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# 8800 Triple Quadrupole ICP-MS Special Issue



The Measure of Confidence



Agilent Technologies

# Agilent's New 8800 Triple Quadrupole ICP-MS: Hardware and Technology Introduction

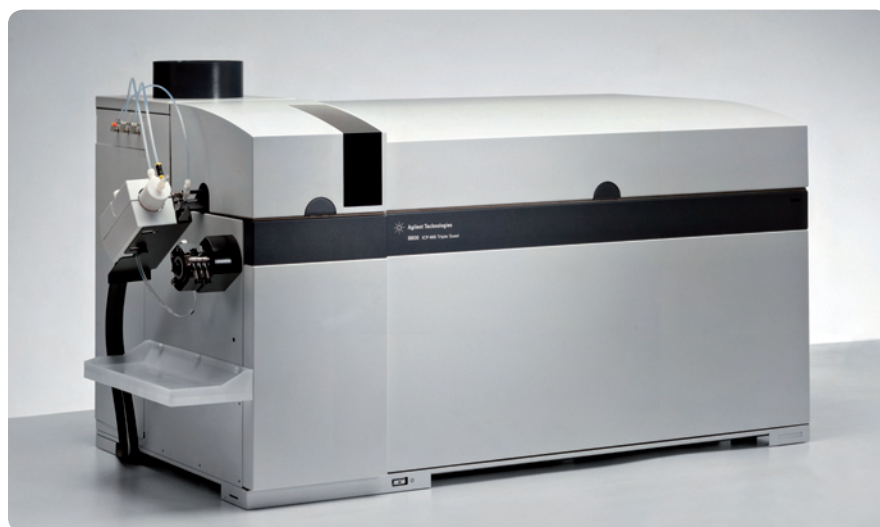
Ed McCurdy, Nori Yamada and Naoki Sugiyama  
Agilent Technologies

Building on the success and market leadership of the 7700 Series ICP-MS, Agilent has announced an exciting and important development that transforms ICP-MS technology – the Agilent 8800 Triple Quadrupole ICP-MS.

## Current ICP-MS Technology

The vast majority (around 90%) of ICP-MS systems sold worldwide use a quadrupole mass spectrometer (ICP-QMS), with alternative configurations (double-focusing sector field, and time of flight) making up the remainder. The benefits of the QMS in terms of sensitivity, simplicity, cost, speed and flexibility are apparent from the acceptance of ICP-QMS as the preferred method for trace metal analysis across virtually all industries.

The development and near universal acceptance of collision/reaction cell (CRC) technology in the last decade has further extended the scope of ICP-MS, allowing previously difficult samples and problematic interfered analytes to be measured more reliably and at lower levels.



## Interference Removal using CRCs

Collision/reaction cells in quadrupole ICP-MS may use either:

- Collision mode, using an inert cell gas (usually helium), or
- Reaction mode, using a reactive cell gas such as H<sub>2</sub>, O<sub>2</sub> or NH<sub>3</sub>.

The benefits of helium (He) mode on the current single-quadrupole 7700 Series ICP-MS have been well documented as delivering reliable multi-element analysis in complex, variable and often high-matrix samples. He mode is used routinely in a wide range of different industries and applications around the world, but it does have some limitations: He mode cannot separate isobaric overlaps (such as <sup>40</sup>Ar on <sup>40</sup>Ca) and it is not effective against doubly-charged interferences. The reactive cell gas approach can provide faster and more complete removal of some very intense interferences, which may be required in applications where interfered elements must be

measured at very low (ppt) levels, such as in high purity semiconductor materials. However, reaction gas methods using conventional quadrupole ICP-MS have gained limited acceptance in the analysis of more complex or varied samples due to the fact that:

- Reactions targeted at removing specific interferences may cause new interferences from other reaction product ions.
- In analyte mass-shift methods, where a reactive analyte is moved to a new mass away from the original interference, other analyte or matrix elements or reaction product ions may also be present at that new analyte product ion mass.

These limitations are especially problematic if the sample matrix is unknown or variable, since the ions and therefore the reaction processes in the cell are affected by the sample composition.

