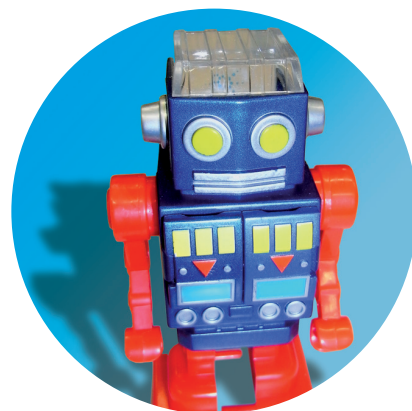


Agilent ICP-MS Journal

September 2009 – Issue 39



**7700
Series
ICP-MS
Special
Issue**

Inside this Issue

- 2 Clearly Better ICP-MS: Introducing the New 7700 Series, Agilent 7700 Series Stars on YouTube
- 3 New ORS Insures More Effective Interference Removal in Complex Samples
- 4-5 Improved Data Quality and Productivity with 7700x Analysis of Drinking Water
- 6-7 Simple, Rapid Analysis of Trace Metals in Foods using the 7700x
- 8 New! 7700 Interactive Web Animation, Free ICP-MS e-Seminar Series, Conferences, Meetings, Seminars, New ICP-MS Publications, Free Wall Poster



Agilent Technologies

Introducing the New 7700 Series

Don Potter and Ed McCurdy

ICP-MS Product Marketing, Agilent Technologies

ICP-MS performance has been steadily improving since the technique was developed over 20 years ago. However, some of the weaknesses that were identified in the very earliest commercial instruments can still cause analytical problems today.

Agilent has been leading the development of ICP-MS since the early days of the technique, and our most recent innovations have resulted in the new Agilent 7700 Series (Figure 1). Replacing the 7500 Series (the most widely used ICP-MS ever), the 7700 Series – comprising the 7700x workhorse and 7700s semiconductor configured mainframe, finally delivers the performance that ICP-MS has always promised.

Key technology and performance of the new 7700 Series includes:

- New, fast frequency-matching RF generator, which will tolerate highly volatile organic solvents
- High Matrix Introduction (HMI) capability (standard on the 7700x)
- Redesigned ion lens that delivers higher sensitivity and lower background
- 3rd generation Octopole Reaction System (ORS³), providing dramatically improved performance in helium (He) collision mode
- Nine orders dynamic range detector – from DL to >500 ppm
- New Agilent MassHunter software with Excel 2007, for comprehensive data analysis and reporting functions
- 30% smaller than the 7500 Series, and weighing only 115 kg (253 lbs).

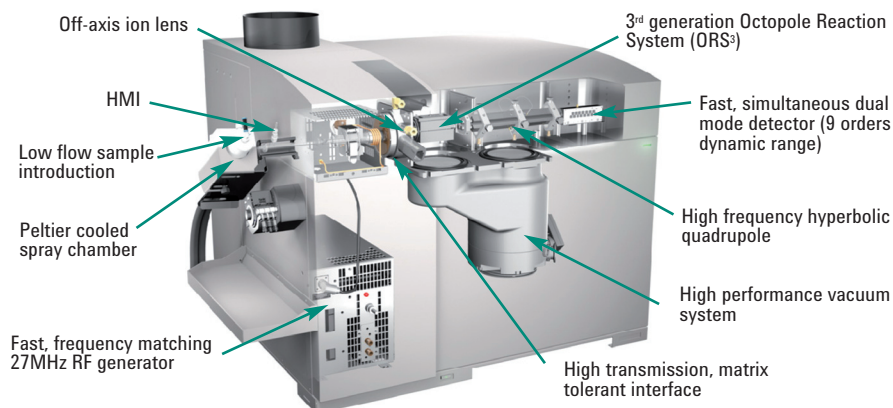


Figure 1. Agilent 7700x ICP-MS in detail

Agilent 7700x – workhorse unit for reliable high throughput analysis of unknown samples

These developments allow the 7700x to address the three main practical limitations of most modern ICP-MS instruments, which are poor matrix tolerance, limited dynamic range and errors due to polyatomic interferences:

- 1) The 7700x with HMI handles much higher matrix levels than any other ICP-MS (% TDS), which previously required dilution or analysis by ICP-OES.
- 2) The 7700x has an unparalleled 9 orders dynamic range, from sub-ppb to >500 ppm, in a single acquisition.
- 3) ORS³ in He mode provides effective removal of all polyatomic interferences, without the complexity and uncertainty associated with reactive cell gases.

