Introducing the New Agilent 7700 Series ICP-MS; Improved Performance for Speciated Analysis

Agilent Technologies

Introduction

Agilent ICP-MS systems are widely used for speciation analysis in both research and routine laboratories, with many examples of applications combined with HPLC, GC and CE (see reference 1).

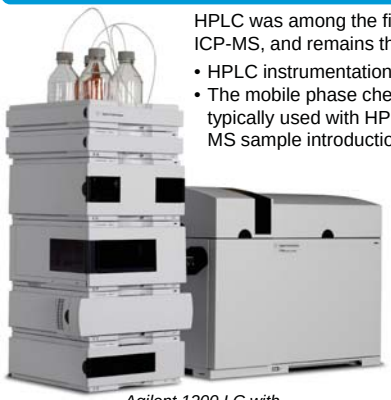
Speciated analysis with ICP-MS has proved useful in many applications including:

- Organo-tin speciation in marine sediments, marine fauna and consumer products
- Arsenic speciation in food and drinking water
- Chromium speciation in environmental samples, manufactured goods and building materials.

However, ICP-MS has also proved useful for the measurement of more unusual analytes, notably silicon, phosphorus, sulfur and the halogens. Applications include petrochemical, environmental, pharmaceutical, nutritional and clinical monitoring, and the improvement in the capability of ICP-MS to measure these difficult elements has been a key development priority at Agilent.

Here we present data for several speciation applications, and discuss improvements in speciation analysis due to developments in ICP-MS instrumentation.

LC-ICP-MS



HPLC was among the first separation techniques coupled to ICP-MS, and remains the most widely-used because:

- HPLC instrumentation and expertise is widely available
- The mobile phase chemistries and solution flow rates typically used with HPLC are generally compatible with ICP-MS sample introduction systems.

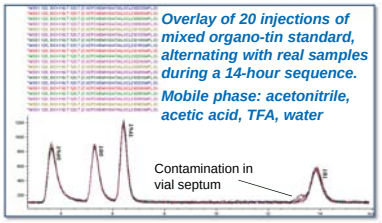
Reproducibility and long-term stability are key requirements for routine analysis by LC-ICP-MS.

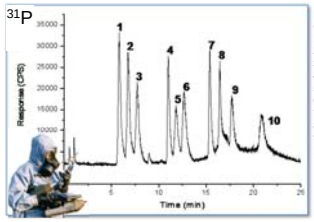
The figure below shows the overlaid chromatograms from 20 injections of a check standard, alternated with real samples during a 14 hour unattended sequence.

The retention time and peak height/ area precision was excellent.

Agilent 1200 LC with Agilent 7700 ICP-MS

Determination of previously difficult analytes such as P now allows direct quantification of toxic and hazardous compounds at trace levels, illustrated by the measurement of ^{31}P in chemical warfare agent (CWA) breakdown products using HLPC-ICP-MS (below) *(courtesy of University of Cincinnati)*





Elution Order
1. MPA
2. H_3PO_4
3. EPA
4. DMHP
5. PPA
6. EMPA
7. IMPA
8. DEHP
9. IPHEP
10. IBHMP

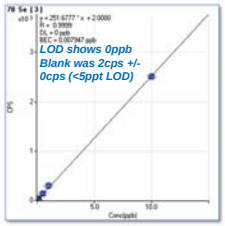
Degradation Products	Method	Detection Limits ng mL^{-1}(ppb)
MPA, IMPA, EMPA (VX Acid)	Ion Mobility Mass Spectrometry	560 - 1700 [1]
	LC-ESI-TOF	100 - 1000 [2]
	Electrophoresis Microchip with Contactless Conductivity Detector	48 - 86 [3]
	IP-RP-HPLC-ICP-MS	0.139 – 0.263 *

(1) Steiner, W. E.; Clowers , B. H.; Matz, L. M.; Siems , W. F.; Hill, H. H., Jr. Analytical Chemistry 2002 , 74, 4343 - 4352
(2) Liu, Q.; Hu, X.; Xie, J. Analytica Chimica Acta 2004 , 512 , 93 - 101
(3) Wang, J.; Pumera , M.; Collins, G. E.; Mulchandani , A. Analytical Chemistry 2002 , 74, 6121 -6125
* Doug Richardson et al, University of Cincinnati – Ion Pairing Reversed Phase HPLC-ICP-MS

7700 Series Design and Performance

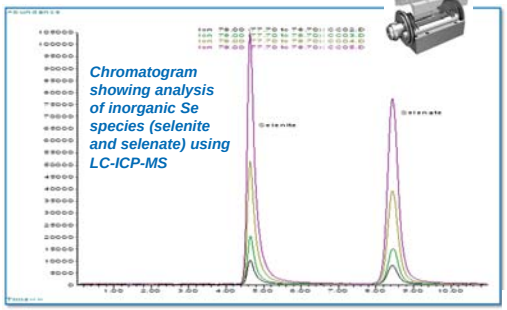
The completely redesigned, 3rd generation Octopole Reaction System (ORS^3) of the 7700 delivers improved removal of interferences in helium (He) collision mode.

- Using the ORS^3, many traditionally difficult elements (Si, P, S & Se – see chromatogram below) can now be measured at the same time (same cell mode) as other analytes, and at significantly lower detection limits than was previously possible,
- No need for complex, single-element reaction gas modes.