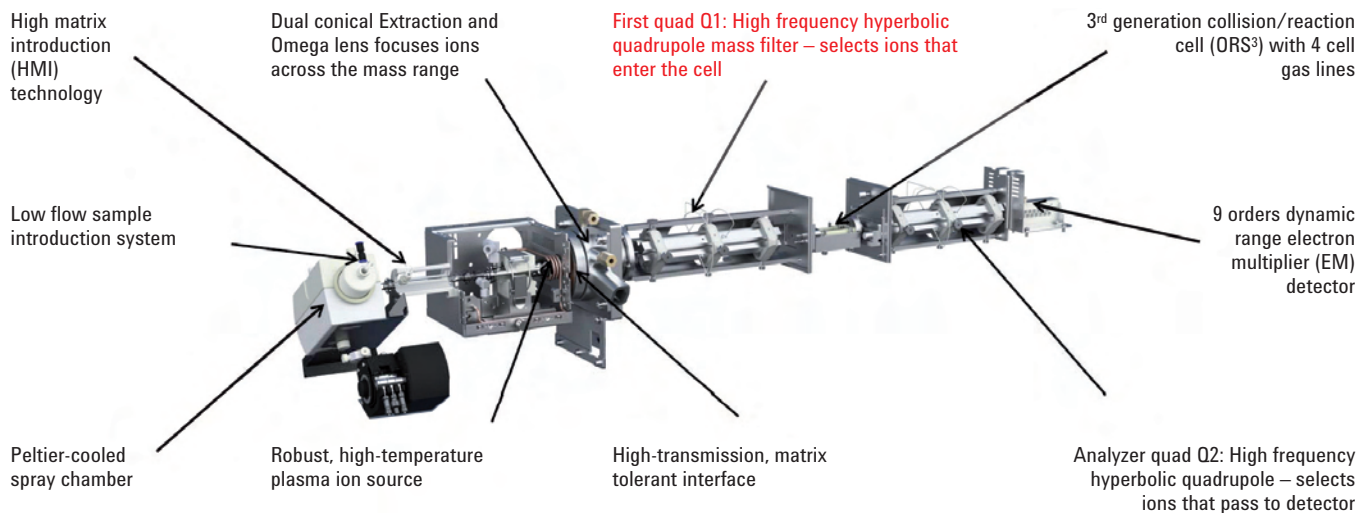


Figure 1. Schematic of the Agilent 8800 Triple Quad ICP-MS

## 8800 ICP Triple Quad

The new Agilent 8800 has been developed to address these problems of reactive cell gases in quadrupole ICP-MS, as the tandem mass spectrometer configuration enables operation in MS/MS mode. In this mode, the first quadrupole functions as a unit mass filter, so it rejects all masses except the target analyte and any on-mass interferences. This means that the ion beam that enters the cell is not only much simpler in composition, but it also remains consistent even if the sample matrix and other analyte concentrations change. ICP-MS/MS transforms reaction cell operation from its current limited role, into a widely-applicable and reliable analytical tool.

The Agilent 8800 is the world's first and only commercial triple quadrupole ICP-MS, but the basic instrument layout is similar to the 7700, with the exception of the first quadrupole placed in front of the ORS<sup>3</sup> cell. As with the 7700, the cell uses an octopole ion guide; the term "triple-quad" follows the conventional generic description for organic tandem mass spectrometers that utilize two quadrupole mass spectrometers separated by a collision/reaction cell. This is shown in the schematic (Figure 1), which also illustrates the familiar sample introduction and Q2/detector sections of the 8800.

Enhancements to the interface and vacuum system mean that when the 8800 is operated in "Single-Quad" mode, typical sensitivity is increased about 2x, while background is reduced about 5x compared to a 7700.

The 8800 is designed for both high performance (see following article) and flexibility. The system utilizes four argon gas mass flow controllers (for plasma, auxiliary, make-up/dilution and nebulizer gases), as well as a 5<sup>th</sup> gas line for optional gases such as O<sub>2</sub>/Ar (for organics), He carrier gas (for laser ablation) or N<sub>2</sub>

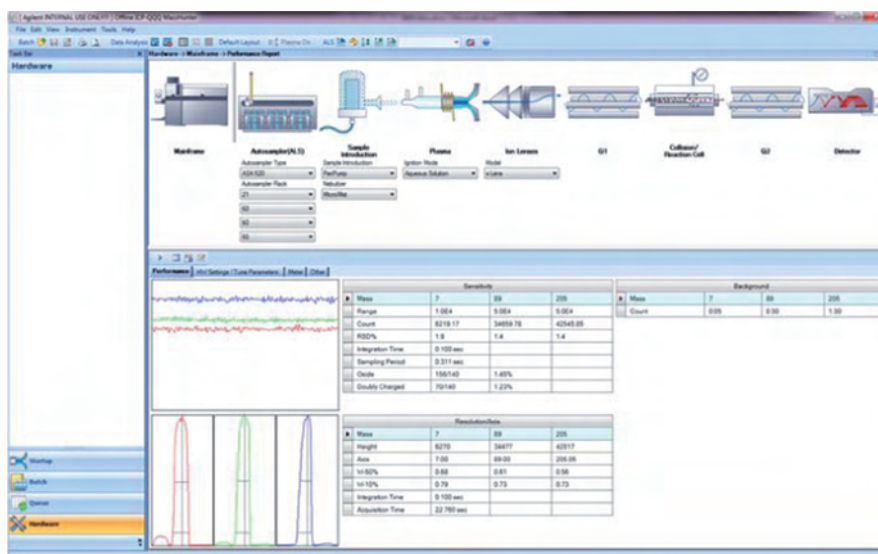


Figure 2. MassHunter screenshot of Agilent 8800 ICP Triple Quad hardware

(to modify plasma conditions).

Flexible cell mode functionality is provided by the 4-channel cell gas flow controller. One channel provides He mode performance comparable to the 7700x, while three additional reaction gas lines, two for high flow rates (up to 10 mL/min) and one for low flows (up to 1 mL/min), allow ultimate flexibility.

### Based on the Proven 7700 Platform

Sharing proven 7700 Series ICP-MS sample introduction and plasma technology guarantees excellent reliability and serviceability, as well as commonality of consumables. The robust plasma design and integrated High Matrix Introduction system (HMI) mean that the 8800 shares the same unparalleled matrix tolerance as the 7700.

This common configuration also means that sample introduction options and autosamplers are consistent across both instrument platforms. Additional 7700 peripherals are also supported on the 8800, such as Agilent GC and LC modules including the new bio-inert

LC and Capillary LC systems. These LC and GC modules are controlled directly from the 8800's software which is based on the same widely acclaimed MassHunter suite as used on the 7700 Series. MassHunter for the 8800 (Figure 2) provides a range of system optimization and method development tools to ensure consistent operation and reliable data. As with the 7700, Startup provides automated optimization of the system hardware (such as the torch axis and EM settings), while autotune can optimize other system parameters for a given batch or application.