The 7700x provides more accurate data across a wider range of analyte concentrations and sample matrix types, while simplifying method development and routine operation. At the same time, the 7700x retains the performance and flexibility required for advanced research applications.

Agilent 7700s - unparalleled semiconductor analysis

Based on the same mainframe as the 7700x, the 7700s is configured for analysis of high-purity materials such as those monitored in the semiconductor industry. With a high efficiency sample introduction system, plus 5th gas line (typically used for oxygen 20%) and second (reaction) cell gas line fitted as standard – the 7700s offers high performance for the removal of intense interferences in known and consistent matrices.

Agilent 7700 Series Stars on YouTube

Mina Tanoshima

ICP-MS Product Marketing, Agilent Technologies



Meet Bill, Agilent's testing robot (front cover photo), in this new Agilent 7700 Series ICP-MS video, now available on YouTube.

Shot on location in Tokyo, the 7 minute clip features interviews with five Agilent ICP-MS R&D scientists, each explaining different aspects of the 7700 design and the key new features of the system.

Bill the Robot makes his appearance during the stringent quality and durability testing undertaken by the new instrument and its accessories. The punishing testing which took place at an independent test center included a 1 m drop shock test, a separate drop test onto concrete, with and without packaging, and a rigorous vibration test (enjoyed by Bill). Following all of this upheaval, the 7700 was powered up and the plasma ignited first time!

To see the video, go to the YouTube web site at www.youtube.com and search on Agilent 7700 ICP-MS or use the direct url:

www.youtube.com/watch?v=1nNXzR9MYCQ

New ORS Insures More Effective Interference Removal in Complex Samples

Naoki Sugiya and Ed McCurdy
ICP-MS Product Marketing, Agilent Technologies

The 3rd generation Octopole Reaction System (ORS³) featured in the 7700 Series has been developed to give the highest performance in helium (He) collision mode, using kinetic energy discrimination (KED). This requires:

- 1) Extremely narrow initial ion energy spread (delivered by the ShieldTorch System), and
- 2) High ion transmission at the high cell gas pressure needed for effective KED (provided by the small internal volume, octopole-based ORS³ cell).

KED makes use of the fact that polyatomic (or molecular) ions are larger than analyte ions of the same mass, and so collide more frequently with the He cell gas, losing energy with each collision. By the cell exit, the polyatomic ions have less residual energy than the analyte ions and can be stopped from entering the quadrupole by applying a fixed bias voltage “step”.

Helium Mode in the ORS³

The spectra in Figure 1 illustrate the relative performance of different cell modes for the removal of multiple interferences on the 1st row transition elements.

The spectra show no gas mode (top), H₂ reaction mode (middle) and He mode (bottom) for the same matrix (5% HNO₃, 5% HCl, 1% H₂SO₄ and 1% IPA), on the same scale.

The complex polyatomic interferences created in this matrix mix are evident in the no gas mode spectrum shown in Figure 1 (top).

H₂ reaction mode (middle spectrum) leaves some residual overlaps, and creates several new ones. By contrast, the bottom spectrum in Figure 1 confirms that He mode effectively removes all the matrix-based polyatomic interferences in this complex matrix.

