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Determination of Iopromide in Environmental Waters by IC-ICP-MS

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Iopromide is an iodinated contrast medium (ICM) that is generally given to patients in g/L concentrations and is excreted within 24 hours in the patient's urine. It is very hydrophilic and non-ionic, properties that make it quite persistent in the environment. ICMs are known to be resistant to sewage treatment and studies have shown they are relatively poorly removed by conventional treatment processes.

Iopromide and other ICMs can form toxic iodinated disinfection by-products (I-DBPs) during oxidation and disinfection water treatment processes. Certain I-DBPs are known to be several times more toxic than chlorinated and brominated disinfection by-products but, as yet, are not regulated by the US Environmental Protection Agency

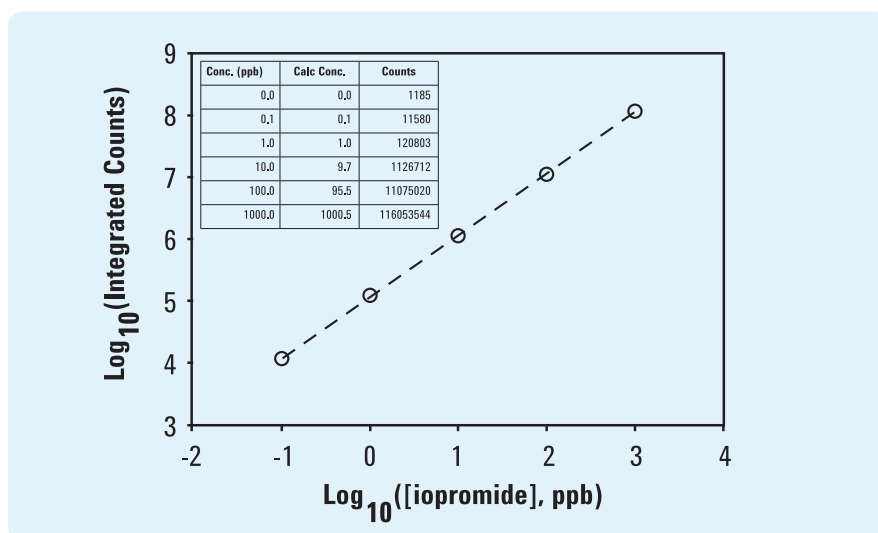


Figure 1. Calibration curve (log log) obtained for iopromide. Inset table lists calibration standard responses.

Experimental

Environmental water samples were collected at established monitoring points along the rivers and creeks in the state of California, including locations near water treatment plants. The water samples were prepared for analysis by IC-ICP-MS. Details can be found in Ref [1].

These sample extracts were injected into an Agilent 1260 HPLC coupled to an Agilent 7700x ICP-MS, with an injection volume of 500 μ L. The

chromatographic separation was performed using a Dionex AG16 4 x 50 mm guard column followed by a Dionex AS16 4 x 250 mm analytical column. Details of the gradient elution parameters and procedure can also be found in Ref [1].

The Agilent 7700x ICP-MS was operated with HMI sample introduction (0.6 L/min dilution gas, 0.5 L/min carrier gas, sample depth = 9 mm) and in helium collision mode (He flow 3.5 mL/min). Iodine (m/z 127)