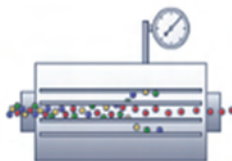
### Conclusions

The 7700 remains the benchmark for high performance quadrupole ICP-MS, with unmatched He mode performance providing reliable analysis of complex and variable samples across a range of industries and applications. The 7700 is now joined by the ground-breaking new 8800 ICP Triple Quad, which transforms ICP-MS technology and redefines reaction-mode performance.



#### Q1 – controls ions that enter the cell

- Consistent reactions even if sample composition changes



#### ORS<sup>3</sup> – collision/reaction gas added

- Ions react and are neutralized or moved
- Product ions are formed



#### Q2 – selects the target analyte mass

- Interference-free analyte ions passed to EM



# Agilent's 8800 ICP Triple Quad: Modes of Operation

Ed McCurdy, Glenn Woods and Nori Yamada

Agilent Technologies

In addition to providing improved performance compared to conventional quadrupole ICP-MS, the new Agilent 8800 offers several unique modes of operation due to its tandem mass spec configuration. As well as offering unmatched flexibility to carry out advanced research, these novel modes enable a level of performance not previously possible with quadrupole ICP-MS.

## Unique Modes of Operation

The triple-quad configuration of the new Agilent 8800 provides an unprecedented degree of control over the processes in the cell, giving more predictable and reliable data when using reactive cell gases. The advanced MS/MS modes enabled by the tandem MS configuration are discussed later, but the 8800 can also operate in modes familiar to users of conventional quadrupole ICP-MS (no gas, He mode, hot and cool plasma, and so on). In this setup, the 8800 is operated in "Single Quad" mode, with the first quadrupole (Q1) operating as an ion guide.

## Single-Quad Mode

In cases where consistency with an existing quadrupole ICP-(Q)MS method is required, the 8800 can be operated in "single-quad" mode. In this mode, Q1 can be operated either as:

- A simple ion guide (no mass selection). All masses are passed to the cell, so the 8800 is functioning like a conventional single quadrupole ICP-MS with a passive cell, such as the 7700; or
- An adjustable bandpass filter, (where the Q1 low-mass and high-mass cutoffs are scanned synchronously with the Q2 mass). In this mode, the ions outside the bandpass window are rejected by Q1 and so don't enter the cell, but otherwise this mode emulates a conventional quadrupole ICP-MS with a "scanning" cell based on a quadrupole ion guide.



## MS/MS Mode

The real breakthrough in performance offered by the 8800 is derived from its tandem MS configuration. The 8800 uniquely offers the capability to operate in MS/MS mode, where both Q1 and Q2 function as unit mass filters. In this mode, Q1 controls the ions that enter the collision/reaction cell, so non-target masses are rejected and any changes in co-existing analyte or matrix elements do not disrupt the reaction processes used to remove the on-mass interferences. This revolutionizes the performance and applicability of reaction mode ICP-MS.

MS/MS mode performance on the 8800 is illustrated in the calibration plot and spectrum shown in Figures 1 and 2. Figure 1 shows the calibration for V in high-purity H<sub>2</sub>SO<sub>4</sub> (measured directly at 9.8%, after a 1:10 w/w dilution). The use of NH<sub>3</sub> cell gas provides very fast and effective removal of the SO<sup>+</sup> and SOH<sup>+</sup> overlaps on V at mass 51, allowing <sup>51</sup>V<sup>+</sup> to be measured directly at extremely low levels; background equivalent concentration (BEC) and detection limit (DL) were 0.13 ppt.

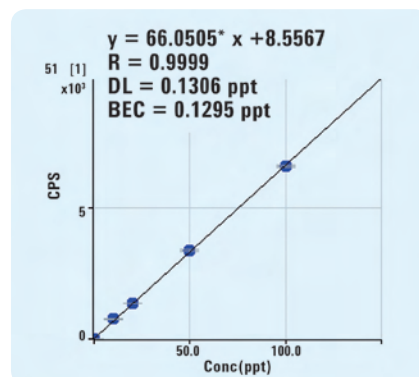


Figure 1. Calibration for V in 9.8% H<sub>2</sub>SO<sub>4</sub>. BEC and DL of 0.13 ppt

In cases where the analyte is reactive and the interference is not, MS/MS mode with Mass-Shift can be used to measure the analyte as a new cell-formed product-ion at a mass free from interference. This mode is illustrated in Figure 2 for the analysis of sulfur, measured as SO<sup>+</sup> ions produced by reaction with O<sub>2</sub> cell gas. O<sub>2</sub><sup>+</sup> does not react with the O<sub>2</sub> cell gas to form O<sub>3</sub><sup>+</sup>, so <sup>32</sup>S can be moved away from the <sup>16</sup>O<sub>2</sub><sup>+</sup> overlap at the original analyte mass. Figure 2 shows the mass spectrum for SO<sup>+</sup> confirming that the S isotopic abundance is maintained in the SO<sup>+</sup> peak pattern.

