The New Agilent 7700 Series ICP-MS

the best ICP-MS just got better!

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Life Sciences and Chemical Analysis

Global Footprint

► Five Product Development Centers (R&D/MKTG)
► Three Manufacturing Centers, Four Logistics Centers
► Sales and Customer Service in 100+ Countries
Tokyo Analytical Division (TAD)

- 300 Employees
- Focus on Innovation and highest possible build quality
- Accredited ISO 9001, ISO 14001 manufacturing facility
- Worldwide R&D and Production of Agilent ICP-MS
- Products for worldwide shipment
  - Agilent ICP-MS mainframe
  - Agilent ICP-MS Software
  - Agilent ICP-MS peripherals
Agilent’s History of Innovation in ICP-MS – 1987 to 2009

1987 – PMS 100 introduced – First computer-controlled ICP-MS
1988 – PMS 200 introduced – Second generation ICP-MS with off-axis Qpole lens
1989 – 1st ETV accessory for semicon analysis by ICP-MS
1990 – PMS 2000 introduced – Omega off-axis lens. Lowest random background ICP-MS
1992 – ShieldTorch interface developed - Ar interferences virtually eliminated in cool plasma, enabling ppt analysis of K, Ca, Fe by ICP-MS
1994 – 4500 Series introduced - World's first benchtop system. Hyperbolic profile quad, motorized torch XYZ, cool plasma
1998 – First real time ICP-MS chromatographic software – PlasmaChrom. T-mode reaction interface introduced
1999 – 4500 Series 100, 200 & 300 introduced: 1st applications-specific ICP-MS.
2000 – Agilent 7500 Series introduced - 7500a, 7500i and 7500s - the next generation in ICP-MS instrumentation. 9 orders detector range
2001 – Agilent 7500c launched – 1st generation ORS for high matrix samples.
2003 – Agilent 7500cs launched – 2nd generation ORS for high purity semicon samples.
2004 – Agilent 7500ce launched – 2nd generation ORS for high matrix samples.
2005 – Low flow cell gas MFC’s for Xe NH₃, O₂, etc added to 7500ce/cs.
2006 – Agilent acquires 100% of Agilent/Yokogawa joint venture
2007 – Agilent 7500cx introduced: He only mode ICP-MS
2008 – High Matrix Interface developed – enables 2% TDS samples to be run by ICP-MS
2009 – Agilent 7700 Series introduced – replaces 7500 Series. MassHunter Software introduced - common platform with other Agilent MS. ISIS-DS Discrete sampling system, for ultra high throughput analysis
Agilent ICP-MS Instrument Development

Agilent 7700 Series
The new face of ICP-MS

Agilent 4500 Series

Agilent 7500 Series
Atomic Absorption Spectroscopy

Light having a wavelength characteristic of the analyte is passed through the sample that has been atomised in a cell.

The amount of light absorbed is proportional to concentration.
Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

Energy from the plasma promotes an electron to a higher energy level (excitation). Electron falls back and emits light at a characteristic wavelength.

Light emission is proportional to concentration.

Grating driven by motor allows users to scan through the wavelengths.

Monochromator system (Sequential type)
Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

Energy from the plasma ejects electron from shell (ionization). The result is a positively charged analyte ion.

Ions are separated by the mass spectrometer and measured. Ions measured are directly proportional to analyte concentration.
What is ICP-MS?

- **Inductively Coupled Plasma Mass Spectrometry**
- **Elemental analysis with:**
  - Wide elemental coverage
  - Very low detection limits
  - Fast analysis times (all elements at once)
  - Wide analytical working range (up to 9 orders)
    - Simple spectra
  - High matrix tolerance
  - Isotopic information
- **Viable alternative to ICP-OES or GFAAS**

*OES = Optical Emission Spectrometry, GFAAS = Graphite Furnace Atomic Absorption Spectrometry*
**ICP-OES** – for majors and high matrix samples
Multi-element, ~2 min/ sample
10’s ppb to 1000’s ppm

