

# **Agilent** ICP-MS Journal

February 2016 – Issue 64

# Inside this Issue

- 2 Agilent Pays Tribute to Prof. Joseph "Doc" Caruso
- 2-5 Distinguished Former Colleagues and Students Remember Prof. Joe Caruso
- 6-7 Fast Arsenic Speciation for Food and Urine Analysis
- 8 Scientists for a Day: Inspiring The Next Generation; Four New Elements Verified by IUPAC; Fun In The Desert! Reflections from the 2016 Winter Conference on Plasma Spectrochemistry; Conferences, Meetings, Seminars; Latest Agilent ICP-MS Publications





# Agilent Pays Tribute to Joseph "Doc" Caruso

Steve Wilbur, Amir Liba and Jenny Nelson, Agilent Technologies

#### Leading authority in the field of trace metals analysis who pioneered research into metallomics

Over the forty-plus years that he worked for The University of Cincinnati's Chemistry Department, Professor Joe Caruso inspired many of his peers, colleagues and students with his knowledge, intellect, absolute love of science in all its forms and the contagious, genuine enthusiasm he conveyed to all who had the pleasure of meeting him.

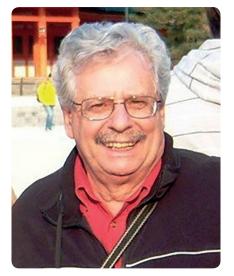
With research interests ranging from environmental monitoring to neurophysiology and phyto-engineering, Prof. Caruso firmly established himself and his research group as leading authorities in trace metals research, primarily using ICP-MS.

Since 2007, Agilent has worked closely with The University of Cincinnati (UC) to promote and support research in all fields related to metallomics - the analysis of metals, metal species and their interactions within biological and ecological systems. Agilent's Toshiaki Matsuda liaised with Prof. Caruso to establish the Metallomics Center of the Americas. The collaboration was highly successful in supporting development of applications including neurological research, metalloproteomics, metal tags for ultra-trace-level organic compound determination, and environmental monitoring throughout the Americas and beyond.



Left to right: Toshiaki Matsuda, Judy and Joe Caruso, Toshifumi Matsuzaki, General Manager of Agilent Tokyo Analytical Division, and Kimiko Matsuda.

Prof. Caruso's legacy will not only be the hundreds of publications (more than 330, cited more than 8000 times), coauthor of 5 books, invited lectures (more than 300), academic awards (including the Rieveschl Award for Distinguished Scientific Research), committee memberships and editorial boards he was involved with, but his students. These are the people who will continue to push the frontiers of the science they learned to love under his guidance.



Professor Joseph A. Caruso who passed away on November 23, 2015

Nearly 80 PhD candidates moved through Joe's group since it began in 1971 and many now hold influential positions in government, industry and academia all around the world. They are all still part of the "family" as many continued to stay in touch with Joe and his family, as well as with each other, on an academic and personal level.

In this special issue of the Agilent ICP-MS Journal, we'd like to take the opportunity to honor Prof. Caruso by giving some of his many friends the opportunity to express what the Doc meant to them.

#### Gary M. Hieftje,

# Distinguished Professor and Mann Chair in Chemistry, Indiana University

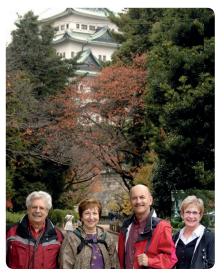
I had the privilege of knowing Joe Caruso for virtually my entire professional life. Joe began his research at the University of Cincinnati about the same time I started at Indiana, and we worked in overlapping fields. We attended most of the same conferences, published in the same journals, served on the same editorial boards, and drank the same wine. As our careers developed, we were often accompanied at conferences by our wives, Judy and Susan. All of us became close friends.