High sensitivity is maintained in He

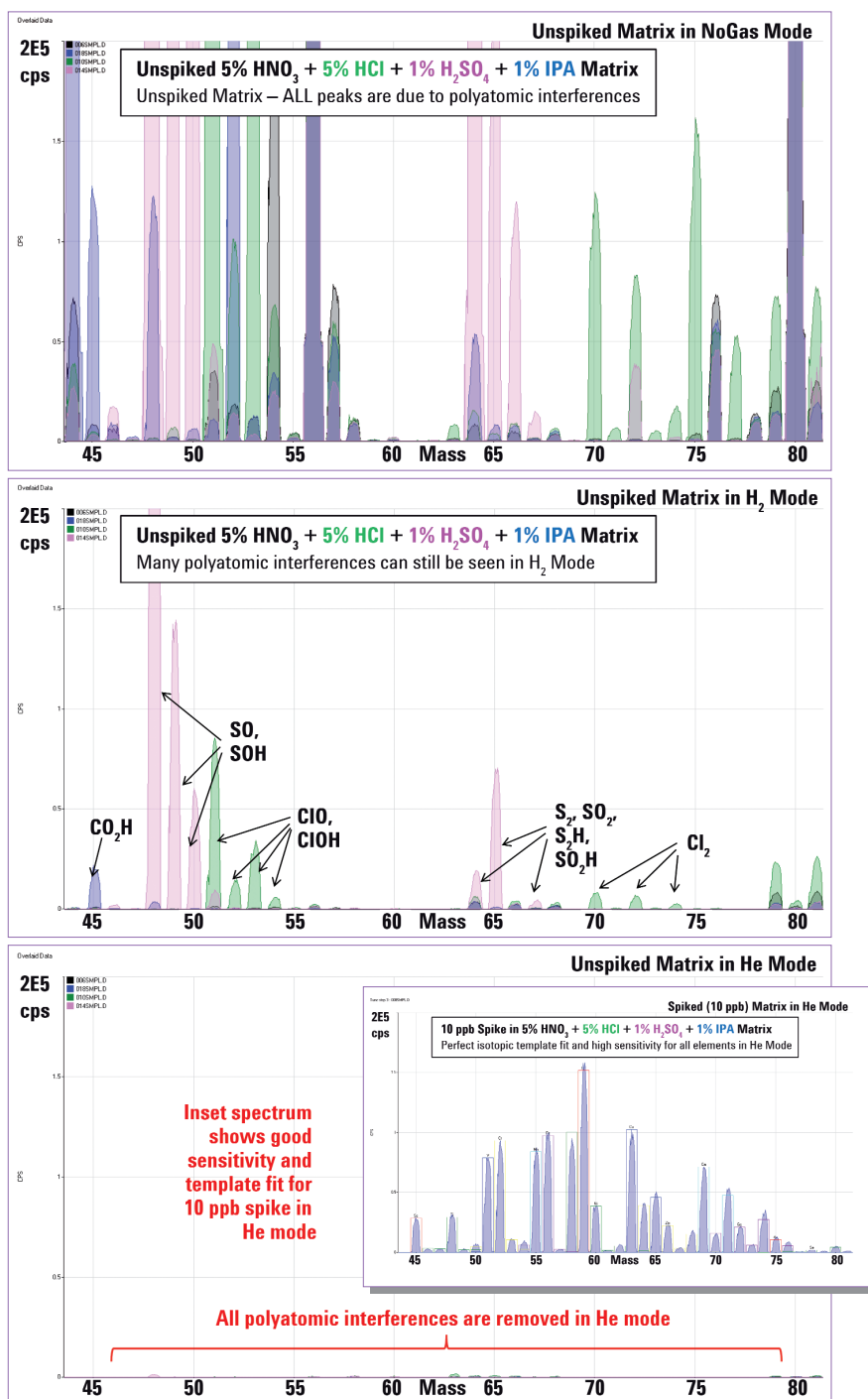


Figure 1. Mixed matrix in no gas, H₂ and He mode (inset: with 10 ppb spike)

mode, as shown by the inset spectrum of a 10 ppb spike, measured under the same conditions (bottom inset).

Conclusions

The spectra illustrate two of the unique capabilities of He mode; it removes polyatomic interferences on multiple analytes and in multiple matrices, and it is inert so it does not create any new interferences. This means that 7700x ORS³ users can report reliable multi-element

data across a range of sample types, using simple instrument conditions that are independent of analyte or sample type.

Further reading

Agilent publication: Comparing Collision/Reaction Cell Modes for the Measurement of Interfered Analytes in Complex Matrices using the Agilent 7700 Series ICP-MS, 5990-3236EN

Improved Data Quality and Productivity with 7700x Analysis of Drinking Water

Steven Wilbur ICP-MS Specialist, Agilent Technologies

Introduction

The Agilent 7700x ICP-MS brings 3rd generation Octopole Reaction System (ORS³) technology to routine environmental analysis of trace metals in drinking waters. Building on the cell technology used in the successful Agilent 7500 Series ICP-MS, the ORS³ has been completely redesigned to significantly improve the performance of helium (He) collision mode for the removal of polyatomic interferences. He mode is simple and universal, enabling the 7700x ICP-MS to remove all polyatomic interferences under a single gas mode without requiring any previous knowledge about the sample, and without the need for complex mathematical corrections. The result is easier set-up, lower detection limits, faster analysis, and improved interference removal, especially for the difficult plasma based interferences affecting iron and selenium. Selenium detection limits less than 20 parts per trillion (ppt) are now routinely achievable in He mode.