**ICP-MS** – for trace elements and clean samples
Multi-element, ~4 min/ sample
Single ppt to 10’s ppm

**GFAAS**
Single element, ~6 min/ sample
10’s ppt to 100’s ppb

**Hydride/AFS**
Few elements, ~2 min/ sample
Single ppt to 10’s ppb

**LOD**

**ppt**

**ppb**

**ppm**

Few Elements/Samples

Many Elements/Samples
Inorganic Analysis Techniques in a Typical Laboratory

- **ICP-OES**
  - Multi-element
  - ~2 min/sample
  - 10's ppb to 1000's ppm

- **ICP-MS**
  - Multi-element
  - ~4 min/sample
  - Single ppt to 10's ppm

- **GFAAS**
  - Single element
  - ~6 min/sample
  - 10's ppt to 100's ppb

- **Hydride/AFS**
  - Few elements
  - ~2 min/sample
  - Single ppt to 10's ppb

### LOD

- **7700 ICP-MS**
  - Single cell gas mode and productivity tools reduce run time
  - HMI for samples with % level solids
  - Single ppt (incl. hydride and Hg) to 100's ppm (1000's ppm with HMI)

Uniquely, the Agilent 7700 can replace all these separate techniques, providing high throughput, matrix tolerance, wide elemental coverage and low LODs in a single run.
### Agilent ICP-MS - 3 Sigma Detection Limits

<table>
<thead>
<tr>
<th>Element</th>
<th>Detection Limit</th>
</tr>
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<tbody>
<tr>
<td>H</td>
<td>&lt;0.1 ppt</td>
</tr>
<tr>
<td>Li 0.8</td>
<td>0.1 - 1 ppt</td>
</tr>
<tr>
<td>Be 0.2</td>
<td>1 - 10 ppt</td>
</tr>
<tr>
<td>Na 15</td>
<td>&gt;10 ppt</td>
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<tr>
<td>Mg 1</td>
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<tr>
<td>Al 5</td>
<td></td>
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<tr>
<td>Si 500</td>
<td></td>
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<tr>
<td>P 40</td>
<td></td>
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<tr>
<td>S &gt;1000</td>
<td></td>
</tr>
<tr>
<td>Cl &gt;1000</td>
<td></td>
</tr>
<tr>
<td>Ar 100</td>
<td></td>
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<tr>
<td>K 200</td>
<td></td>
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<tr>
<td>Ca 300</td>
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<td>Sc 3</td>
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<tr>
<td>Ti 4</td>
<td></td>
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<tr>
<td>V 0.7</td>
<td></td>
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<tr>
<td>Cr 2</td>
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<tr>
<td>Mn 0.2</td>
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<tr>
<td>Fe 100</td>
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<tr>
<td>Co 1</td>
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<tr>
<td>Ni 0.1</td>
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<tr>
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<td>Zn 1</td>
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<td>Ga 0.7</td>
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<td>Ge 0.9</td>
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<tr>
<td>As 2</td>
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<tr>
<td>Se 20</td>
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<tr>
<td>Br 100</td>
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</tr>
<tr>
<td>Kr 100</td>
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<tr>
<td>Rb 0.1</td>
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</tr>
<tr>
<td>Sr 0.06</td>
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<tr>
<td>Y 0.09</td>
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<tr>
<td>Zr 0.08</td>
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<tr>
<td>Nb 0.1</td>
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<tr>
<td>Mo 0.3</td>
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<tr>
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<tr>
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<tr>
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<tr>
<td>Pb 0.3</td>
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<tr>
<td>Bi 0.1</td>
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<tr>
<td>Po 0.3</td>
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<tr>
<td>At 0.1</td>
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<tr>
<td>Rn 0.8</td>
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<tr>
<td>Fr Ra</td>
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<tr>
<td>AC</td>
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<tr>
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<tr>
<td>Eu 0.1</td>
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<td>Dy 0.2</td>
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<tr>
<td>Tm 0.03</td>
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</tr>
<tr>
<td>Yb 0.2</td>
<td></td>
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<tr>
<td>Lu 0.04</td>
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**Standard pneumatic nebuliser**

3 sec/mass integration time
Inductively Coupled Plasma Mass Spectrometry

◆ ICP-MS is a fast, multi-element, high sensitivity trace metals analysis technique