#### Joe the Man

Who among us can remember Joe without a smile on his face or a kind word for those around him? At conferences or in discussion, Joe was always the one with a crowd around him, not only his students and former students but also the cadre of friends and colleagues. He would dance at the drop of a hat, and could play a mean accordion or concertina. "Don Giuseppe" was justifiably proud of his Sicilian heritage, which came nicely to the fore during the European Winter Conference on Plasma Spectrochemistry, held in Taormina, Sicily and co-chaired by Joe and Maria Betti. Immediately clear also was Joe's lack of selfishness and his infectious sense of humor. He was always willing to support a colleague, sometimes to his own detriment, and could always find the humor in any situation, uncomfortable or not.

#### Joe the Scientist

Quite simply, Joe Caruso established and maintained a reputation as one of the most impressive and prominent chemists working in the field of analytical atomic spectrometry. He achieved a top international reputation for his work in the development, characterization and application of microwave-induced plasmas for atomic spectrometry, for the investigation of techniques in plasma source mass spectrometry, for the development of methods for elemental speciation, and for the design and development of other kinds of atomic spectrometric instrumentation. He presaged some of the recent findings on the perils of smoking hookah tobacco and e-cigarettes. His published work is widely read, highly regarded, and his students are eagerly sought by industrial recruiters and academic scientists. He served as the first non-British Chairperson of the editorial board of the Royal Society of Chemistry (which Joe usually termed "The Royals") Journal of Analytical Atomic Spectrometry (JAAS) and was the moving force behind the establishment of the RSC journal Metallomics, serving also as its first editorial-board Chair.



Joe and Judy Caruso with Gary and Susan Hieftje in Japan

#### Joe the Administrator

These scientific accomplishments are all the more remarkable considering the added administrative responsibilities that Joe assumed during his career. Unlike most scientists, whose work declines in importance or productivity when they take on administrative burdens. Joe's research became ever more important, more focused, and more prominent. Amazingly, his scientific reputation climbed even more rapidly while he was department head at Cincinnati than earlier. Even his assumption of a Deanship did not lessen his productivity or the scholarly respect others held for him.

#### Joe the Mentor

Joe has had a "Who's Who" of scientists working in his laboratory. His outstanding training of students, postdoctoral associates, and visitors has had a tremendous impact on the entire field of analytical chemistry. The success of these colleagues and the affection they hold for Joe is apparent in their work and in how they interacted with him. Joe and Judy always seemed to have room in their home and hearts for these colleagues, and promoted a group "spirit" that is exceedingly rare and enviable.

#### Jon Carnahan,

#### Professor and Chair, Department of Chemistry and Biochemistry, Northern Illinois University

It is with sadness that I compose this tribute to Professor Joseph A. (Doc) Caruso. But, it is with a great amount of fondness and admiration that I look upon Joe, his life, his family, his contributions to science, and the positive influence on those his life touched.

Of course, when a person reflects on another's life, they do so from their own perspective. However, Joe treated everyone as a friend, regardless of their relationship with him. I'm betting that my perspective is one that is shared by most of those who knew Joe.

The first time I met "Dr. Caruso," as I knew him at the time, I was visiting Cincinnati just before moving to attend graduate school at the University of Cincinnati. I was doing a "tour" of the analytical division, meeting the faculty and graduate students, with all the insecurities that come with stepping into a completely new situation. All of the faculty, of course, were very amicable and encouraging, as were the UC students. I got a special feeling at UC. As I look back, I see that my impression was shaped by Joe's influence on the department.

When I joined Joe's group, we discussed projects and the direction his laboratory was going. Within that context, I sketched out some ideas that I thought were well organized. I'm sure that Joe, sensing that my enthusiasm far exceeded my current skill set, knew that he could bring out the best in me; at least he was willing to try. He did this with insightful research suggestions, tons of support, and a boatload of patience with me and all of the members of the Caruso laboratory.

"Doc" took his research group, and all of their friends, under his wing. He and Judy ("Judith the Good," as he referred to his wonderful wife and our group "Mom") would regularly host get-togethers. We would talk, have fun, and often end the night with at least a couple of hours of sing-alongs with Joe and his accordion.

While I could go on about "Doc's" leadership as the Chemistry Department Head, the Dean of UC's College of Arts and Sciences, his 300+ publications, his numerous awards from the UC and externally, his stellar record of extramural funding etc., there is one thing that I find the most outstanding about Dr. Caruso: he was one of the most nurturing souls that most of us ever had the pleasure to know.