Better Sensitivity, Better Accuracy, Better Dynamic Range

In addition to a more efficient ORS, the 7700x ICP-MS also includes a new interface and ion lens which deliver higher sensitivity and lower background compared to the 7500cx ICP-MS it replaces. No special tuning or optimization is required for the routine analysis of water samples ranging from clean municipal tap waters to high total dissolved solids (TDS) mineral waters. Complete instrument optimization is fully automated, including the new One-click Plasma Setting function for fast, automated plasma setup, and a faster, more reproducible Expert auto tune function.

Figure 1 shows typical calibrations obtained with the 7700x ICP-MS. All calibration standards contained 1% HNO₃ and 0.5% HCl in order to stabilize Ag, Sb and Hg. While the use of HCl has traditionally been

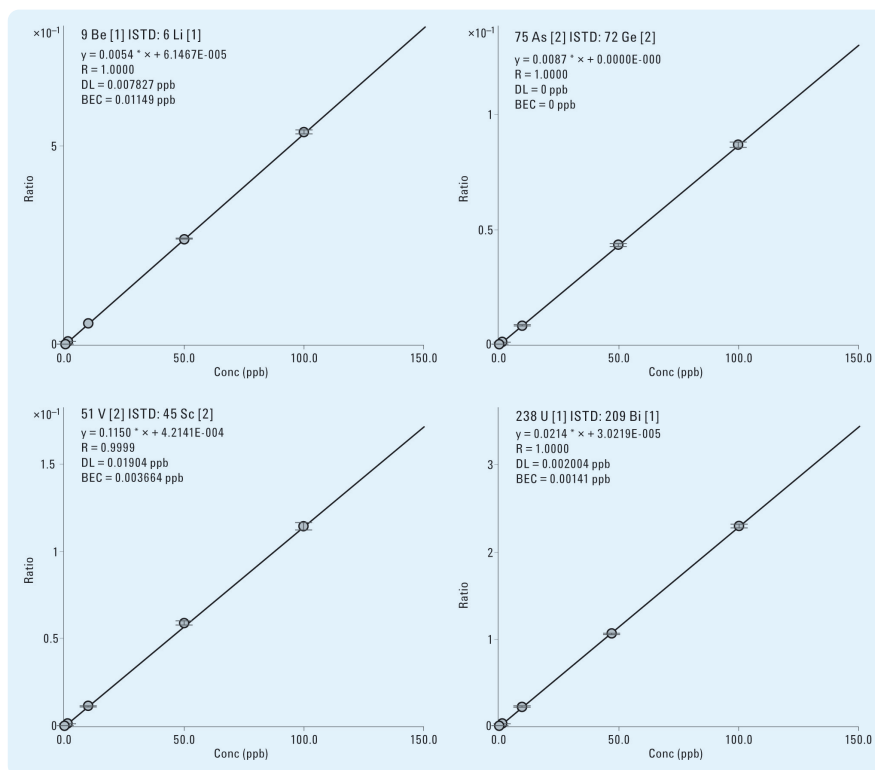


Figure 1. Representative calibration curves for drinking water method. Be and U were analyzed in no gas mode, while As and V were analyzed in He mode. All standards contain 1% HNO₃ and 0.5% HCl. BEC and DL for As calculated based on 0 cps +/- 0 as acquired. Actual BEC and DL would have required longer counting times than those used in this method.

avoided in ICP-MS due to the formation of Cl based interferences on As, Se, Cr and V, the ORS³ is able to remove these interferences in He mode, eliminating the need for hydrogen or other reactive gases. The 3.7 ppt background equivalent concentration (BEC) for vanadium demonstrates the efficient removal of the ClO⁺ interference (mass 51) in He mode on the 7700x. This interference is difficult to remove by other means, typically requiring highly reactive

cell gases such as NH₃ or NH₃/He, which are not suitable for multi-element analysis.

Typical method detection limits (MDLs, 3 sigma of 10 replicates of the low calibration standard) in ppt are shown in Table 1. These MDLs were not generated under highly optimized conditions, but using the default robust plasma conditions obtained by the One-click Plasma Setting routine (Ce⁺/Ce⁺ <1%).