Low ppt Detection Limit for Se (mass 78) in He mode

He mode effectively removes other common polyatomic interferences such as ArCl on As at mass 75



Arsenic speciation in complex matrices is among the most common LC-ICP-MS applications.

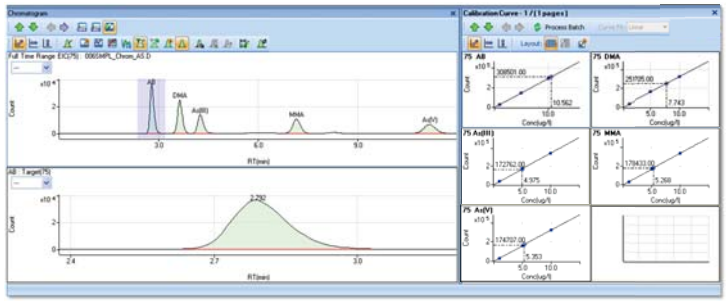
Routine As speciation analysis is carried out in urine, food, drinking water, groundwater, and seafood.

The 7700 Series ICP-MS uses a new frequency-matching RF generator, which responds instantly to a changing plasma load. This improves the tolerance of the 7700 to volatile organic solvents, allowing rapid gradient elution to be carried out with minimal disturbance of the plasma.

Fully Integrated Chromatography Software

The 7700 Series MassHunter workstation chromatography software (illustrated below) provides comprehensive functionality for speciation applications. It includes fully integrated sequencing and chromatographic data analysis, with advanced features such as Compound Independent Calibration, Outlier Flagging, Signal to Noise calculation and Snapshot for real-time data review during acquisition.

MassHunter is a common platform across all Agilent Mass Spectrometers (LC/MS, GC/MS and ICP-MS), simplifying user training and routine operation.

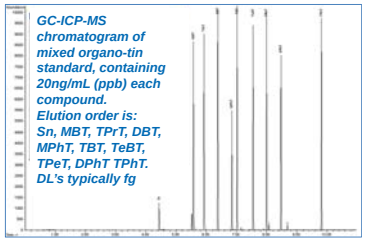


GC-ICP-MS

GC can be considered an ideal sample introduction method for ICP-MS.

- Absence of any sample solution means that the plasma operates at very high temperature and few polyatomic interferences occur.
- ICP provides very good matrix tolerance and selectivity, and most elements (including C, Si, P, S and the halogens) can be measured at relatively low levels.

ICP-MS is an elemental detector, so signal response is independent of compound structure. This means that accurate quantification can be achieved, even without compound–specific calibration standards (compound-independent calibration).



Determination of organo-tin (of which tri-butyl tin (TBT) and tri-phenyl tin (TPhT) are among the most toxic) and methyl mercury (MeHg) are well-established and routine applications for GC-ICP-MS.

- Organo-tins are easy to separate using conventional GC columns and methods
- Can be detected at low levels in environmental, food and consumer product samples, by ICP-MS.

GC-ICP-MS determination of Sn and Hg compounds commonly involves species-specific isotope dilution (SS-IDMS), which has the benefit of lower uncertainty than external calibration, and is independent of compound losses during extraction or derivatization.

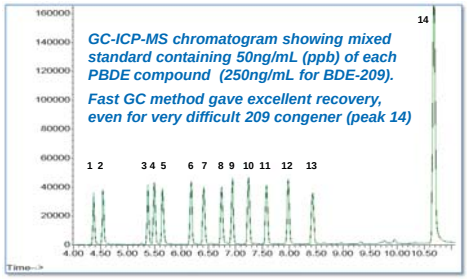
Agilent 7890 GC with Agilent 7700 ICP-MS

GC-ICP-MS is also used for more difficult applications, which require a fully-heated GC transfer line. These advanced applications include:

- Low-level pesticide residues (S, P, Cl, Br and I-based compounds)
- Sulfur compounds in fuels, and Siloxanes in biogas
- Brominated flame retardants in environmental samples and manufactured goods.

Poly-brominated diphenyl ether (PBDE) analysis by GC-ICP-MS is illustrated below

Peak Congener (50ng/mL)
1 - 1,2,2'-TriBDE (BDE-17)
2 - 2,4,4'-TriBDE (BDE-28)
3 - 2,3',4',6-TetraBDE (BDE-71)
4 - 2,2',4,4'-TetraBDE (BDE-47)
5 - 2,3',4,4'-TetraBDE (BDE-66)
6 - 2,2',4,4',6-PentaBDE (BDE-100)
7 - 2,2',4,4',5-PentaBDE (BDE-99)
8 - 2,2',3,4,4'-PentaBDE (BDE-85)
9 - 2,2',4,4',5,6'-HexaBDE (BDE-154)
10 - 2,2',4,4',5,5'-HexaBDE (BDE-153)
11 - 2,2',3,4,4',5'-HexaBDE (BDE-138)
12 - 2,2',3,4,4',5',6-HeptaBDE (BDE-131)
13 - 2,3,3',4,4',5,6-HeptaBDE (BDE-190)
14 - DecaBDE (BDE-209) (250 ng/mL)



Reference 1: Agilent Publication 5989-6160EN, Handbook of Hyphenated ICP-MS Applications, First Edition, August 25, 2015 © Agilent Technologies