apparent concentration of each analyte was

calculated against the calibration in 0.1% HNO<sub>3</sub>, and the resulting concentrations in each cell gas mode were plotted against the matrix name. This gave a series of comparison plots, showing the background equivalent concentration (BEC) plotted against the matrix, for each analyte in each gas mode. Since all the samples in these plots were unspiked matrices, all the results should be zero, so any positive result indicates an incompletely removed or newly-created interference.

Figure 1 shows the BEC for As (measured at mass 75) in each gas mode, plotted against each matrix.

The He mode data were consistent and at a low level, as would be expected since all these samples

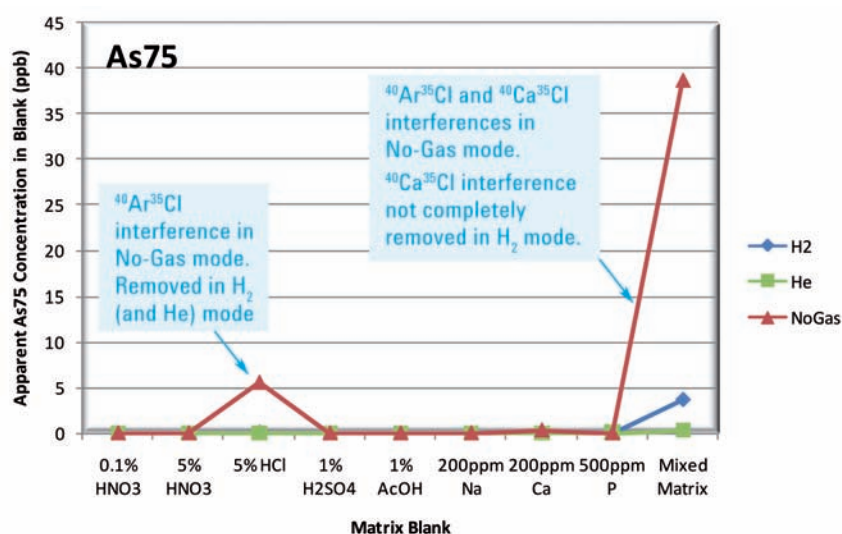


Figure 1. Comparison Plots of As75 BEC vs Sample Matrix for All 3 Cell Gas Modes

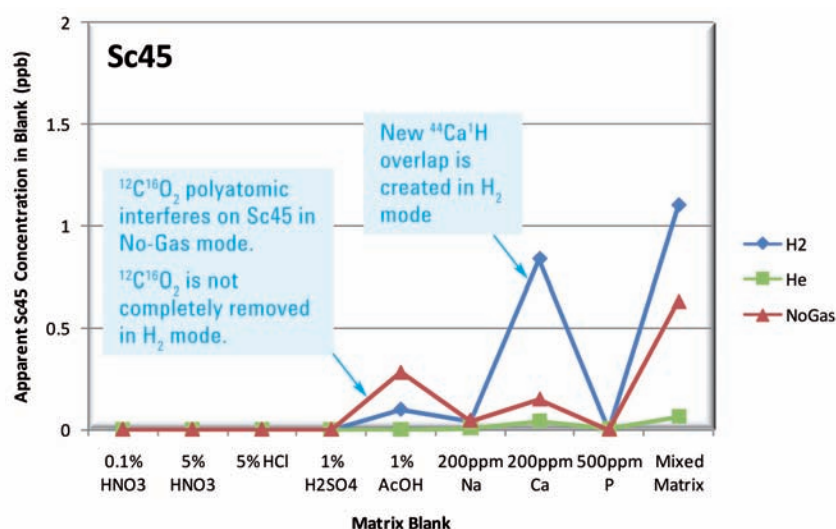


Figure 2. Comparison Plot of Sc45 BEC vs Sample Matrix for All 3 Cell Gas Modes

were unspiked matrices. However, the No-Gas mode data showed a marked positive bias for As in the 5% HCl matrix, due to the interference from ArCl, which contributes to the signal at mass 75. This interference was removed effectively in reaction mode (H<sub>2</sub> cell gas), since ArCl is highly reactive with H<sub>2</sub> cell gas.

In the mixed matrix (the final point on the x-axis), the apparent As concentration in No-Gas mode was much higher than in the HCl matrix alone (about 38ppb, compared to about 5ppb). This is because the mixed matrix contained the Ca derived from the 200ppm Ca matrix, as well as the Cl from the 5% HCl matrix, leading to the formation of a new CaCl polyatomic interference on As, which was not seen in any of the single-component matrices.