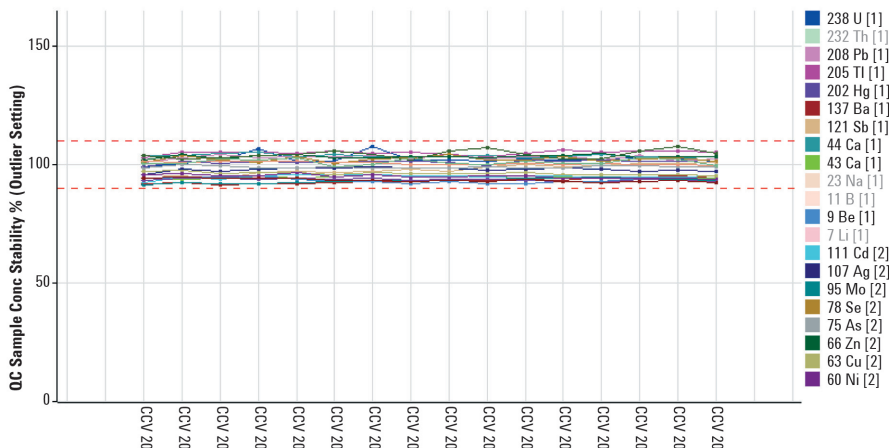


Figure 2. CCV recoveries (20 ppb) for all method analytes for the entire 190 sample sequence. No CCV failures occurred. Mode [1] = no gas, Mode [2] = He mode

| Mass | Element | MDL (ppt) | Cell mode |
|------|---------|-----------|-----------|
| 9 | Be | 5.2 | No gas |
| 11 | B | 5.0 | No gas |
| 23 | Na | 58.5 | No gas |
| 24 | Mg | 2.8 | No gas |
| 27 | Al | 7.9 | No gas |
| 39 | K | 76.9 | He |
| 42 | Ca | 57.8 | He |
| 51 | V | 14.3 | He |
| 52 | Cr | 4.3 | He |
| 55 | Mn | 8.5 | He |
| 56 | Fe | 14.8 | He |
| 59 | Co | 4.4 | He |
| 60 | Ni | 14.7 | He |
| 63 | Cu | 2.7 | He |

| Mass | Element | MDL (ppt) | Cell mode |
|------|---------|-----------|-----------|
| 66 | Zn | 14.0 | He |
| 75 | As | 11.9 | He |
| 78 | Se | 17.6 | He |
| 88 | Sr | 2.1 | He |
| 95 | Mo | 6.9 | He |
| 107 | Ag | 2.3 | He |
| 111 | Cd | 2.9 | He |
| 121 | Sb | 6.1 | He |
| 137 | Ba | 5.7 | He |
| 202 | Hg | 1.2 | He |
| 205 | Tl | 2.4 | He |
| 208 | Pb | 1.3 | He |
| 232 | Th | 1.8 | He |
| 238 | U | 1.7 | He |

Table 1. The 3 sigma Method Detection Limits in ppt for trace elements in drinking waters. Note Fe and Se detection limits are less than 20 ppt in He Mode. Note excellent DL for Be and B at 5 ppt.

The high sensitivity of the 7700x ICP-MS means that accurate analysis of trace level contaminants in waters can be accomplished with ease, while the built-in High Matrix Introduction (HMI) system insures superior robustness and long term stability when running long sequences of high matrix samples. A sequence consisting of drinking waters, two high TDS mineral waters – samples "A" (TDS of 109 mg/L) and "B" (TDS of 309 mg/L), and NIST 1643e reference water (diluted 1/10) was analyzed continuously for 11 hours. The sequence included USEPA mandated continuing calibration verification and continuing calibration blank samples (CCVs and CCBs), giving a total of 190 analyses, performed with an average run time of less than 3.5 minutes per sample. CCV recoveries for all method analytes are plotted in Figure 2 for the entire sequence. No element in any CCV exceeded the USEPA limits of +/-10% (shown in red).

Conclusions

The new ORS³, ion optics and interface of the 7700x have produced an instrument that is fully capable of measuring all required elements at ppt levels in drinking waters without the need for either reactive cell gases or complex interference equations. The result is absolute confidence in data quality with significantly improved productivity. The 7700x has the largest operating dynamic range of any quadrupole ICP-MS, and the integrated HMI system provides ultimate tolerance to high matrix samples. Furthermore, the simple optimization conditions used for drinking water analysis can also be applied to more complex environmental samples such as soils and sludges, eliminating the need for the sample specific optimizations which are required on instruments that can only use reactive cell gases.

Further reading:

Agilent publication: The Agilent 7700x ICP-MS Advantage for Drinking Water Analysis, 5990-4315EN

Simple, Rapid Analysis of Trace Metals in Foods using the Agilent 7700x ICP-MS

Steve Wilbur and Michiko Yamanaka
ICP-MS Applications Specialists, Agilent Technologies

Introduction

The Agilent 7700x ICP-MS is capable of accurately analyzing a variety of trace and major elements in foods, using a single, simple, collision cell method. This approach permits large numbers of samples to be quickly screened for total toxic metals. Samples which are found to contain trigger levels of certain metals where the chemical form (or species) is important can then be further analyzed using Agilent supported hyphenated ICP-MS techniques such as LC-ICP-MS or GC-ICP-MS.