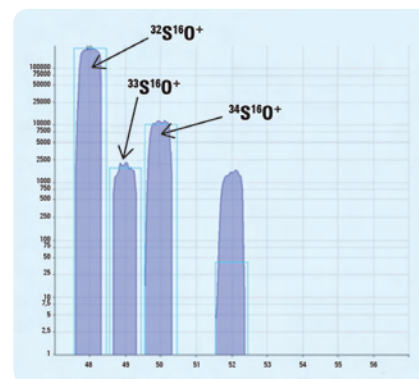


Figure 2. Spectrum of SO<sup>+</sup> product ions, showing S isotope template fit

Reaction gases and mass-shift mode can be used with conventional quadrupole ICP-MS, but there are limitations that can compromise accuracy or reduce the range of applications for which these modes can be used. In the case of S analysis, the SO<sup>+</sup> product ion from the major <sup>32</sup>S isotope appears at mass 48, where there are also native isotopes of Ca and Ti, so high or variable levels of these elements may interfere with the SO<sup>+</sup> analysis. When organic solvents are measured, <sup>36</sup>Ar<sup>12</sup>C<sup>+</sup> also overlaps SO<sup>+</sup> at mass 48.

## Isotopic Analysis in MS/MS Mode

In conventional quadrupole ICP-MS, each reaction product ion can be a mix of several overlapping isobars. In the example of  $\text{SO}^+$ , the product ion  $^{34}\text{S}^{16}\text{O}^+$  (at mass 50) is overlapped by  $^{32}\text{S}^{18}\text{O}^+$ , so variations in the relative abundance of  $^{34}\text{S}$  and  $^{32}\text{S}$  cannot be distinguished, making isotope dilution and isotopic tracer measurements unreliable. However, the 8800 in MS/MS mode allows only one target isotope mass into the cell at a time, and monitors a specific reaction transition ( $+^{16}\text{O}$ ), so the relative isotopic abundance of the target element is preserved. When  $^{32}\text{S}^{16}\text{O}^+$  is measured at mass 48, no  $^{34}\text{S}$  is entering the cell, and when  $^{34}\text{S}^{16}\text{O}^+$  is measured at mass 50, no  $^{32}\text{S}$  is entering the cell, so no mixing of the isotopic pattern can occur.

## Neutral Gain Scan

The  $\text{SO}^+$  measurement described in the example above would typically be performed using the normal peak jumping mode of acquisition, as the target product ions are known in advance. However, the spectrum shown in Figure 2 illustrates another useful mode of analysis enabled by the Triple Quad configuration in MS/MS mode, namely Neutral Gain Scan. In this mode Q1 and Q2 are both scanned, but with a user-defined mass difference between their set masses. For example to measure the product ions from O-atom addition, Q2 would scan at  $\text{Q1} + 16$  amu. Another example of neutral gain scan is shown in Figure 3, which illustrates the  $\text{TiO}^+$  product ions formed at mass 62 to 66 from the analysis of Ti with  $\text{O}_2$  cell gas. Four overlaid spectra are shown, demonstrating the consistent isotope pattern obtained for 1 ppb Ti in a single element standard and in the presence of other elements Ni, Cu, and Zn. The perfect Ti isotopic template