As I continued along my career path, I never forgot how Joe made everyone around him better, especially those that he mentored. Joe always understood that the people he influenced and trained were the most important products in Joe's academic environment. As a faculty mentor throughout my career, I always tried to follow the example that he had set. If I passed on to a student only half of the positive influence Joe passed to me, I'd consider that a success, and would know that the student would be better for it.

"Doc", thank you for being you. We will miss you.

#### Fred Fricke,

#### Director FDA Forensic Chemistry Center, Cincinnati, Ohio

I first met Professor Caruso when he became the science advisor to the Food and Drug Administration's (FDA) laboratory located in Cincinnati, Ohio, in 1972. At that time, the FDA was interested in developing analytical procedures to determine toxic elements such as mercury, lead, arsenic and cadmium in our food supply. During 1972, Joe, always a visionary, decided that he wanted to redirect his research interests. which involved potentiometric titrations in non-aqueous solvents, to the detection of toxic and nutritional elements in food

In 1973, the FDA selected Joe and me to participate in their Science Advisor Research Associate Program and to conduct research on the development of analytical methodology to determine trace elements in food. Joe then attracted several new graduate students that were very interested in doing their graduate work in this area. In 1974, he published his first work involving the detection of trace elements [1].

Later in 1974, we attended an ACS lecture by Rod Skogerboe, a professor at Colorado State, in which he described the use of a microwave plasma for simultaneous multielement analysis. Joe decided immediately that the focus of his research was going to be multielement analysis using a plasma as the ion source. His first work involving a plasma was published in Analytical Chemistry in 1975 [2]. This publication describes the interface of a carbon cup to a microwave plasma and the multielement emission from the plasma focused through a polychrometer to a Vidicon detector.

During the next two decades, Joe and his students modified microwave cavities to permit the introduction of liquids, hydrides, and effluents from chromatographic systems. They also developed sample preparation techniques for foods and biological tissues to permit them to be introduced into the plasma. His work with microwave plasmas allowed many researchers, who could not afford an ICP, to pursue the detection of trace elements in many different matrices.

Due to Joe's research on the detection of toxic and nutritional elements in food, and the collaboration with the FDA lab in Cincinnati, the FDA decided to establish an Elemental Analysis Research Center (EARC) in Cincinnati in 1980. This Center later became FDA's Forensic Chemistry Center.

Joe was well aware of the limitations of the microwave plasma with respect to matrix effects and that the ICP was the future of multielement analysis of liquid samples. Therefore, we convinced the FDA to purchase an ICP polychrometer for the Cincinnati lab. Joe and I thought that since his research interests were so closely aligned with the mission of the FDA that it would be great if we could establish a collaboration that would allow his graduate students to conduct a portion of their PhD work at the FDA's EARC. This collaboration is continuing today and serves as an exemplary model of university and government cooperation. I know all of Joe's students that participated in this program appreciate the opportunity that he gave them to obtain real world experience while pursuing their PhD.

In 1987, Joe published his first work on microwave plasma-mass spectrometry [3]. Joe subsequently obtained an ICP-MS and interfaced a high performance liquid chromatograph to it for the speciation of organotin compounds and published this work in 1989 [4].

Joe was a pioneer in using plasma mass spectrometry as a tool for elemental speciation. He established a partnership with the University of Cincinnati Medical School to determine elemental species in biological tissues and became a world-renowned expert in elemental speciation. Students from around the globe have come to Joe's lab to pursue their doctoral degree and conduct their post-doctoral studies.

2007 saw the appointment of Joe as director of the Metallomics Center of the Americas. The Center's charter is to support research in all fields related to the analysis of metals and metal species and their interactions within biological and ecological systems. In 2009, he was the founding Chair for Metallomics.

Joe was more than an advisor to his students. He truly treated them as members of his family and guided them during their graduate years so that when they left with their PhD they would be wonderful caring human beings and contribute greatly to the advancement of science.