Experimental

In order to test the ability of the Agilent 7700x to analyze a variety of foods for a wide range of metals at highly variable concentrations, several certified reference food samples were analyzed. The 7700x was tuned using One-click Plasma setting for robust plasma conditions and autotuned for optimum sensitivity,

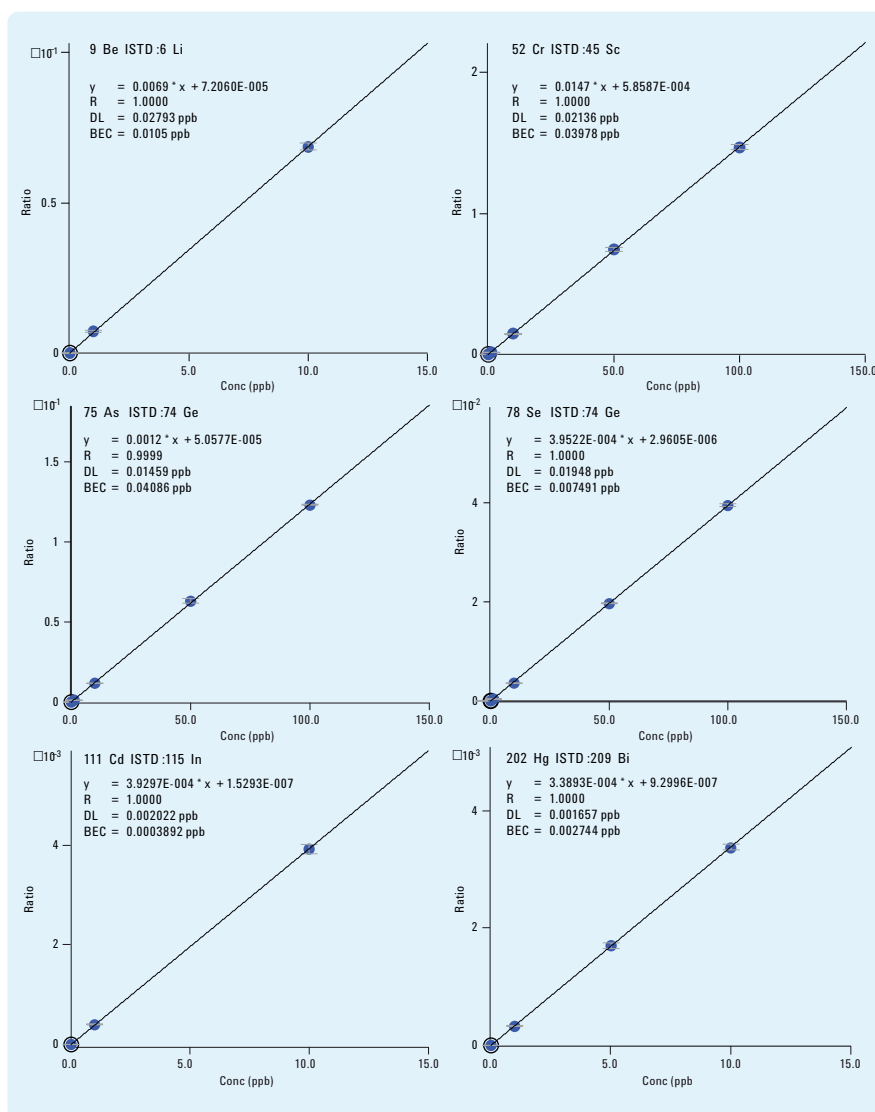


Figure 1. Calibrations for Be, Cr, As, Se Cd and Hg.

mass response and minimal interferences. Operating conditions are shown in Table 1. In order to keep the method as quick and simple as possible, the Octopole Reaction System (ORS³) was operated in a single gas mode, using helium (He) cell gas, which provides a reliable and effective cell method to remove all polyatomic interferences, regardless of the analyte or matrix composition. The following acquisition masses and integration times provided more than sufficient sensitivity to meet all certified values. Total run time per sample was less than 3 minutes.

Example calibration curves for several critical and/or difficult elements are shown in Figure 1.