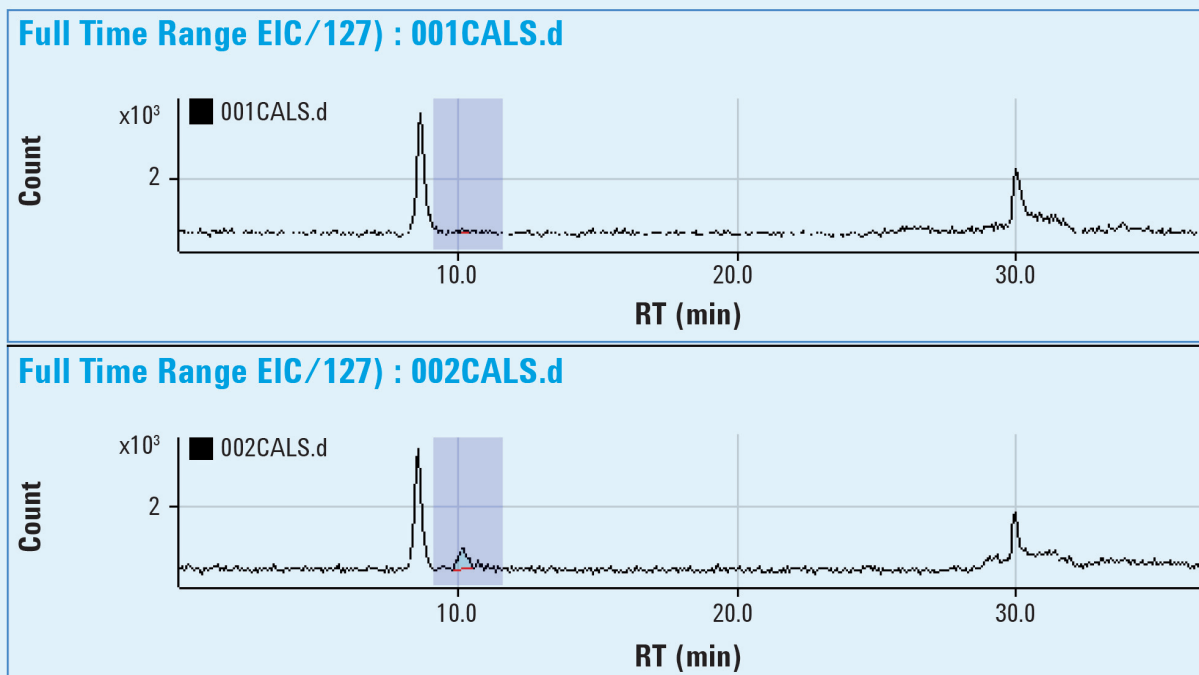


Figure 2. Comparison of the iodine chromatograms (m/z 127) obtained from injections of an aqueous blank extract (top) and an aqueous 0.1 ppb iopromide standard (bottom). The retention time of iopromide is 10.1 minutes.

intensity was monitored in time resolved analysis (TRA) mode using a 2 second integration time over a 37 minute time window. The use of HMI allows for extended analysis of high matrix samples with minimal matrix deposition in the interface cones, and the use of the He collision cell removes potential polyatomic interferences on masses 127 such as $^{126}\text{XeH}^+$. A calibration curve for iopromide was prepared using aqueous standards with concentrations of 0.0, 0.1, 1, 10, 100 and 1000 ppb of intact compound (Figure 1).

Results and Discussion

Using the method described, we have been able to detect iopromide in all non-zero standards used in our study. The chromatogram obtained for the 0.1 ppb injection is clearly distinguishable from the blank injection, as shown in Figure 2, and the calibration curve is linear over four orders of magnitude (Figure 1). This concentration range encompasses the levels at which iopromide has been observed in environmental waters including undiluted wastewater effluent.

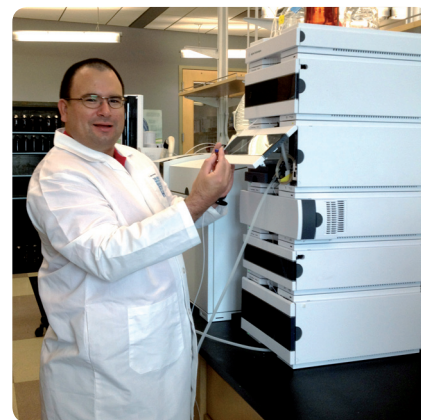
In addition to iopromide, several other iodine containing compounds were detected in our chromatograms. These unknown species can be quantified based on their iodine content, using compound-independent calibrations (CIC), where the iodine content of an unknown compound is calibrated using the iodine response for a known compound, in this case iopromide. ICP-MS is ideally suited for analysis using CIC, as the high temperature ICP ion source ensures that the elemental response of the target element (iodine in this case) is essentially independent of the compound in which the target element is present. The quantitation of the iodine content in these unidentified peaks is listed in Table 1.

Conclusions

The determination of iopromide in environmental waters has been accomplished using an Agilent 7700x ICP-MS coupled to an Agilent 1260 LC. A method reporting limit (MRL) of 0.1 ppb iopromide in the diluted extracts can be achieved, which, in theory, corresponds to a MRL of 2 ppt in the water samples.