It is interesting to note that, while H<sub>2</sub> mode was effective in removing ArCl, it did not completely remove the CaCl interference on As, illustrating the point that not all of the interferences at any given analyte mass may react with the same reaction gas. He mode, since it works by collision processes and is therefore effective against ALL polyatomic ions, gave a consistent low background for As, even in the mixed matrix.

For some combinations of analyte and matrix, another important problem of reactive cell gases was observed, as illustrated in Figure 2, for Sc45 in the Ca matrix.

Once again, the apparent Sc concentration measured in He mode was low and consistent in all the different single matrices, and in the mixed matrix. In the No-Gas cell mode a polyatomic interference was observed on Sc in the 1% acetic acid matrix, due to the formation of the CO<sub>2</sub> polyatomic ion in this matrix. This polyatomic was reduced but not completely removed in H<sub>2</sub> mode. In the Ca matrix, however, the level of polyatomic interference on Sc was relatively low in No-Gas mode (and essentially zero in He mode), but much higher in H<sub>2</sub> mode. This is because H<sub>2</sub>, being a reactive cell gas, reacts with Ca to form a new cell-formed reaction product ion, <sup>44</sup>CaH, greatly increasing the interference on Sc45.

The third potential issue with the use of a reactive cell gas is the loss of analyte sensitivity as a result of the analyte ions reacting with the cell gas. A comparison of the spectra for

the 10ppb calibration standard (i.e. in the absence of any matrix components) is shown in Figure 3. In No-Gas mode (top), the measured peak pattern showed Co59 having the highest intensity, followed by Ni58 and Cu63, with Zn64 and the minor isotopes all being lower.

In He collision mode, this general pattern of peak intensities was maintained, although the spectrum demonstrated a slight increase in mass bias, due to scattering of the lighter ions.

In H<sub>2</sub> mode, by contrast, the highest peak was Zn64, followed by Co, and then the minor Zn isotopes at mass 66 and 68. The relative intensities of the Ni and Cu peaks (and to a lesser extent the Co59) were dramatically reduced, compared to the relative sensitivity in No-Gas mode. The peak intensities are shown relative to Co59, to remove any differences in absolute sensitivity between cell gas and no cell gas modes. It is clear that, since the comparison was based on the sensitivity of the other

analytes relative to Co, which was itself significantly reduced in H<sub>2</sub> mode, in real terms, the relative loss of sensitivity for Ni and Cu was actually much greater than is apparent from these plots, by a factor of 20x, relative to Zn. This loss of analyte sensitivity by reaction is one of the main reasons why reactive cell gases are not widely used successfully for multi-element applications.

## Conclusions

For multi-element analysis in a complex, multi-component matrix, it is clear that He collision mode is more effective and produces more reliable data than H<sub>2</sub> reaction mode. Reaction mode has 3 key drawbacks: inability to remove unreactive interferences, creation of new polyatomic species, and significant loss of sensitivity for certain analytes due to reaction.