Traditionally, covering this range of concentrations for these elements would have required ICP-OES for the major elements (Na and Ca), graphite furnace AA for Pb and Cd, either a dedicated Hg analyzer or cold vapor AA for Hg and possibly hydride AA for As and Se. The Agilent 7700x ICP-MS running in He mode was able to measure all elements in a single run easily. Even elements such as Be and Hg, which are free of common polyatomic interferences and would typically be acquired under no gas conditions, demonstrate excellent sensitivity in He mode (Be detection limit = 28 ppt, Hg detection limit = 1.6 ppt).

| Parameter | Value | |
|---------------------------------------|-------|-------|
| RF power (W) | 1550 | |
| Carrier gas flow (L/min) | 0.99 | |
| Spray chamber temp (°C) | 2 | |
| Sample depth (mm) | 8 | |
| Extract 1 lens (V) | 0 | |
| CeO ⁺ /Ce ⁺ (%) | 1.1 | |
| Ce ⁺⁺ /Ce ⁺ (%) | 1.9 | |
| Sensitivity cps/ppb | Li | 62700 |
| | Y | 92920 |
| | Tl | 87080 |

Table 1. 7700x operating conditions set by One-click Plasma Setting and Autotune

| Mass/ element | NRC-CNRC DORM3 Fish Protein | | NIST SRM 2976 Mussel Tissue | | NIST RM 8415 Whole Egg Powder | |
|------------------|--------------------------------|---------------------|--------------------------------|---------------------|----------------------------------|---------------------|
| | Certified value (mg/kg) | Measured (mg/kg) | Certified value (mg/kg) | Measured (mg/kg) | Certified value (mg/kg) | Measured (mg/kg) |
| 23 Na | - | | - | | 3770 ± 340 | 3807 |
| 43 Ca | - | | - | | 2480 ± 190 | 2703 |
| 52 Cr | - | | - | | 0.37 ± 0.18 | 0.344 |
| 55 Mn | - | | - | | 1.78 ± 0.38 | 1.64 |
| 56 Fe | 347 ± 20 | 324.0 | 171 ± 4.9 | 158.5 | - | |
| 60 Ni | 1.28 ± 0.24 | 1.29 | - | | - | |
| 63 Cu | 15.5 ± 0.63 | 14.4 | 4.02 ± 0.33 | 3.32 | 2.7 ± 0.35 | 2.61 |
| 66 Zn | 51.3 ± 3.1 | 45.86 | 137 ± 13 | 121.2 | - | |
| 75 As | 6.88 ± 0.3 | 6.15 | 13.3 ± 1.8 | 12.57 | - | |
| 78 Se | - | | 1.8 ± 0.15 | 1.87 | 1.39 ± 0.17 | 1.25 |
| 95 Mo | - | | - | | 0.247 ± 0.023 | 0.215 |
| 111 Cd | 0.29 ± 0.02 | 0.28 | 0.82 ± 0.16 | 0.794 | - | |
| 202 Hg | 0.355 ± | 0.359 | 0.061 ± | 0.068 | - | |
| 208 Pb | 0.395 ± | 0.398 | 1.19 ± 0.18 | 1.163 | 0.061 ± 0.012 | 0.055 |

Table 2 Measured and certified values for three certified reference food materials. Recoveries are dependent on digestion efficiency as well as analytical accuracy. All measured values are based on dry sample weight corrected for percent moisture. All certified elements are reported for each sample, not all samples are certified for all elements.

The food certified reference materials were analyzed directly after microwave digestion. Between 0.5 g and 1 g of each sample was weighed (after determination of percent moisture) and digested using 6 mL of HNO₃ + 2 mL of H₂O₂ using microwave assisted digestion. All samples were brought to final volume of 100 mL using ultrapure water. Results are shown in Table 2. The trace elements, Ni, Mn, Cu, As, Se, Cd, Hg and Pb exhibited excellent agreement with the certified values for all three samples. Slight deviations from certified values for Fe, Ca and Zn were attributed to the digestion procedure rather than the analytical measurement.

Conclusions

Multiple sample preparation procedures and analytical techniques are no longer required for the analysis of typical food samples. Instead a simple method based on microwave digestion and

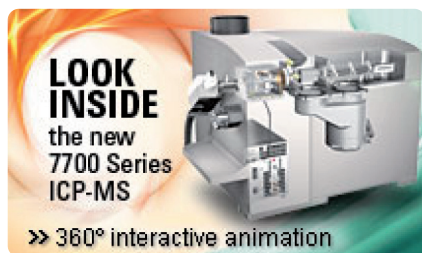
ICP-MS analysis can be used to analyze foods quickly and accurately for trace and major element concentrations.