Joe was my close friend for 43 years and I, along with everyone who had the privilege to know Joe, came away a better person. His contributions as scientist, mentor, and fantastic colleague will be immensely missed.

#### References

- 1. H. L. Huffman and J. A. Caruso, Journal of Agricultural and Food Chemistry (1974), 22, 824.
- 2. F. L. Fricke, O. Rose, J. A. Caruso, Analytical Chemistry (1975), 47, 2018.
- 3. R. D. Satzger, F. L. Fricke, P. G. Brown, and J. A. Caruso, Spectrochim. Acta (1987), 42B, 705.
- 4. H. Suyani, J. T. Creed, D. Heitkemper, and J. A. Caruso, Applied Spec. (1989), 43, 962.

#### Jack Creed, Research Chemist, National Exposure Research Laboratory, US EPA

Doc was a man with an infectious spirit who recognized the value of each and every individual. He was entertaining, quick witted and saw the need to foster a sense of family within everything he did. Some professors have research groups but Doc had a Research Family. I believe he and Judy extended this sense of Family to each and every student. A blessing I can never repay. At a recent birthday party for him, I introduced my 5-year-old to him and he said she was a late comer but a family member none the less (as he bent down to look into her eyes).

Joe's spirit and kind hearted ways were always front and center. I was truly blessed to have known him as a leader, as a mentor, and I consider it a privilege to be a lifelong Caruso Research Family member.

#### **Dr. William Heineman,** Distinguished Research Professor, University of Cincinnati

Joe Caruso was a wonderful colleague, but more importantly, a trusted friend. He was always optimistic, upbeat and enthusiastic, whether it was about a new research project, a conference he was organizing, or a new student joining his research group. Joe was an unusual academician in that he excelled not only in teaching and research, but also in administration. Joe maintained a vigorous research program during 28 years in administration, 13 years of which he was Dean of the McMicken College of Arts & Sciences. Many students, visiting scholars and postdocs worked in his lab and they were all treated as members of his family.

The University of Cincinnati recognized his many contributions when he received two of its highest honors: the George Rieveschl, Jr. Award for Distinguished Scientific Research in 1988, and the Award for Excellence in Doctoral Mentoring in 2004. We at UC will miss Joe's leadership, enthusiasm, research excellence and collegiality.

#### Maria Montes-Bayón,

Department of Physical and Analytical Chemistry. University of Oviedo



Joseph Caruso and Maria Montes-Bayón

Prof. Joseph Caruso's passing has left many of us with feelings of sorrow and orphanhood that will be difficult to overcome. His scientific contribution to the field of analytical chemistry and in particular, plasma spectrochemistry are well documented. Not only was he prolific with a high number of publications, but also scientifically significant to the field (H-index of 54).

Joe's supervision of students was successfully combined with important administrative work: first as departmental head (1981-1987) and then Dean of Arts and Sciences (1987-2000) at the University of Cincinnati. Such political/administrative work can only be done by someone with charm and intelligence. Even during these times, his office door was always open to students or post-docs. Joe had a special capability to get all of his work done without ever looking stressed or tense.

Finally, Joe's charisma was magnetic. He could joke or make an amusing comment about any situation and deal with the most complex circumstances in a very relaxed way. He tried to spread his good mood around and I hope we have learnt from him that science is not everything. A special mention to Joe's family and to Judy, his wife, who helped him create a home for students and friends in their house. Such a warm atmosphere was specially appreciated by foreign visitors, like me, so not to feel lonely during the first weeks of being away.

The plasma spectrochemistry community will miss an excellent scientist and a charming character. I will miss also an excellent mentor and a beloved friend.

#### Jenny Nelson, Applications Scientist, Agilent Technologies, USA



Jenny Nelson with Joe and Judy Caruso

I had the great privilege of meeting Professor "Doc" Caruso as an undergraduate chemistry student at the University of Cincinnati in 1998. Six years later, I had the even greater privilege of being taken under his wing: I had no idea what ICP-MS was or what his group did when I asked to join it. I just knew I needed to be around someone like him; I knew he was a person who would positively impact my life—but I had no idea how profound that impact would be.