## Reference

1. ICP-MS Primer, page 34, Agilent Technologies publication 5989-3526EN

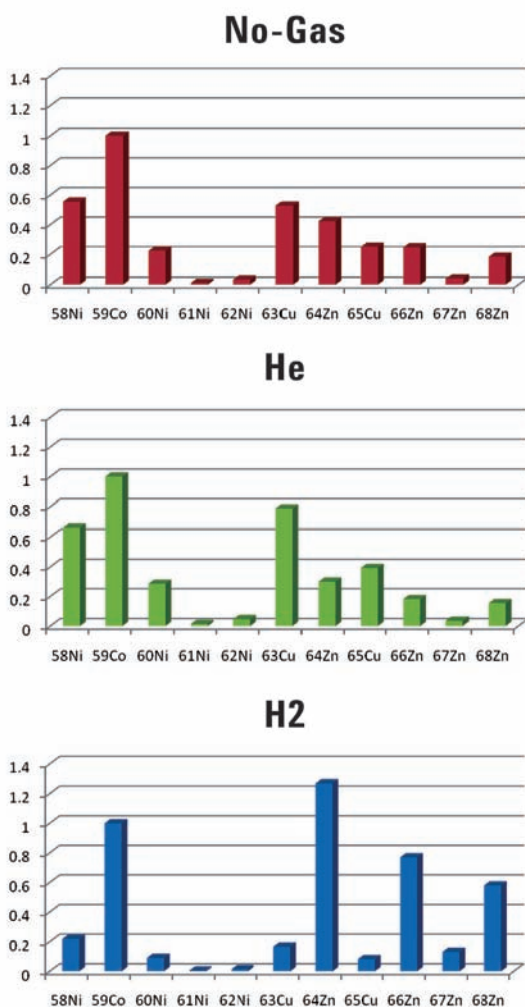


Figure 3. Relative Sensitivity for Analytes Co, Ni, Cu and Zn, in No-Gas Mode (top), He collision mode (middle) and H<sub>2</sub> reaction mode (bottom)

# Food Regulatory Lab Moves to HMI/7500cx ICP-MS

Robert Sheridan, Thomas King

Division of Food Laboratory at New York State Department of Agriculture and Markets, USA  
[www.agmkt.state.ny.us](http://www.agmkt.state.ny.us)

## Introduction

A team from the Division of Food Laboratory at New York State Department of Agriculture and Markets, USA recently evaluated ICP-MS in a move to strengthen its trace metal analytical capability. The department supports consumer protection by testing foods for purity, quality and accurate labeling in New York State. The chemistry section of the food lab is responsible for trace metals analyses from pesticide residues to heavy metals in foods and animal feed. Example applications include:

- Pb and As levels in fruit juices
- Pb in candy
- Pb in maple syrup
- Pb, As, Cd, Cr and Se in various other foods.

To date the lab has relied on ICP-OES for these analyses but achieving desired limits of detection (LOD) can be difficult because of the inherent lack of sensitivity of ICP-OES. Current method LOD's are very close to the regulatory limits and certain matrix interferences can cause further problems with ICP-OES.

ICP-MS is well known for its high sensitivity compared to ICP-OES. Matrix interference management by ICP-MS is also well established through the use of collision/reaction cell (CRC) technology. A final barrier to overcome, and of particular relevance to food testing, has been the matrix tolerance of ICP-MS compared to ICP-OES. Traditionally samples prepared for analysis by ICP-MS have needed to contain less than 0.1% total dissolved solids (TDS). However, the recent development of the High Matrix Introduction (HMI) system for the Agilent 7500cx allows samples containing higher levels of TDS, up to 1%, to be analyzed by ICP-MS.

## Evaluation of ICP-MS

ICP-MS instruments from Agilent plus two other manufacturers were evaluated. The main criteria were:

- Exceed regulatory limits for Pb, As, Cd, Cr and Se in various food matrices

- Capability to analyze additional elements at low levels
- Improve sample throughput
- Ability to handle high matrix samples.

## Agilent 7500cx with HMI

Bert Woods, the Applications Specialist who demonstrated the Agilent 7500cx didn't request detailed information about the sample prep stage of the food samples or their TDS content. He was confident that samples prepared for analysis by ICP-OES could be analyzed by the HMI/7500cx in helium mode with no or limited dilution.

## Experimental

As and Pb were determined in two different candy digests (sample A and B) and a pear juice sample by the HMI/7500cx in He mode – see Table 1 for operating conditions.

Table 1. HMI/7500cx ICP-MS operating conditions

Parameter	Setting
RF power	1600W
Sample depth	10 mm
Carrier gas	0.3 mL/min
Make up gas	0.82 mL/min
He gas flow	4 mL/min
HMI setting	Robust (software setting)

## Results

The candy digests were analyzed directly and 10:1 diluted in 1% HNO<sub>3</sub>/0.5% HCl. The results (Table 2), which were the same for the undiluted and the 10:1 diluted samples, show that the sample can be analyzed directly, with good sensitivity. The calibration curve for As (Figure 1) confirms that the <sup>40</sup>Ar<sup>35</sup>Cl interference on <sup>75</sup>As has been removed by adding helium gas to the Octopole Reaction System (ORS).