Using a single cell gas mode (He mode), the Agilent 7700x provides sensitive, accurate, interference free analysis of a variety of metals in common foods. Because He mode is both sensitive and universal, it is applicable to trace analysis of all metals in any food sample digest. No prior information about the sample matrix or composition is required.

Further reading:

Agilent publication: Simple, Rapid Analysis of Trace Metals in Foods Using the Agilent 7700x ICP-MS, 5990-4539EN

New! 7700 Interactive Web Animation



Visit www.agilent.com/chem/icpms and check out the 7700 Series with the interactive animation:

- Rotate the ICP-MS 360° for a full view of the outer cabinet and back panel.
- Watch a simulation of the cooling air flow through the compact system
- Get full details on 11 major components of the 7700 – from the HMI and ORS³ to the Hyperbolic Quadrupole and Electron Multiplier Detector, with a simple click of the mouse

You also can download the 7700 Series animation (6.5 MB) to your computer – great as a teaching/training aid.

New 7700 Recorded Webinar

If you missed the recent 7700 Webinar, hosted by Spectroscopy Now, you can view the recording at a time to suit you.

Title: Redefining ICP-MS Analysis; Higher Matrix Levels, Simpler Operation; Better Removal of Interferences: The Extraordinary New 7700 Series ICP-MS

Go to: www.spectroscopynow.com and look for the “Webinars” link:

Webinars 

Upcoming e-Seminar

e-Seminar: EPA Methods Update for Metals Analysis, presented by Steve Wilbur, Agilent Technologies, Inc.
Date: Sept 24, 2009, 2:00pm EDT

To register go to www.agilent.com/chem/icpms-eseminars

- Follow instructions provided

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5990-4489EN

Conferences, Meetings, Seminars

A large number of 7700 launch seminars have already taken place, and many more are coming up. A few examples of events, including User Group Meetings being held in Sept/Oct/Nov, are given below. Be sure to check with your local Agilent office or www.agilent.com/chem/events web page for details of events in your area.

Europe

Pau, Massy, Namur
Basel, Waldbronn, Hamburg,
Berlin, Würzburg, Graz,
Stockholm, London, Ireland,
Netherlands, Italy.

Japan

11 venues, Sept. 29 to Nov. 6, 2009
www.chem-agilent.com/contents.php?id=1000794

FACSS 2009

Oct. 18-22, 2009
Louisville, KY, USA
www.facss.org

BCEIA 2009

Nov. 25-28, 2009
Beijing, China
www.bceia.cn

2010 Winter Conference on Plasma Spectrochemistry

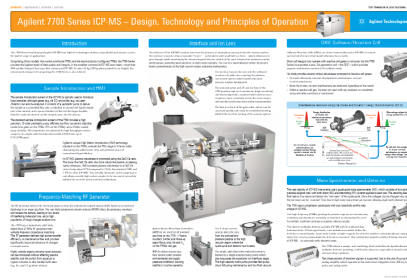
Jan. 4-9, 2010
Fort Myers, Florida, USA
www.icpinformation.org

Agilent ICP-MS Publications

To view and download these latest publications, go to www.agilent.com/chem/icpms and look under "Library Information"

- **Brochure:** The Agilent 7700 Series ICP-MS: Extraordinary design. Unparalleled performance, 5990-4025EN
- **Application Note:** The Agilent 7700 Advantage for Drinking Water Analysis, 5990-4315EN
- **Application Note:** Simple, Rapid Analysis of Trace Metals in Foods Using the Agilent 7700x ICP-MS, 5990-4539EN
- **Technical Overview:** Comparing Collision/Reaction Cell Modes for the Measurement of Interfered Analytes in Complex Matrices using the Agilent 7700 Series ICP-MS, 5990-3236EN
- **Advertorial:** Clearly Better ICP-MS: Introducing the Agilent 7700 Series, 5990-4302EN
- **Advertorial:** 7700x ORS³ and Helium Mode: More effective interference removal in complex samples, 5990-4408EN
- **Access Agilent e-Newsletter Sept '09:** Simpler removal of spectral interferences in ICP-MS with the Agilent 7700 Series ORS³

Free ICP-MS Wall Poster



Visit Agilent's ICP-MS web site to download a pdf or order a print copy of the new 7700 Wall Poster.

Poster: Agilent 7700 Series ICP-MS – Design, Technology and Principles of Operation. 5990-4234EN

Front page photo: Bill, Agilent's testing robot – as featured in the Agilent 7700 Series video on www.youtube.com

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