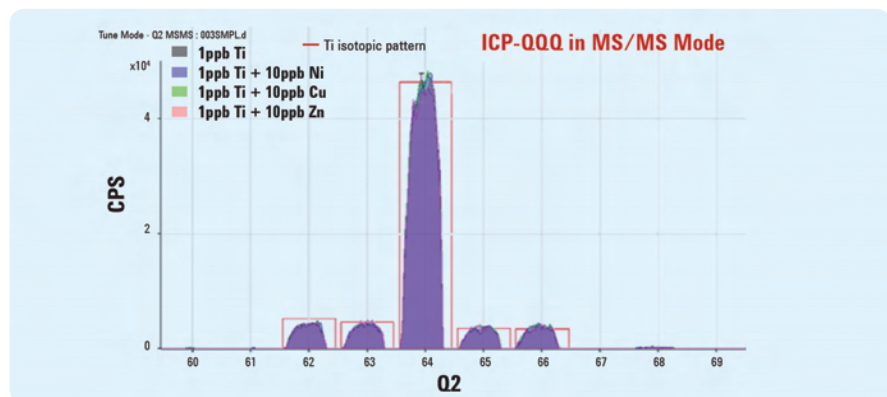


Figure 3. 8800 MS/MS mode Neutral-Gain Scan of Ti with  $\text{O}_2$  cell gas

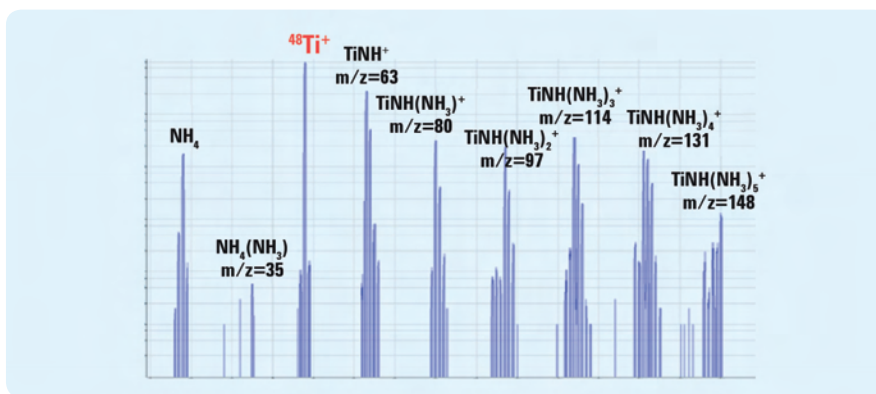


Figure 4. 8800 MS/MS mode Product Ion Scan for Ti with  $\text{NH}_3$  cell gas

fit in all samples demonstrates the unmatched ability of MS/MS to separate the  $\text{TiO}^+$  product ions from the potential  $\text{Ni}^+$ ,  $\text{Cu}^+$  and  $\text{Zn}^+$  overlaps. In conventional quadrupole ICP-MS, the  $\text{TiO}^+$  product ions can be formed, but the  $\text{TiO}^+$  peaks can all be overlapped by isotopes of  $^{62}/^{64}\text{Ni}$ ,  $^{63}/^{65}\text{Cu}$  or  $^{64}/^{66}\text{Zn}$  at their native masses.

## Product Ion Scan

In Product Ion Scan mode, Q1 is set to a user-defined mass, allowing only the target precursor ion mass to enter the cell, while Q2 scans over a selected mass range to measure all the product ions formed in the cell. In addition to allowing a unique window onto the reaction processes occurring in the cell, Product Ion Scan is a genuinely useful analytical mode, as the product ions measured are all derived from the single analyte mass that Q1 allows to enter the cell.

This means that highly reactive cell gases such as  $\text{NH}_3$  can be used for complex and variable samples, with confidence that the product ions measured are not overlapped by any native or cluster ions formed from another element. This is a major advance compared to conventional

quadrupole ICP-MS analysis, where all ions enter the cell, and so reaction product ions can form from any of the co-existing element or matrix ions.

The MS/MS mode Product Ion Scan in Figure 4 shows the cluster ions formed when  $^{48}\text{Ti}$  is measured using  $\text{NH}_3$  cell gas. Although many cluster ions form and the spectrum looks complex, every cluster ion peak was formed from only  $^{48}\text{Ti}$ , as all other masses were rejected by Q1. In  $\text{NH}_3$  cluster ion analysis on conventional quadrupole ICP-MS, the analytically useful Ti cluster ions,  $^{48}\text{TiNH}^+$  (mass 63),  $^{48}\text{TiNH}(\text{NH}_3)_3^+$  (114), and  $^{48}\text{TiNH}_2(\text{NH}_3)_3^+$  (115) are all overlapped by multiple other elements. For example the  $^{48}\text{TiNH}^+$  cluster is overlapped by  $^{63}\text{Cu}$ , while  $^{114}\text{Cd}$ ,  $^{114}\text{Sn}$ ,  $^{64}\text{ZnNH}_2(\text{NH}_3)_2^+$  and  $^{63}\text{Cu}(\text{NH}_3)_3^+$  all appear at mass 114.

## Precursor Ion Scan

Precursor Ion Scan mode is similar to Product Ion Scan, except that Q2 is set to a user-defined target ion, while Q1 scans over a user-set mass range to select the precursor ions that can enter the cell and react.

This mode allows monitoring of a specific product ion mass for all ions in the Q1 scan range.

## Conclusions

This brief overview gives an indication of the significant improvements in data quality and consistency that can be achieved with the new Agilent 8800 ICP Triple Quad, especially in reaction mode. We have also introduced the unparalleled flexibility that MS/MS mode brings to reaction mode analysis. The 8800 offers a unique approach to removing interferences and investigating reaction chemistry, and opens an exciting new chapter in the ICP-MS story.

# On-line Isotope Dilution Analysis of Selenium using the Agilent 8800 ICP-QQQ

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## Introduction

Selenium (Se) is a difficult element to quantify accurately by conventional ICP-MS for several reasons. Firstly, the required Limit of Quantification (LOQ) range for Se is often extremely low. Secondly, Se has a high ionization potential (IP = 9.75 eV), which reduces its ionization in the plasma, causing low sensitivity, increasing susceptibility to matrix suppression, and making it more difficult to find a suitable Internal Standard (ISTD) element. Thirdly, all Se isotopes suffer from severe interferences.

Isotope Dilution (ID) methodology is well known for its absolute accuracy due to its ability to minimize the effects of signal drift and matrix suppression, which are common problems in ICP-MS. However the application of isotope dilution to Se quantification has been limited because of the high number of potential interferences that affect all four analytically useful Se isotopes. IDA requires two isotopes that are free of interferences.

## Cell Gas Mode Investigation

An investigation to determine the optimum 8800 ORS<sup>3</sup> cell gas conditions and the best Se isotopes for the online isotope dilution analysis (OIDA) method showed that O<sub>2</sub>/H<sub>2</sub> yielded the best background equivalent concentrations (BECs) for the Se isotopes: <sup>80</sup>Se (49.61% abundant) and <sup>78</sup>Se (23.77%), and <sup>82</sup>Se (8.73%), measured as the SeO<sup>+</sup> cell-formed product ions, than either gas alone.