References

1. Agilent Application Note: Determination of iopromide in environmental waters by ion chromatography-ICP-MS, 5991-1044EN



Dr. Durazo pictured with an Agilent 1260 coupled to a 7700x ICP-MS

Further Information

Learn more about the Snyder Research Group at the University of Arizona at:

www.snyderlab.arizona.edu

Sample Data file	Acq. date and time	Type	Sample name	Unknown 1 RT 5.2 min Conc. (ppb)	Unknown 1 RT 5.2 min Conc. (ppb)	Iopromide (as I) Conc. (ppb)	Unknown 1 RT 5.2 min Conc. (ppb)	Unknown 1 RT 5.2 min Conc. (ppb)
007SMPL.d	4/20/2012 2:03 PM	Sample	SJC1	268.55	0.24	118.17	5.77	9.64
008SMPL.d	4/20/2012 2:48 PM	Sample	LAR Ref	1.43	0.23	0.07	0	55.55
009SMPL.d	4/20/2012 3:33 PM	Sample	LAR Eq blank	1.86	0.2	0.09	0	0.38
010SMPL.d	4/20/2012 4:18 PM	Sample	LAR 6	84.23	0.29	1.21	6.16	17.13
011SMPL.d	4/20/2012 5:04 PM	Sample	LAR 5	98.87	0.39	0.95	7.11	11.95
012SMPL.d	4/20/2012 5:49 PM	Sample	LAR 4	128.68	0.38	1.17	12.97	13.03
013SMPL.d	4/20/2012 6:34 PM	Sample	LAR 3	116.4	0.27	0.89	10.21	12.62
014SMPL.d	4/20/2012 7:19 PM	Sample	LAR 2	156.01	0.22	0.28	12.86	14.87
015SMPL.d	4/20/2012 8:04 PM	Sample	LAR 1	189	0.29	1.05	12.15	22.51
016SMPL.d	4/20/2012 8:50 PM	Sample	Eq blank	0.28	0.19	0	0	0.21
017SMPL.d	4/20/2012 9:35 PM	Sample	100 ppb STD	0	0.15	51.47	N/D	0.11
018SMPL.d	4/20/2012 10:20 PM	Sample	SGR ref	0.34	0.21	0.07	0	0.29
019SMPL.d	4/20/2012 11:05 PM	Sample	SGR 6	142.49	0.16	15.69	8.46	20.54
020SMPL.d	4/20/2012 11:51 PM	Sample	SGR 5b	194.45	0.12	16	11.2	14.92
021SMPL.d	4/21/2012 12:36 AM	Sample	SGR 5a	7.27	0.19	0.81	0.37	54.73
022SMPL.d	4/21/2012 1:21 AM	Sample	SGR 5	326.73	0.1	40.91	11.29	5.66
023SMPL.d	4/21/2012 2:06 AM	Sample	SGR 3b	24.46	0.28	0.54	3.95	6.56
024SMPL.d	4/21/2012 2:52 AM	Sample	SGR 3a	322.5	N/D	52.01	11.36	3.14
025SMPL.d	4/21/2012 3:37 AM	Sample	Blank	0.03	0.23	0.03	0	0.18

Table 1. Summary of the concentrations of iodinated compounds (expressed as iodine concentration) measured in all extracts.

First ICP-QQQ in US Delivers More Accurate Results for Complex Sample Analysis

Introduction

Birmingham-based Southern Research Institute took delivery of the first Agilent 8800 Triple Quadrupole ICP-MS (ICP-QQQ) in the United States in June 2012. Southern Research is a not-for-profit scientific research organization that invests in the latest technology in order to offer the best R&D service to its clients. Previously Southern Research took delivery of the first GC-QTOF from Agilent. Their experience with an existing 7700x single quadrupole ICP-MS allowed the team to set up the 8800 with final checks and installation tests carried out by Agilent's Field Service engineer, John Deszell. The instrument passed its check out specifications without a hiccup.