Doc taught me what a true leader and boss should be like, and he led by example: he was understanding and respectful to everyone, no matter their background or previous challenges; he did whatever he could to help his students find contract work, post docs, and even permanent positions; and he encouraged his students to present and participate in conferences, often funding all of our travel. I will never forget the year he paid for his entire graduate group to travel to Sicily for the winter plasma conference.

His leadership and integrity set the tone for a graduate student atmosphere and a lab that was free from the drama that so often plagues pressured academic environments.

Doc would pay for students or post-docs research out of his own pocket if there was a lapse in their grants or funding at any point, or if they really needed financial help: that was just the type of guy he was. Most people don't know this about him, because he never talked about it: that was also the type of guy he was.

He knew that international students like me had another layer of challenges to work through in order to acclimatize to life in graduate school at UC, and he always went the extra mile to make us feel welcome.

The times I spent with Doc and his wife are among my warmest memories: group dinners and parties at their home, boat rides, and even a visit from Doc after he helped me get my first job at Chevron. In fact, he was the first person to come and visit me in Berkeley, CA, where I'd moved for the Chevron job. I will be forever grateful to Doc for helping me become the scientist, and more importantly, the person I am today.

#### Julio A Landero,

# **Research Assistant Professor, University** of Cincinnati

I met Joe in 2006, when I was a second year grad student under Kasia and Kasimierz Wrobel in Guanajuato Mexico. Following his remarkable lecture, he invited me and another student to visit his lab and run some samples. I visited for two weeks early the next year, and was invited to stay at his house. As a result, two publications were produced, but much more importantly, I found my second family. It is not an exaggeration to say that for me, my wife and my two sons, the Caruso's house was our second home.



By 2010, I started a one-year postdoc research in his lab but my stay continued to two, three, four, five, six years and counting!

His remarkable negotiating abilities, his perpetual good mood and his friendliness are qualities that I have on my personal improvement list. He showed me how to be successful with collaborations and students without being repressive, how to see the best in every person, and how to enjoy the personal and academic growth of the younger generations.

As a mentor, he was always that shade in the desert, the wisdom to be consulted, and a friend to share good and bad times, without judgment, making it easier to confront challenges and the uncertainty of our beloved profession.

It is an honor to follow in his footsteps and to represent him; I will always be a Joe Caruso group member.

## Fast Arsenic Speciation for Food and Urine Analysis

#### **Brian Jackson and Amir Liba**

Dartmouth College, Hanover, NH, USA Agilent Technologies Inc., USA

#### Introduction

Inorganic arsenic (iAs) is a class one carcinogen, top of the 2015 Agency for Toxic Substances and Disease Registry (ATSDR) Substance Priority List of pollutants, and a major health hazard worldwide. The major exposure routes to humans are through water and food consumption, and the major elimination route is through urine. Because As exists in different forms (species) in the environment, speciation analysis is necessary to quantify these individual species. The organic forms of As are less toxic than iAs and thus regulations mainly focus on the threshold limits for iAs, rather than total As.

Although many chromatographic methods have been applied to separate As species, ion chromatography (IC) coupled to ICP-MS is a wellestablished, yet still state of the art approach for As speciation analysis. A common analytical method is to use an anion exchange column interfaced with collision/reaction cell ICP-MS. The limiting step for sample throughput, however, is the length of the chromatographic separations, which are typically 10-20 minutes depending on the chromatographic method and the range of As species to be separated. Reducing the chromatographic runtime is highly desirable to improve sample throughput. Many populationbased human exposure studies measure thousands of urine samples, and the current Centers for Disease Control and Prevention (CDC) method involves a 17-minute runtime [1].

The impending EU food regulations for iAs in rice of 0.2 mg/kg for white rice and 0.1 mg/kg for rice products aimed at infants and young children will surely increase the need for high sample throughput iAs analysis, and similar regulations may well be forthcoming in the US (personal opinion).