RF power (W)	1550
Sampling depth (mm)	8
Carrier gas (L/min)	1.05
Scan mode	MS/MS
KED (V)	-6
Cell gas	O <sub>2</sub> and H <sub>2</sub>
Cell gas flow (mL/min)	O <sub>2</sub> = 0.4 H <sub>2</sub> = 2

Table 1. 8800 ICP-QQQ operating conditions

SeO<sup>+</sup> measurement with O<sub>2</sub> cell gas can be used with conventional collision/reaction cell ICP-MS, but the method is limited if the sample contains Zr, Mo or Ru since their isotopes overlap with the target SeO<sup>+</sup> ions at m/z 94, 96 and 98. In contrast, with the 8800 operating in MS/MS mode, the first quadrupole (Q1) rejects all the potential overlapping ions, so the method can be applied to samples regardless of their composition.

## Selenium Online Isotope Dilution Analysis

A <sup>82</sup>Se enriched standard (<sup>82</sup>Se 97.43%, <sup>80</sup>Se 1.65% and <sup>78</sup>Se 0.51%) was purchased from Oak Ridge National Laboratory (USA), dissolved in ultrapure HNO<sub>3</sub> and diluted to the appropriate concentration. This spike solution was then added online to all samples using the Agilent ISTD mixing kit. Isopropyl alcohol (IPA) was added via the spike solution to give a final concentration of about 1% in the sample, to enhance the ionization of Se in the plasma. Refer to reference 1 for details of OIDA.

## Analytical Results

The concentration of Se was determined in twelve different certified reference materials (CRMs) using the developed OIDA method. The tested samples were all diluted so that the expected Se concentration fell between 100 ppt and 5 ppb. An integration time of 1 sec. was used for each of the three Se isotopes

measured, and ten replicates were acquired for averaging. This provided the measured isotope ratio value <sup>4</sup>R<sub>m</sub>. The Se concentration was then calculated by applying the IDA formula [1].

Figure 1 shows the Se quantification results expressed as recovery % relative to the certified value. The measured results for Se were in good agreement with the CRM values (90-112%) for all samples including those where REE<sup>++</sup> interferences could have caused errors.

## Conclusions

The accurate determination of selenium has been demonstrated using online-IDA with the new Agilent 8800 ICP Triple Quad. The ability of the 8800 to control the ions that enter the ORS<sup>3</sup> using Q1 located before the cell fundamentally changes the way that reaction chemistry is performed with ICP-MS. The Agilent 8800 provides consistent reaction processes in complex and variable samples, and offers much more selective interference removal than is possible with conventional quadrupole ICP-MS.

In this study, it was found that using O<sub>2</sub>/H<sub>2</sub> as a reaction gas in the ORS<sup>3</sup> can effectively solve the interferences on Se isotopes caused by REE<sup>++</sup>.

The new method can also be applied to samples containing Zr, Mo and Ru, as they are removed by the first quadrupole. Further it was demonstrated that online IDA using O<sub>2</sub>/H<sub>2</sub> cell gas with the new Agilent 8800 ICP Triple Quad can be used for accurate Se quantification in a wide range of CRMs.

## References

- Giuseppe Centineo, et al, Agilent publication, 5990-9171EN, 2011, available from [www.agilent.com/chem/icpms](http://www.agilent.com/chem/icpms)
- A. Henrion, Fresenius J Anal Chem, 1994, 350, 657-658

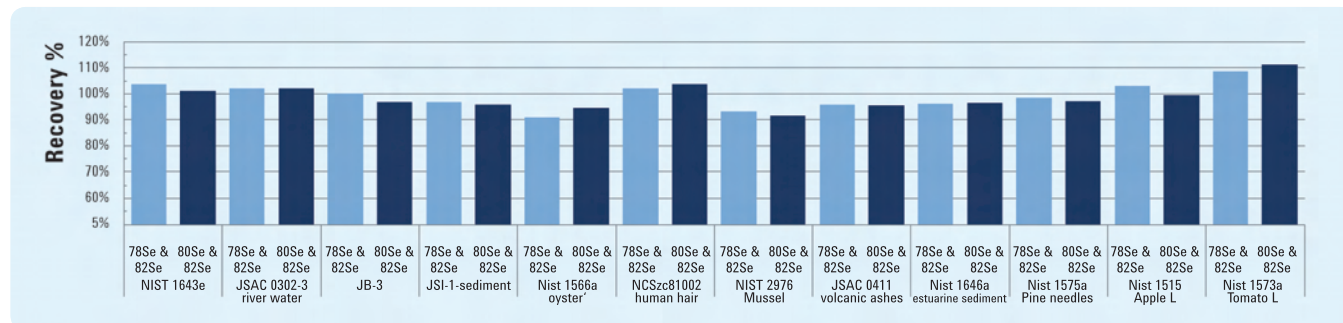


Figure 1. Agilent 8800 MS/MS mode results of Se determination in twelve CRMs by OIDA using O<sub>2</sub> + H<sub>2</sub> mass shift method



# Analysis of Highly Purified Chemicals using the 8800 ICP Triple Quad with MS/MS Mode

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## Introduction

The semiconductor industry requires the assay of elements at ultra-trace levels in a range of highly pure chemicals used during the manufacturing of semiconductor devices. With its exceptional sensitivity and wide elemental coverage, ICP-MS is the standard technique used for this application. However, some elements are difficult to analyze at low enough levels in certain matrices using conventional quadrupole ICP-(Q)MS. These problematic elements can now be measured reliably with the Agilent 8800 ICP Triple Quad.