The Company

Southern Research conducts advanced engineering research in materials, systems development, environment and energy, and preclinical drug discovery and development. The ICP-QQQ is located in the Environmental Science Laboratory which supports the development of technical solutions to improve air quality, provide clean water, and produce cleaner energy. According to Southern Research's Senior Research Scientist, Mark Kelinske, the new instrument will be used for diverse applications, working 50% of the time as a research tool and 50% as a companion workhorse instrument to the existing ICP-MS to manage the lab's large sample workload. In Kelinske's opinion the ICP-QQQ offers all of the same capabilities as the 7700 single quadrupole ICP-MS but expects to see significant improvements in data quality and consistency that can only be achieved using the MS/MS modes enabled by the tandem MS configuration of the 8800.

More Accurate Results for Complex Samples

The ICP-QQQ will deliver lower detection limits for challenging elements such as As, Hg, Se, and Cr in flue gas desulfurization (FGD)



Southern Research Institute's Mark Kelinske with the 8800 ICP-QQQ

wastewaters. The advantage of using an ICP-QQQ for complex matrices such as FGD with up to 6% w/v total dissolved solids (TDS) content is that the analyst can select which analytes enter the ORS³ cell, using the first quadrupole (Q1) as a mass filter, and rejecting all other masses. Because plasma and matrix ions are eliminated by Q1, this ensures that the reaction processes in the ORS³ are precisely controlled allowing accurate measurements to be performed even in complex, high-matrix samples.

Additionally the ICP-QQQ will be coupled to an HPLC to investigate the speciation of As, Hg, Se, Cr and S in FGD samples.

Before the end of the year, the U.S. Environmental Protection Agency (USEPA) is expected to update the Steam Electric Power Generating effluent guidelines that apply to wastewater discharges from steam electric power plants. The team at Southern Research is already working with industry leaders to anticipate the new limits by developing robust methods for the accurate and precise measurements of inorganic pollutants.

Pioneering Application Development

Another research area of interest to Southern Research and its clients in the electric power industry is the development and evaluation of post-combustion carbon capture and sequestration (storage) technologies that reduce harmful emissions and track greenhouse gas reduction.

One method that is used to capture the CO₂ from flue gases after hydrocarbon combustion is to absorb it in a solvent. There is a requirement to analyze these solvents for pollutants such as Cr, As, Se, S and P that accumulate over time and can ultimately affect the effectiveness of the solvent to capture CO₂.

The challenges of analyzing organic solvents by ICP-MS have largely been overcome by technological advances including a Peltier-cooled spray chamber and fast variable-frequency impedance matching solid state RF generator. However the analysis of phosphorus and sulfur in organic solvents remains challenging due to intense interferences from polyatomic ions based on carbon, nitrogen and oxygen, which are difficult to completely remove using conventional single quadrupole ICP-MS.

ICP-QQQ provides significantly enhanced sensitivity and interference reduction for these elements by utilizing MS/MS mode, where the first quadrupole eliminates all but the target Q1 mass and any interfering ions at the same m/z. By rejecting all non-target masses, the reaction chemistry in the cell is controlled and consistent, resulting in highly efficient and predictable interference removal. Q1 also rejects any off-mass ions that might overlap the analyte product ions created in the cell, thereby greatly improving the accuracy and reliability of reaction gas modes that use mass-shift.

For the first time, Southern Research will be in a position to offer its clients a reliable and sensitive method for the measurement of sulfur and phosphorus in the 30% MEA (monoethanolamine) solvents used in CO₂ sequestration using ICP-QQQ.

Team Effort

Mark Kelinske will lead a team of 6-7 research staff, all of whom currently operate the 7700x, to operate the ICP-QQQ. As the 8800 shares many hardware components and its software platform with the single-quad ICP-MS, the team is already familiar with the operation of the new instrument.

Further Information

Visit Southern Research Institute's web site at:
www.southernresearch.org

Pioneering Research Institute Purchases First European 8800

The first 8800 to be installed in a European lab has gone to German-based HZG Helmholtz Zentrum Geesthacht Institute for Coastal Research, formerly known as GKSS. The same lab also took delivery of the very first 7500 ICP-MS in Jan 2000 and was one of the early labs to own a 7700 ICP-MS. The two photos show the Marine Bioanalytical Chemistry's department head, Prof. Andreas Prange (center) with Agilent's ICP-MS Service Engineer, Hans Kavka (right), and Agilent's European ICP-MS Support Specialist, Glenn Carey (left) with the respective instruments, more than 12 years apart! The installation of the first 8800 in Geesthacht underlines the continuation of Agilent's successful long-term and proud collaboration with HZG.