	Candy A	Candy A	Candy A	Candy A
Dilution	10	10	1	1
<sup>75</sup> As	0.68	0.49	0.50	0.57
<sup>208</sup> Pb	0.74	0.72	0.70	0.70
	Candy B	Candy B	Candy B	Candy B
Dilution	10	10	1	1
<sup>75</sup> As	1.23	1.26	1.25	1.25
<sup>208</sup> Pb	1.99	1.94	1.96	1.95

Table 2. Repeat results for As and Pb in two candy digests (ppb), using different dilution factors

	Pear	Pear	Pear	Pear
Dilution:	50	50	10	10
<sup>75</sup> As	63.3	61.2	67.2	67.6
<sup>208</sup> Pb	4.87	4.56	4.48	4.42

Table 3. Repeat results for As and Pb in pear juice (ppb), using different dilution factors

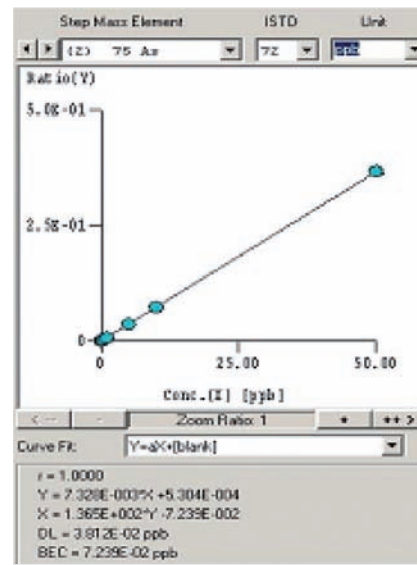


Figure 1. As calibration curve

The pear juice was analyzed at 50 and 10:1 dilutions. Again the diluent was 1% HNO<sub>3</sub>/0.5% HCl.

The Pb results (Table 3) were identical for both dilutions and the arsenic results were within 10%. This means that pear juice can be analyzed at 10:1 dilution with the HMI/7500cx. Avoiding a larger dilution, which may result in error and poorer levels of detection, is a key advantage of the method.

## Advantages of HMI/7500cx

New York State Department's Food Lab decided in favor of the Agilent HMI/7500cx ICP-MS system because

of its ability to analyze high matrix food samples and digests prepared for ICP-OES analysis, with ICP-MS levels of detection following minimal or no dilution.

The sample throughput capability of ICP-MS was also evaluated. Operating the 7500cx in a single analysis mode (helium mode) reduced the analysis time per sample to less than one minute.

### Future Proofing

Investing in ICP-MS also insures that the lab is well placed to meet any future regulations pertaining to foods that the US Food and Drug Administration (FDA) may introduce. Currently the FDA has a limit of 0.1 ppm for Pb in candy and 10 ppb As in drinking water.

As the capability for detecting more analytes improves, regulators may consider rules for products previously not considered, such as As and Pb in fruit juice.

## Design Changes on GC Interface for the 7500 Series

### Tomo Yamada

ICP-MS Product Manager, Tokyo Analytical Division, Agilent Technologies

A modified Agilent GC Interface – used to couple Agilent’s GC to the 7500 Series, is available from August 2008. The design has been changed for easier installation and better performance. The new product number is G3158B.



Figure 1. New alignment tool

### Enhancements:

- The injector alignment tool (Figure 1) has been modified for easier alignment. The new design allows

the injector center position to be located more easily. The Z-position can also be set with the tool.

- The torch connecting clamp screws have been modified for easier assembly.
- The Ar preheat tubing coil is now located in the GC valve box. This insures that the temperature of the makeup Ar is kept stable during GC oven temperature changes.
- The modified interface now supports the Agilent 7890 GC in addition to the 6890 GC. New 7890 GC firmware (revision A.01.09) has a preconfigured heat zone setup specifically for the modified GC-ICP-MS interface.

## Tip and Trick: Much Faster Gas Switching Times with H<sub>2</sub> Vent Valve

### Tomo Yamada

ICP-MS Product Manager, Tokyo Analytical Division, Agilent Technologies

7500cs (G3273B version) mainframes, and 7500cx mainframes (G3272B version) fitted with the optional H<sub>2</sub> cell gas line, are now equipped with a new cell gas evacuation assist valve. The valve allows much more efficient venting of H<sub>2</sub>, and so reduces the stabilization time needed when switching from H<sub>2</sub> to another gas mode.

With the vacuum assist valve, the recommended order in which gas modes are used is now:

No-Gas → He → H<sub>2</sub>.

Using this new sequence will shorten total analysis time, since H<sub>2</sub> mode stabilizes and vents quickly (typically 10-15 sec), and No-Gas mode stabilizes during rinse and sample uptake.