The objective of our work was to develop a faster chromatographic

#### Chromatography

For this study we scaled down methods developed for a 250 mm Hamilton (Reno, NV) PRP-X100 column, to apply them to the new 50 mm column with a 5  $\mu$ m particle size.

Table 1. Mobile phase composition

Eluent A	Eluent B
50 mM (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	DI H <sub>2</sub> 0
3% methanol	3% methanol

The mobile phase was adjusted to 3% methanol to enhance the As sensitivity of the ICP-MS. The eluent flow rates were varied from 1 - 2 mL/min during the run. An Agilent 1260 Infinity Quaternary LC was used, interfaced via PEEK tubing connecting the column directly to the nebulizer of an Agilent 8800 ICP-QQQ. The 1260 LC was controlled directly from the ICP-MS MassHunter software. Two elution profiles were developed, one an isocratic separation suitable for the analysis of four As species in food, and the other a gradient method (Table 3), suitable for the separation of six As species in urine.

#### ICP-MS

We used an Agilent 8800 ICP-QQQ in MS/MS mode using  $O_2$  as the reaction cell gas. The first quadrupole, Q1, was set to 75 amu, preventing all ions from entering the cell, apart from As and any on-mass (m/z 75)interferences, while the second quadrupole, Q2, was set at 91 amu, allowing for the detection of the reaction product ion AsO+; As reacts quantitatively with  $O_2$  while common interferences or doubly charged species do not. The major operating parameters are shown in Table 2. Operating the ICP-QQQ peristaltic pump at 0.5 rps ensures that the spray chamber is properly drained at these high eluent flow rates.

Table 2. ICP-000	operating	conditions
------------------	-----------	------------

Carrier gas flow rate (L/min)	1.1
Nebulizer peristaltic pump (spray chamber drain) speed (rps)	0.5
O <sub>2</sub> cell gas flow (% of full scale)	30
Integration time As $(75 \rightarrow 91)$ (sec)	0.05

#### Reagents

Arsenobetaine (AB), mono-methylarsonic acid (MMA) and dimethylarsinic acid (DMA) were obtained as reagent grade solids (Chem Service, West Chester, PA), and 1 mg/L stock solutions were prepared in deionized water. As(III) and As(V) were purchased as 1000 ppm certified standards aqueous (Inorganic Ventures, Christiansburg, VA), and 1 ppm sub stock solutions were prepared daily. Arsenocholine (AC) was kindly supplied by Kevin Kubachka, USFDA. For quality control of the food analysis we used NIST (Gaithersburg, MD) SRM 1568b rice flour, certified for MMA, DMA and iAs, and for urine we used NIST 2669 urine SRM level 1 and level II. Sodium carbonate used for the mobile phase was purchased from Alfa Aesar (Ward Hill, MA).

#### Sample preparation

Rice flour (250 mg) was extracted in 9 mL 1.8% HNO<sub>3</sub> and 1mL 30%  $H_2O_2$ with heating at 80°C for 1 hour. The extract was centrifuged and an aliquot was diluted a further 5-fold in deionized water prior to analysis. Urine samples were diluted 5x in deionized water.

#### **Results and Discussion**

Our hypothesis was that the 50 mm x 5 um anion exchange column would provide sufficient resolving power to separate the variety of As species currently separated on longer versions of this column, and that the short column length and high flow rates would yield fast chromatography without requiring the high pressures used with UHPLC. The main chromatographic issue with the PRP-X100 column for As species is obtaining sufficient resolution for the early-eluting species (As(III) and DMA) and the non-retained compounds, AB and AC, which generally require a weak eluent and lower flow rate. After these species have eluted, the flow rate and eluent strength can be increased to elute the more strongly retained species, MMA and As(V). The method can be simplified greatly if As(III) is oxidized to As(V) prior to the chromatographic separation. This scenario is useful for food analysis, where the dilute acid extraction can promote method-induced changes in the inorganic As oxidation-state, and from a regulatory standpoint, it is only necessary to separate and quantify total iAs from the other species.