Agilent 7500 ICP-MS in HZG lab taken in January 2000

To understand how the 8800 will benefit the Marine Bioanalytical Chemistry lab, Environmental Scientist, Dr. Daniel Proefrock, answered a few questions.

- **What is the lab's main inorganic work? What other instrumentation do you have?**

Our department is concerned with the determination of the spatial and temporal distribution of selected bio-accumulating and toxic (PBT) substances in the North Sea. Indications of possible effects in marine organisms are also of special interest. Currently, the focus is on poly-brominated diphenyl ethers (PBDEs) and heavy metals such as cadmium and nickel, as well as elements and element species (e.g. arsenic, tributyltin) and protein biomarkers (detoxification enzymes e.g. CYP450 and stress markers e.g. HSP).



Agilent 8800 ICP-QQQ installed in HZG lab in Geesthacht, Germany

A major objective is to develop new integrative methods to enable a spatially and temporally resolved determination of hazardous compounds together with an effect-oriented quantitative measurement of biochemical parameters. This should provide a new basis for improved assessment of contamination levels in the marine environment in terms of the impacts of anthropogenic inputs and activities (water quality).

In order to obtain time-integrated, and therefore more representative results, passive samplers (e.g. polymer sheets) as well as active samplers (caged, pre-conditioned, blue mussels) will be used for the enrichment of PBT substances. The deployment of active samplers will allow the measured chemical parameters and biological effects to be linked and thus substance-related effects to be revealed by the use of biomarkers.

Besides CRC-ICP-MS, our lab is also equipped with a high res ICP-MS, various chromatographs which we couple to ICP-MS for speciation studies, plus ESI and MALDI based MS techniques.

- **Thinking about your ICP-MS workload, what are your main analytical challenges?**

The main analytical challenges are caused by the requirement to run complex environmental sample matrices often with quite low analyte concentrations, which makes the determination of certain elements challenging.

- **Are there particular limitations with your current ICP-MS methods that led you to evaluate the 8800?**

In addition to sensitivity, problems with the accurate determination of selected elements in some matrices and the restriction to a limited set of cell gases were some of the main

reasons we evaluated ICP-QQQ.

- **How did you evaluate the instrument?**

We did some lab testing with different samples related to our current research work. The results were promising, which finally led to the decision to buy the new instrument.

- **What are your initial plans for the 8800?**

Currently, we are working on the transfer of some methods used for environmental applications e.g. analysis of biota samples originally run on a 7500cx. We expect to benefit from the new possibilities related with the extended selection of cell gases available for the 8800, as well as the MS/MS capabilities of the ICP-QQQ system.

- **What are the main benefits of the 8800 for your application.**

In general, we expect to be able to handle certain interferences much more effectively, which currently hamper the accurate determination of some important elements such as Cr, V, As or Se. Also the possibility that arises due to the application of O₂ as cell gas with respect to the more sensitive measurement of phosphorus or sulfur will help to improve some of our applications within the field of environmental proteomics.

- **How many people do you expect to train up and how easy do you expect the instrument will be to use?**

Initially 4 to 5 people will be trained to operate the new system. Since we have had plenty of experience over the years with earlier Agilent ICP-MS, such as 7500s, 7500c, 7500cx-cs and 7700cx, we anticipate that the 8800 will be easy to operate.

Quantification of Arsenic Species in Apple Juice using LC-ICP-MS

Tetsushi Sakai and Ed McCurdy
Agilent Technologies, Japan and UK

Concern has been expressed over the levels of inorganic As in apple juice, with some reports stating that the levels can exceed the United States Environmental Protection Agency (USEPA) limit for As in drinking water (10 ug/L or ppb). Arsenic in apple juice may be derived from the historical use of the As-based pesticides, lead hydrogen arsenate and calcium arsenate. Widespread use of these chemicals ceased in the 1970s, but they are persistent and may still affect crops grown on contaminated land.