Check your mainframe model number to confirm you have the evacuation assist valve fitted: all G3273B and G3272B (with H<sub>2</sub> line) mainframes incorporate the valve.

## Quick Start Guide to Agilent 7500 Series ICP-MS ChemStation

### Paul McMahon

ICP-MS Technical Marketing Engineer, Tokyo Analytical Division, Agilent Technologies

The ICP-MS ChemStation Quick Start Guide is a brief (40 page) color manual that describes how to use the Agilent 7500 Series ICP-MS ChemStation from instrument startup through to sample analysis and data handling. The guide is useful to all ICP-MS ChemStation users, but anyone doing environmental analysis will find it especially useful.

### Topics discussed include:

Startup, Getting Started, Things to check before analysis, Prepare tuning solutions, Ignite the plasma, Check tuning, Quantitative method setup, Set up the Sequence, Analyze the samples, Analyze the data (Quantitative Analysis), Other Features, Quality Control (Intelligent Sequencing), Semi-quantitative Analysis, Batch View.

Download a copy of the guide from the Agilent ICP-MS User Forum.

To access the Forum, you will need to log-in to the Agilent web site, or register, if you haven't done so previously. You will be asked to enter your instrument's serial number on your first visit only. Look for the link to the ICP-MS User Forum from: [www.agilent.com/chem/icpms](http://www.agilent.com/chem/icpms)

# NEW! MassHunter Software Platform for the 7500 Series

**Tomo Yamada**

ICP-MS Product Manager, Tokyo Analytical  
Division, Agilent Technologies

## Introduction

Agilent has developed an all-new software platform which will replace the ChemStation software: the MassHunter Workstation software is based on the highly successful MassHunter LC/MS software and will become common to all Agilent MS products.

There are many new features, but key features of MassHunter Workstation (A.01.01) are:

- Batch-at-a-glance data review (Figure 1)
- Outlier flagging (Figure 2)
- Reporting via Excel 2007

## Batch-at-a-glance Data Review

From this new data analysis window, all necessary information can be easily reviewed – e.g. analytical results, spectra, calibration plots, ISTD stability chart, allowing the user to:

- Review samples more easily
- Review sample data in real time
- Customize the window layout.

## Outliers - Automatic flagging

Outlier values, such as count RSD, ISTD recovery, calibration linearity, calibration range, etc. are automatically flagged and displayed in the batch table.

- Color coding allows the user to identify and review the outliers.
- Potential analytical problems can be located immediately.
- Filters can display only samples that have outliers.

## Reporting via Excel 2007

- MassHunter Workstation ships with Microsoft Excel 2007.
- Export menu directly generates an Excel file - saving time.
- Analytical results or calibration information including BEC and DL are exported to Excel.
- The reporting templates are Excel files.

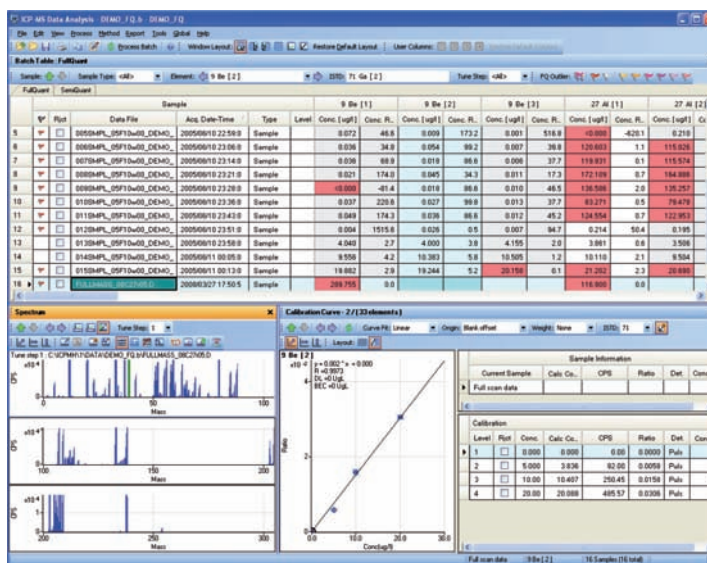
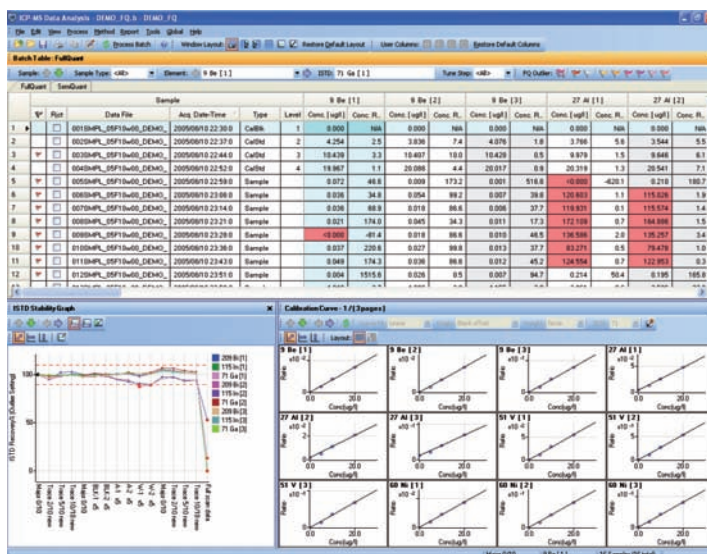