◆ Key applications are:
  - Environmental
  - Foods
  - Semiconductor
  - Clinical
  - Chemical/Petrochemical
  - Pharmaceutical
  - Consumer Goods
  - Forensic
  - Geological
  - Nuclear
  - Academic/Research

◆ ICP-MS market continues to grow as technology improves and GFAA and ICP-OES instruments are replaced with ICP-MS
7700 Series – New Product Highlights

New ORS$^3$ Collision/Reaction Cell

◆ Longer, narrower rods, higher cell pressure and frequency – MUCH better performance in He mode

New RF Generator

◆ Fast, frequency-matching 27MHz generator, for better tolerance to changing matrix (incl. organics)

Increased Matrix Tolerance

◆ High Matrix Introduction (HMI) standard on 7700x model

Much smaller cabinet

◆ >30% smaller footprint than any other ICP-MS

Simple software; reliable Auto-Tuning

◆ MassHunter software – intuitive and easy to learn. Pre-set plasma conditions and fast lens auto-tuning
7700 - Smaller Cabinet; Longer Ion Path!!

The 7700 has >30% smaller footprint than any other ICP-MS, but access for maintenance has been improved (all service from front)

Despite much smaller external cabinet size, the ion path of the 7700 is actually 30mm longer than the 7500

Extra length due to redesigned cell (longer octopole rods) and lens
7700 Series Sample Introduction

- Low-flow (typically 0.15mL/min)
- Temperature stabilized (Peltier cooled spray chamber)
- Wide Torch Injector ID (2.5mm)
- No O-rings in spray chamber end-cap – reduced risk of contamination
- Auto-alignment of torch after maintenance
- Fast frequency-matching RF generator
- Simple setup, using “pre-set” plasma conditions and auto-tuning

Provides most robust plasma of any ICP-MS under standard conditions
Other Benefits of High Plasma Temp.

- Reducing CeO⁺/Ce⁺ ratio from 3.0% to 1.0% (3x reduction) removes ~70% of many matrix-based interferences (ArCl⁺, ClO⁺, CaO⁺, etc)
- Hotter plasma is less affected by a variable matrix (more robust)
- Better matrix decomposition reduces interface and lens contamination and therefore reduces maintenance
- Hotter plasma improves the ionization of poorly ionized elements, so MUCH lower LODs possible for Be (right), B, As, Se, Cd, Hg (below), etc

<table>
<thead>
<tr>
<th>BEC (ppt)</th>
<th>LOD (ppt)</th>
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<tbody>
<tr>
<td>Hg</td>
<td>9.49</td>
</tr>
<tr>
<td>Be</td>
<td>0.465</td>
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Sub ppt Be detection limit!
High Matrix Introduction (HMI) – How it Works

HMI is a sample dilution technique but, uniquely, it dilutes the sample in the gas state, using aerosol dilution. This removes the main problems of liquid sample dilution:

• Time
• Reagents
• Errors
• Contamination
Variable matrix sample – Ground water emulating sample (Vittel, Contrex, 1/5 seawater, ICS and mixed sample etc)

- 7500cx robust tuning
- Severe matrix suppression with some high matrix samples, and relatively high drift

- Improved long term stability with 7500cx and HMI
- Reduced matrix suppression with high-matrix enviro samples
Agilent 7700x ICP-MS System with Collision Reaction Cell (CRC)

- High temperature 27MHz plasma generator
- Low flow sample introduction system
- Multi-element interference removal by on-axis octopole reaction cell
- Fast simultaneous dual mode detector (9 orders dyn. range)
- High frequency hyperbolic quadrupole

Agilent Technologies
7700 Series Ion Lens

• Ion Lens focuses ions into the cell, and rejects photons and neutrals
• 7700 uses a combined Extraction/Off-axis Lens – located outside high-vacuum region, so easy to access for maintenance
• Provides ideal combination of high transmission across the mass range, low random background, and protection from matrix contamination in the high vacuum region
• No need to set up variable voltages to increase ion transmission at specific masses – all masses are transmitted all the time!

7700 Series has the highest ion transmission across the mass range of any ICP-MS, in standard, matrix tolerant conditions
All New Octopole Reaction System (ORS$^3$)

The 7700 uses a completely new collision/reaction cell, with:

- **18% longer rods**
- **15% smaller ID**

and operates at

- **16% higher pressure**
- **20% higher frequency**

ORS$^3$ also operates with a much larger energy discrimination step.