Experimental

In this study, we used an Agilent 1200 Infinity LC coupled to an Agilent 7700x quadrupole ICP-MS to separate and measure the As species in six commercial apple juices purchased from a Japanese supermarket. The apple juice samples were prepared by simple filtration and a 2x dilution in deionized water, ensuring that species inter-conversion was minimized and detection limits were maintained. The species of interest, As(III), As(V) and the organic forms arsenobetaine (AB), dimethylarsinic acid (DMA) and monomethylarsonic acid (MMA) were separated using an anion

exchange guard column (Agilent part number G3154-65002, 4.6 mm id x 10 mm polymethacrylate) followed by As speciation column (Agilent part number G3288-80000, 4.6 mm id x 250 mm polymethacrylate).

Results and Discussion

Calibrations for the toxic inorganic As species are shown in Figure 1. The calibration range was from 10 to 500 ng/L (ppt) illustrating the good sensitivity and linearity at these sub-ppb levels.

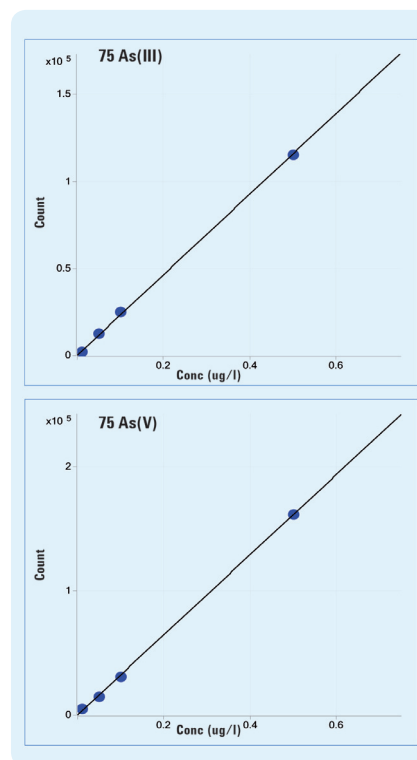


Figure 1. Calibrations for As(III) and As(V) from 10 to 500 ng/L (ppt).

The chromatograms of As species in four apple juice samples are shown overlaid in Figure 2. It should be noted that the first peak that elutes is arsenobetaine (AB), which is not retained on the column and so elutes in the void volume, where it might also co-elute with other neutral or cationic species that are not retained on the column. While AB can be measured using the LC-ICP-MS method described, the results may be biased if other co-eluting species are present in the sample; but this limitation is not a problem for food safety applications, since AB is considered to be non-toxic even at very high concentrations.

For the purposes of food safety, the critical species that must be separated and quantified accurately and at low level are As(III) and As(V), the sum of which can be reported as 'total inorganic arsenic'.

Concentrations for each species in all six apple juice samples are shown in Table 1. The results are corrected for the 2x dilution factor applied during sample preparation. From these results, it is clear that all the samples contained some inorganic As, but the levels were low (less than half the USEPA limit for drinking water).

Conclusions

The 7700x is suitable for the analysis of As species at low 10's ppt levels in apple juice. The simple sample preparation, low dilution and rapid, reliable chromatographic separation ensure that this method is suitable for routine monitoring of As species in beverages.