Figure 1. Two examples of batch-at-a-glance data review

## Other New Features:

- Standard Addition calibration plot is automatically converted to an external calibration plot – no user intervention required.
- SemiQuant factors are automatically corrected in the sequence – no need for manual update.
- Spectrum overlay.
- Plus many other improvements.

0.007	39.8	120.603	1.1	115.026	1.9
0.0	FullQuant Outlier(s)				
0.0	Al: Concentration value = 83.27 is outside the calibration range [0, 20]				
0.010	46.5	6.586	2.0	135.257	3.4
0.013	37.7	83.271	0.5	79.478	1.0

Figure 2. Outlier flagging

## Agilent MassHunter Workstation

The MassHunter Workstation for the 7500 Series is part of the Agilent

MassHunter Workstation family – currently used on Agilent TOF, Q-TOF and triple quadrupole LC/MS systems. LC/MS users will find the same familiar interface now on ICP-MS.

## Availability

The 7500 Series will be available with either ChemStation or MassHunter (with new ICP-MS orders only) during the transition period. Contact your ICP-MS specialist to find out which one is recommended. An upgrade for existing 7500 users will be available at the end of the year.

## Take Agilent's Mass-ter-Mind Challenge

**Rich Quashne**

Marketing Program Manager, Agilent Technologies, Little Falls, USA



Put your ICP-MS knowledge to the test against other experts from around the world. Answer the most questions correct in the shortest time to win FREE Agilent products.

Every player will receive a special offer for ICP-MS supplies.

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[www.mass-ter-mind.com](http://www.mass-ter-mind.com)

## Receive FREE ICP-MS Vials with ICP-MS Supplies

Purchase over \$1200 (USD) of Agilent ICP-MS supplies and receive a FREE pack of ICP-MS vials for the Agilent Integrated Autosampler (I-AS).

Your choice of either:

- 6ml vials for Tray A-89 position, 200/pk (Part #G3160-65303)
- 18ml vials for Tray B-53 position, 55/pk (Part #G3160-65304)

### Better Results with Genuine Parts

With Agilent ICP-MS supplies and accessories, the difference is in the details. All our products have been engineered or selected by our instrument design teams, manufactured to our demanding specifications, and tested under a variety of conditions. You can be sure that you're getting the best possible results for your analysis by using genuine Agilent ICP-MS components.

## New Elemental Bio-Imaging Facility

**Tony Crocker**

Agilent Technologies, Australia



Agilent's Rudy Grimm (center) and Toshiaki Matsuda (right) celebrate with UTS on the opening of the Elemental Bio-Imaging Facility in June 2008.

Considered a world first, the Elemental Bio-Imaging Facility based at the University of Technology, Sydney (UTS), Australia, will be involved in metallomics research that examines the study of metals and their interactions with proteins in the body.

With increasing evidence that the excessive accumulation or imbalance of metals play a role in the development of neurodegenerative disorders such as Alzheimer's and Parkinson's diseases, scientists at UTS are pioneering the use of laser ablation ICP-MS in their research. The technique involves ablating thin sections of human/animal tissue such as the brain and employing two-dimensional imaging to map the levels and distribution of various elements. When combined with other diagnostic technologies, LA-ICP-MS results may lead to a breakthrough in understanding these conditions.