## Instrumentation

An Agilent 8800 was used for all measurements. A PFA nebulizer, standard quartz spray chamber and 2.5 mm injector quartz torch was used for the analysis of HCl and H<sub>2</sub>SO<sub>4</sub> and an inert sample introduction kit consisting of a PFA spray chamber and platinum injector torch was used for the analysis of samples that contain HF. A Pt sampling cone with Cu base and a Pt skimmer cone with Ni base were used for all measurements. The ORS<sup>3</sup> reaction gases used were oxygen (100%), hydrogen (100%) and ammonia (10% in He). The metallic impurities of all the chemicals used for the analysis were guaranteed to be less than 100 ppt by the manufacturers.

## V, Cr, Ge and As in 20% HCl

The major spectral interferences seen in Cl-matrices include <sup>35</sup>Cl<sup>16</sup>O

	V	Cr	Ge	As
ORS <sup>3</sup> gas	NH <sub>3</sub>	NH <sub>3</sub>	O <sub>2</sub>	O <sub>2</sub>
Q1/Q2	<sup>51</sup> V/ <sup>51</sup> V	<sup>52</sup> Cr/ <sup>52</sup> Cr(NH <sub>3</sub> ) <sub>2</sub>	<sup>74</sup> Ge/ <sup>74</sup> Ge <sup>16</sup> O	<sup>74</sup> Ge/ <sup>74</sup> Ge <sup>16</sup> O <sub>2</sub>
BEC (ppt)	0.4	13	4	3
DL (ppt)	0.4	8	1.5	2.5

Table 1. BEC and DLs for V, Cr, Ge and As in 20% HCl using the 8800 ICP Triple Quad

on <sup>51</sup>V, <sup>35</sup>Cl<sup>16</sup>O on <sup>52</sup>Cr, <sup>35</sup>Cl<sup>37</sup>Cl on <sup>72</sup>Ge, <sup>37</sup>Cl<sup>37</sup>Cl on <sup>74</sup>Ge and <sup>40</sup>Ar<sup>35</sup>Cl on <sup>75</sup>As. The Background Equivalent Concentrations (BECs) and 3 sigma Detection Limits (DLs) of these elements in 20% HCl measured using the 8800 in MS/MS mode were at the single or sub-ppt level (Table 1).

## P, Ti and V in 10x Diluted H<sub>2</sub>SO<sub>4</sub>

Certain elements are difficult to analyze at the ppt level in H<sub>2</sub>SO<sub>4</sub> because of spectral interferences from <sup>32</sup>S<sup>16</sup>O on <sup>48</sup>Ti and <sup>34</sup>S<sup>16</sup>O on <sup>51</sup>V. These elements, together with phosphorus (subject to interference by <sup>14</sup>N<sup>16</sup>O and <sup>15</sup>N<sup>16</sup>O) were analyzed in 9.8% H<sub>2</sub>SO<sub>4</sub> by the 8800 in NH<sub>3</sub> reaction mode. The BECs are compared with those achieved using the Agilent 7700s ICP-MS in He collision mode (Table 2).

	<sup>31</sup> P	<sup>48</sup> Ti	<sup>51</sup> V
ORS <sup>3</sup> gas	NH <sub>3</sub>	NH <sub>3</sub>	NH <sub>3</sub>
Q1/Q2	<sup>31</sup> P/ <sup>31</sup> P <sup>14</sup> NH <sub>3</sub>	<sup>48</sup> Ti/ <sup>48</sup> Ti <sup>14</sup> NH	<sup>51</sup> V/ <sup>51</sup> V
BEC	0.16 ppb	2 ppt	0.1 ppt
7700s BEC	20 ppb	60 ppt	3 ppt

Table 2. BECs for P, Ti, and V in 9.8% H<sub>2</sub>SO<sub>4</sub> using the 8800 in NH<sub>3</sub> reaction mode

## P and Ti in 2000 ppm Si Matrix

Most manufacturers of semiconductor devices routinely analyze Si VPD samples which contain HF and may have a Si matrix as high as 2000 ppm. This matrix gives rise to polyatomic ions that interfere with <sup>31</sup>P (<sup>30</sup>SiH) and <sup>48</sup>Ti (<sup>29</sup>Si<sup>19</sup>F and <sup>28</sup>SiFH). The 8800 was equipped with a low uptake nebulizer (50 uL/min) and robust plasma conditions were applied for the analysis of the high Si matrix samples. The calibration curves (Figures 1 and 2) demonstrate the effective elimination of all interferences on P (using H<sub>2</sub> as the reaction gas) and Ti (using NH<sub>3</sub> as the reaction gas) in a 2000 ppm Si matrix.