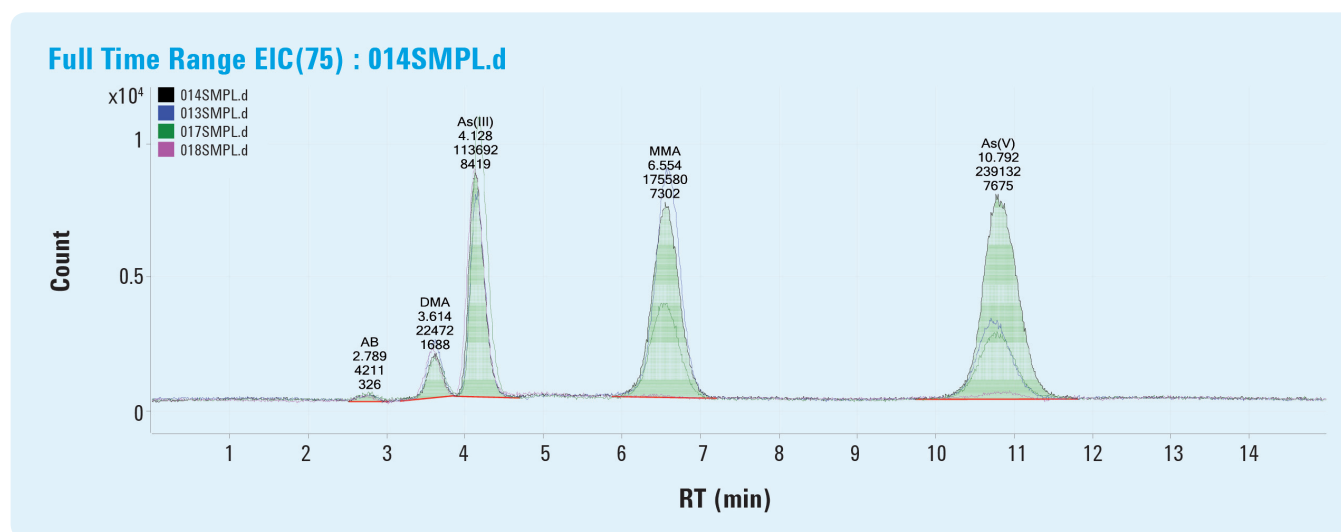


Figure 2. Overlaid chromatograms of As species in four apple juice samples

Sample		AB	DMA	As(III)	MMA	As(V)	Total Inorganic As (As(III) + As(V))
Sample Name	Dilution	Conc. [ug/L]	Conc. [ug/L]	Conc. [ug/L]	Conc. [ug/L]	Conc. [ug/L]	Conc. [ug/L]
Apple Juice 1	2	0,036	0,189	0,724	N/D	0,651	1,375
Apple Juice 2	2	0,026	0,022	0,041	N/D	0,058	0,099
Apple Juice 3	2	0,02	0,267	0,883	1,587	0,758	1,641
Apple Juice 4	2	0,039	0,208	0,992	1,466	1,958	2,95
Apple Juice 5	2	0,043	0,209	1,256	0,785	0,709	1,965
Apple Juice 6	2	0,036	0,235	1,098	N/D	0,068	1,166

Table 1. Concentrations of five As species in six apple juice samples. N/D = not detected

Further Information

The same apple juice samples were also measured using this LC method coupled to the Agilent 8800 ICP-QQQ,

which provides higher sensitivity and lower detection limits than the 7700 Series ICP-MS. The 8800 data is presented in Application Note

5991-0622EN.

www.agilent.com/chem/accelerate-food

Tips & Tricks: Recommended Reference Web Sites

The web is a massive source of valuable information but, on occasions, it can be difficult to locate exactly what you need, when you need it. To make your life easier, we have compiled a list of freely available resources including a must-join listserv and web sites that Agilent staff use regularly in their work.

Listserv Mailing Group

- **PLASMACHEM-L Listserv Group:** The PLASMACHEM Listserv is a moderated listserv, hosted by Syracuse University, that facilitates the sharing of information between plasma spectroscopists all across the world. For more information and to join the list, search on PLASMACHEM-L or go to: <https://listserv.syr.edu/scripts/wa.exe?A0=PLASMACHEM-L>

Reference Web Sites

- **EVISA:** a primary source of information about trends, new technologies, new products, upcoming legislation and other news related to element species and speciation analysis. www.speciation.net

- **Unit Conversion Online**
www.onlineconversion.com
- **IUPAC Compendium of Chemical Terminology**
<http://goldbook.iupac.org/index.html>
- **Quantifying uncertainty in analytical measurement**
www.measurementuncertainty.org
- **US government list of environmental monitoring methods**
www.nemi.gov
- **Pharma compliance web site**
www.labcompliance.com
- **Interactive online periodic table**
www.webelements.com
- **An Index of the Literature for Bimolecular Gas Phase Cation-Molecule Reaction Kinetics**
<http://trs-new.jpl.nasa.gov/dspace/handle/2014/7981>
- **Chemical resistance of pump tubing**
www.omega.com/pdf/tubing/technical_section/chemical_chart_1.asp

Journals and Science Web Sites

- **Journal of Analytical Atomic Spectrometry**
<http://pubs.rsc.org/en/journals/journalissues/ja>
- **Spectroscopy**
www.spectroscopyonline.com
- **Spectroscopy Europe/Asia**
www.spectroscopyeurope.com
- **SpectroscopyNOW**
www.spectroscopyNOW.com
- **Select Science**
www.selectscience.com
- **Laboratory News**
www.labnews.co.uk