The centre has recently purchased their second Agilent 7500 ICP-MS, linked to a New Wave Research laser.

## All Aboard for the French User Group Meeting

**Jérôme Darrouzès**

ICP-MS Specialist, Agilent Technologies, France



On 13th April 2008, twenty five 7500 and 4500 users boarded the "La Bretagne" barge, moored close to the Eiffel Tower, for the 3rd annual French Agilent ICP-MS user meeting.

ICP-MS Specialists, Jérôme Darrouzès and Laurent Naëls, updated users on the latest technological, application and support developments from Agilent and were on hand to provide feedback during the various Q and A sessions.

Special thanks go to Frédéric Candaudap (LMTG, Toulouse) and Stéphane Dubascoux (LCABIE, Pau) for their respective excellent contributions on the capability of He mode in geological matrices and the coupling of FFF (field flow fractionation) to ICP-MS for the study of colloidal fractions.

Lunch provided the opportunity for informal chat while cruising on the Seine and taking in the sights of Paris.

Look out for details of the next French ICP-MS User Meeting already planned for early 2009.

Details of the Worldwide User Group meeting to be held at the Winter Plasma Conference in Graz in Feb 2009 will be available on-line soon from [www.agilent.com/chem/icpms](http://www.agilent.com/chem/icpms)

## TDK Win Global Environmental Award

On February 20<sup>th</sup> 2008, TDK Corporation was awarded the highest honor at the Fuji Sankei Group's 17<sup>th</sup> Global Environmental Award Grand Prize ceremony for their pioneering work on trace metal contamination of electronic components.

TDK have significantly reduced the amount of acid and heat they use in their sample prep procedures by directly analyzing constituent parts and solid materials with their Agilent laser ablation-ICP-MS system. Compared to the old methodology, the LA-ICP-MS approach requires 90% less reagents and has cut CO<sub>2</sub> emissions by about 60%.

The judges were also impressed by TDK's openness to share their technological advances across the TDK group and industry through their participation at conferences and seminars.

## Trade Shows and Conferences

**JAIMA Show**  
September 3-5, 2008  
Chiba, Japan

**Isotracc 2008**  
September 9-11, 2008  
Lyon, France

**German ICP-MS  
All User Conference**  
September 17-19, 2008  
Dresden, Germany

**Winter Conference on  
Plasma Spectrochemistry**  
Feb 15-20, 2009  
Graz, Austria  
[www.winterplasmagraz.at](http://www.winterplasmagraz.at)

## New Agilent ICP-MS Users

A very warm welcome to all companies and institutions that have recently added an Agilent ICP-MS to their analytical facilities. Remember to join the Agilent web-based ICP-MS User Forum – the place where you can exchange information relating to your new ICP-MS. You will also find a host of resources in the Forum designed to help you get the most from your 7500.

To access the Forum, you will simply need to log-in to the Agilent web site, or register if you haven't done so previously, and enter your instrument's serial number on your first visit only. Look for the link to the ICP-MS User Forum from: [www.agilent.com/chem/icpms](http://www.agilent.com/chem/icpms)

## 4500 Series Reaches EOS

The Agilent 4500 Series ICP-MS has reached its End of Support (EOS). For more information on transitioning to the 7500 Series, contact your Agilent support sales representative or visit: [www.agilent.com/chem/icpms](http://www.agilent.com/chem/icpms)

## Agilent ICP-MS Publications

To view and download these latest publications, go to [www.agilent.com/chem/icpms](http://www.agilent.com/chem/icpms) and look under "Library Information"

Agilent has developed a new, informative poster specifically for your lab wall. By outlining the basic principles of ICP-MS in an easy to follow and clear way, the poster will help you when you need to explain the technique to your colleagues or visitors.

- Title: "Fundamental Principles of the Agilent 7500 Series ICP-MS with Octopole Reaction System", 5989-8628EN.



- Dimensions: 36" x 28" (96 x 71cm).

- Order a copy of the poster on-line or from your local Agilent office.

**Mass Card:** Periodic Table/Relative Isotopic Abundance Table, 5989-8540EN

**Technical Feature:** Heavy Metals Content of Dietary Supplements by Agilent 7500 ORS ICP-MS, 5989-8813EN

### Front page photo:

Peter Planitz, long serving Agilent ICP-MS Application Engineer based in Germany.

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