## Conclusions

The 8800 ICP-QQQ works very well

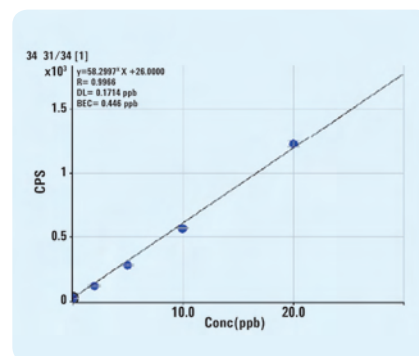


Figure 1. Calibration curve of <sup>31</sup>P in a 2000 ppm silicon matrix

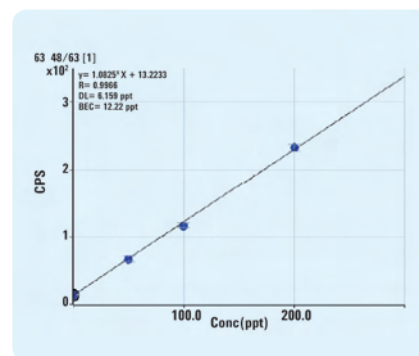


Figure 2. Calibration curve of <sup>48</sup>Ti in a 2000 ppm silicon matrix

to address spectral interferences which cannot be solved by ICP-QMS collision/reaction cell technology. The 8800's unique tandem MS configuration enables MS/MS operation, giving precise control of reaction processes in the ORS<sup>3</sup> and providing more effective interference removal and therefore more reliable data than the equivalent cell gas modes used in conventional quadrupole ICP-MS. MS/MS mode controls the ions that can enter the cell, making it possible to determine ultra trace impurities even in complex mixtures of high purity chemicals such as HF, HNO<sub>3</sub>, HCl, H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub>, and in high Si matrices typical of VPD samples.

## Available Now: Second Edition of Agilent's Speciation Handbook

**Steven Wilbur** Senior Applications Chemist, Agilent Technologies



The 2<sup>nd</sup> edition of Agilent's Handbook of Hyphenated ICP-MS Applications is now available in print, electronically as a pdf file (5989-9473EN), and as a new interactive e-book. Find out more at: [www.agilent.com/chem/speciation2](http://www.agilent.com/chem/speciation2)

The new Handbook is divided into chapters based on the hyphenated instrument configuration, LC-ICP-MS, GC-ICP-MS, LC-Multi-MS, and a newly added chapter devoted to field flow fractionation (FFF)-ICP-MS primarily for the analysis of nanoparticles. Each chapter includes an introduction to the technique by a well known researcher, followed by contributed application notes. The GC and LC sections also include detailed troubleshooting sections designed to help users to resolve problems and get the best performance from their systems.

Publishing the Handbook as an e-book gives us the ability to make it a living document, which can be easily updated as new technologies and applications arise. For example, we will be adding ICP Triple Quad speciation applications as they are developed using the new Agilent 8800. Being able to easily update the Handbook in real time will also allow us to incorporate user feedback into the troubleshooting sections.

This information is subject to change without notice.

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## Agilent 8800 ICP-QQQ Draws a Crowd at WPC 2012



More than 80 delegates gathered to hear Agilent's Ed McCurdy introduce the new 8800 ICP Triple Quad during a lunch-time seminar at the 2012 Winter Plasma Conference, held in Tucson, Arizona, in January. Feedback on the new instrument from attendees at the conference was overwhelmingly positive, as analysts recognize the potential of the 8800 for solving many of the more challenging analytical problems that existing ICP-MS systems can't address.

Agilent specialists presented 2 oral and 5 poster presentations further explaining the technology and application capability of the 8800. In addition, there were 6 poster presentations by Agilent chemists on the Agilent 7700 Series ICP-MS, which remains the benchmark for high-performance, cost-effective ICP-QMS, delivering unmatched interference removal in He mode.

To learn more about the 8800, go to: [www.agilent.com/chem/icpqqq](http://www.agilent.com/chem/icpqqq)

## Conferences. Meetings. Seminars.

- **ACS Spring:** Mar 25-29, San Diego, CA, USA, [www.acs.org](http://www.acs.org)
- **Arab Lab:** March 26-29, Dubai, UAE, [www.arablab.com](http://www.arablab.com)
- **Analytica:** April 17-20, Munich, Germany, [www.analytica.de/en](http://www.analytica.de/en)  
[Visit Agilent in Hall A2, Booth 105/206](http://www.analytica.de/en)
- **ASMS Conference:** May 20-24, Vancouver, BC, Canada, [www.asms.org](http://www.asms.org)
- **Nordic Plasma Conference:** June 10-13, Loen, Norway, [www.nordicplasma.com](http://www.nordicplasma.com)
- **Spectr'Atom:** June 19-22, Pau, France, [www.spectratom.com](http://www.spectratom.com)
- **11th European Workshop on Laser Ablation:** June 19-22, [www.ewla.es](http://www.ewla.es)
- **Interphex Japan:** June 27-29, Tokyo, [www.interphex.jp/en](http://www.interphex.jp/en)
- **NEMC 2012:** August 6-10, Washington DC, [www.nemc.us](http://www.nemc.us)
- **Asia Pacific Winter Conference:** 26-29 Aug, Jeju, Korea, <http://apwc2012.dankook.ac.kr/>
- **JASIS (formerly JAIMA):** Sept 5-7, Tokyo, Japan, [www.jasis.jp/2012/english/index.html](http://www.jasis.jp/2012/english/index.html)
- **21st International Symposium of Forensic Sciences ANZFSS:** Sept 23-27, Hobart, Tasmania, [www.anzfss2012.com.au](http://www.anzfss2012.com.au)

## Agilent ICP-MS Publications

To view and download the latest ICP-MS literature, go to [www.agilent.com/chem/icpms](http://www.agilent.com/chem/icpms) and look under "Literature Library".

- **Primer:** Handbook of Hyphenated ICP-MS Applications, 5989-9473EN
- **Application note:** Low-level speciated analysis of Cr(III) and Cr(VI) using LC(IC)-ICP-MS, 5990-9366EN
- **Application note:** Benefits of the HPLC-ICP-MS coupling for mercury speciation in food, 5991-0066EN
- **Application note:** Trace elemental analysis of distilled alcoholic beverages using 7700x ICP-MS, 5990-9971EN

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