Agilent Resources

- **ICP-MS Primer, 5989-3526EN**
www.chem.agilent.com/Library/primers/Public/ICP-MS_Primer-Web.pdf
- **2nd Edition eHandbook of Hyphenated ICP-MS Applications, 5990-9473EN**
www.agilent.com/chem/speciation2
- **Elemental Impurity Analysis in Regulated Pharmaceutical Laboratories, 5991-0436EN**
www.agilent.com/chem/usp-232-233

Agilent's 8800 ICP-QQQ named "Product of the Show" at ASMS



Agilent introduced its 8800 Triple Quadrupole ICP-MS less than six months ago and it's already making headlines. In May, Instrument Business Outlook (IBO) named this industry-breakthrough triple quad as the outstanding new product at this year's sixtieth annual conference of the American Society of Mass Spectrometry (ASMS). IBO is the well-established newsletter aimed at providing decision-makers with the latest information available about analytical and life science instruments.

"This is extremely significant as this has been a year of some major product introductions and, more important, ASMS has historically been focused on LC/MS and GC/MS. To have an inorganic technique like ICP-MS become 'Product of the Show' is a huge honor," said ICP-MS Marketing Manager Ken Suzuki.

The first and only instrument of its kind, the 8800 ICP-QQQ, with its high performance MS/MS mode, redefines application and measurement possibilities beyond conventional ICP-MS. As a result of these unique capabilities, the new instrument is enjoying strong initial sales success with more than 30 units already installed or destined for labs all across the world. Interestingly, the 8800 is going to a range of companies and institutions for a variety of applications such as environmental, materials, semiconductor, pharmaceutical, forensics, chemical and energy as well as general research.

This information is subject to change without notice.

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Agilent Celebrates Unprecedented ICP-MS Sales Success in Japan

Agilent Technologies is proud to acknowledge the sale of more than 1,500 ICP-MS units to Japanese customers. This equates to >70% share of the Japanese ICP-MS market according to Agilent sales data.

Agilent's ICP-MS business can trace its origins back to the late 1980's, with the first units been developed and sold by Tokyo-based Yokogawa Electric Corporation.

Over the years, Japanese customers requiring high sensitivity and accurate measurements at ultra-trace levels have helped to drive the development of the technique. Each and every model of ICP-MS released by Agilent, from the 4500 up to the 8800 Triple Quad, has found widespread approval from Japanese customers, many of whom own multiple Agilent ICP-MS.

Your Invite to an Agilent Event at the Winter Plasma Conference 2013

Meet our team of atomic spectroscopy experts at the Agilent booth #8.9 and at the following informative events:

- **Presentation of the European Award for Plasma Spectrochemistry, donated by Agilent Technologies**, Sunday evening 10 February 2013.
- **Agilent Lunch Seminar** – hear about our latest developments: Tues 12 February, with refreshments.
- **Agilent Technologies Evening Event** – join our popular user group meeting, Wed 13 February, with dinner and drinks!

More details will be posted to www.agilent.com/chem/wpc

Conferences. Meetings. Seminars.

- **Eastern Analytical Symposium**
November 12-14, 2012, Somerset, New Jersey, USA, www.eas.org
- **European Winter Conference on Plasma Spectrochemistry**
February 10-15, 2013, Krakow, Poland, www.chemia.uj.edu.pl/ewcps

Agilent ICP-MS Publications

To view and download the latest ICP-MS literature, please use the search facility or follow the links from www.agilent.com/chem/ms

- **Application note:** Application of a new capillary HPLC-ICP MS interface to the identification of selenium-containing proteins in selenized yeast, 5991-0882EN
- **Application note:** Arsenic speciation analysis in apple juice using HPLC-ICP-MS, 5991-0622EN
- **Application note:** Determination of iopromide in environmental waters by IC-ICP-MS, 5991-1044EN
- **Application note:** Direct measurement of trace REEs in high purity REE oxide using the 8800 Triple Quadrupole ICP-MS with MS/MS mode, 5991-0892EN

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