Result is **much more effective removal of interferences** in He mode with KED

*Major improvement compared to 7500 Series, which already offered by far the best He mode performance of any ICP-MS*
Helium interactions in an Octopole Reaction Cell

Collision: Energy discrimination

Molecular interference (ArCl) has larger cross section than the analyte (As).

More frequent interactions with He.

A significant reduction in kinetic energy relative to the analyte (As). Energy filtering can be used to ensure only the analyte enters the quadrupole analyzer.
Helium as a collision gas
Unspiked Mixed Acid Matrix \((5\% \text{HNO}_3, 5\% \text{HCl}, 1\% \text{IPA}, 1\% \text{H}_2\text{SO}_4)\)

ALL peaks in NoGas mode are due to polyatomic interferences

Many polyatomic interferences in NoGas Mode.
Note intensity scale of \(2E7\) cps
Let’s zoom the intensity scale 100x
Unspiked Mixed Acid Matrix (5% HNO₃, 5% HCl, 1% IPA, 1% H₂SO₄)

All peaks in NoGas mode are due to polyatomic interferences

Intensity scale 100x lower (2E5cps) – interfering polyatomic peaks at almost every mass in NoGas Mode

Now let’s look at the individual acid matrices – same scale
Acid Matrix in NoGas Mode
(HNO$_3$)

Unspiked 5% HNO$_3$ Matrix
Unspiked Matrix – ALL peaks are due to polyatomic interferences

NoGas Mode
Acid Matrices in NoGas Mode
(HNO₃ + HCl)

Unspiked 5% HNO₃ + 5% HCl Matrix
Unspiked Matrix – ALL peaks are due to polyatomic interferences
Acid Matrices in NoGas Mode 
\(\text{(HNO}_3 + \text{HCl} + \text{H}_2\text{SO}_4)\)

Unspiked 5% HNO\(_3\) + 5% HCl + 1% H\(_2\)SO\(_4\) Matrix

Unspiked Matrix – ALL peaks are due to polyatomic interferences

NoGas Mode
Acid Matrices and IPA in NoGas Mode
(HNO₃ + HCl + H₂SO₄ + IPA)

Unspiked 5% HNO₃ + 5% HCl + 1% H₂SO₄ + 1% IPA Matrix
Unspiked Matrix – ALL peaks are due to polyatomic interferences

Multiple polyatomic interferences affect almost every mass – Interferences are matrix-dependent
What happens to all these polyatomics in He Mode?

NoGas Mode
Single Acid Matrices and IPA in He Mode (HNO₃ + HCl + H₂SO₄ + IPA) – same scale as NoGas

Unspiked 5% HNO₃ + 5% HCl + 1% H₂SO₄ + 1% IPA Matrix

ALL polyatomic interferences are removed in He Mode (same cell conditions)

How does this compare to Reaction (H₂) Mode?

All polyatomic interferences are removed in He Mode
Unspiked Mixed Acid Matrix (5% HNO$_3$, 5% HCl, 1% IPA, 1% H$_2$SO$_4$)

ALL peaks in NoGas mode are due to polyatomic interferences
Many polyatomic interferences remain (or new ones are created) in H$_2$ Mode.

Interferences are different in each Matrix!

Let’s go back to He Mode
Mixed Acid Matrix in He Mode (100x zoom)
Same Scale as NoGas Mode

Unspiked 5% HNO$_3$ + 5% HCl + 1% H$_2$SO$_4$ + 1% IPA Matrix

ALL polyatomic interferences are removed in He Mode (same cell conditions)

How is the sensitivity for a 10ppb Spike in He Mode?
Mixed Acid Matrix in He Mode
Consistent sensitivity and perfect template match

10ppb Spike in 5% HNO₃ + 5% HCl + 1% H₂SO₄ + 1% IPA Matrix
Consistent high sensitivity for all isotopes of all elements in He Mode

High sensitivity for all spike elements at 10ppb Spike. Perfect template fit for all elements – no residual interferences and no loss of analyte sensitivity