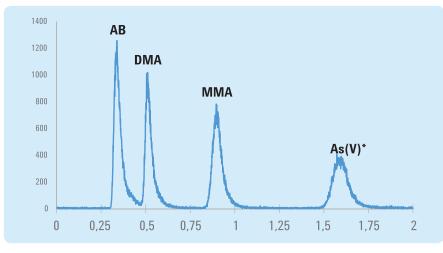


Figure 1. Isocratic separation of four arsenic species at 500 ng/L (ppt). 50 mM ( $NH_{4)2}CO_3$  at 1.5 mL/min flow rate. Column temp. of 35 °C. As(V)\* is the sum of As(V) and As(III) after oxidation.

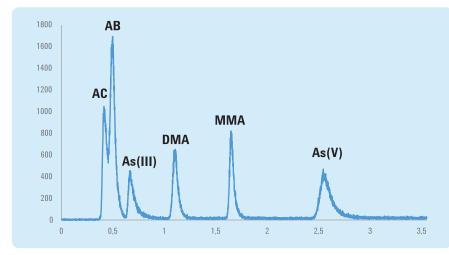


Figure 2. Separation of six arsenic species spiked at 400 ng/L (ppt) in urine using gradient conditions given in Table 3. Column temp. of 35 °C.

Table 3. Gradie	nt elution	profile for	urine sample
-----------------	------------	-------------	--------------

Time, min	Eluent A: (50mM (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> in 3% methanol, %	Eluent B: 3% methanol, %	Flow rate, mL/min
0	20	80	1
1.45	20	80	1
0.46	100	0	2
2.5	100	0	2
2.51	20	80	1
3.5	20	80	1

We used an isocratic separation with 50 mM ( $NH_4$ )<sub>2</sub>CO<sub>3</sub> (Eluent A) and a flow rate of 1.5 mL/min to separate, AsB, DMA, MMA and As(V)\*, where As(V)\* is now the total iAs (Figure 1). The separation was complete in 1.7 minutes and instrument detection limits of ca. 10 ng/L (ppt) for AB, DMA, MMA, and 50 ng/L for iAs, measured as As(V), were achieved. Recoveries for SRM 1568b (n=3) were 104%, 106%, 96% for DMA, MMA and total iAs, respectively, and

the CV of the triplicates was < 10% for each of the species.

For As speciation in urine, we used a gradient separation (Table 3) varying both flow rate and eluent strength to separate AC, AB, As(III), DMA, MMA, and As(V) (Figure 2).

AC was only partially separated from AB, but these compounds elute in the void volume and are often grouped together as 'un-retained organic arsenic'.

In practice, we have not detected AC in any of the urine samples analyzed in our laboratory. The resolution between As(III) and DMA improved at higher temperatures at the expense of longer retention times for MMA and As(V). As(V)elutes at 2.5 minutes and a further 1-minute re-equilibration time is included to re-establish initial conditions, giving a total runtime of 3.5 minutes, which compares favorably with the CDC method runtime of 17 minutes. Recoveries of NIST 2669 level 1 and II are generally very good (Table 4), although in multiple samples of these SRMs we have observed the time-induced oxidation As(III) to As(V) from the inevitable exposure to atmospheric oxygen. As(III) is detected in many of the urine samples we have run in our laboratory, and spike recoveries for As(III) were at nearly 100%. This suggests that the method is not causing the oxidation; rather it is more likely that the SRMs have been compromised, likely through thawing at some stage, causing the oxidation of As(III) to As(V). Also, tetramethylarsenic oxide (TMAO) and AC are added to NIST level II and are only partially resolved or unresolved from AB in this SRM.

Table 4. Recoveries for AB, DMA, MMA and As(V) in NIST 2669 levels 1 and 2. AB recoveries are just reported for level 1 (n = 14), DMA, MMA, As(V) are averages for levels I and II (n = 31)

Species	Recovery, %
AB	102 ±10
DMA	103 ±17
MMA	92 ±9
As(V) (sum total iAs)	110 ±16

#### **Conclusions**

These methods are 4 to 7 times faster than comparable separations reported in the literature, and should be very useful for increasing sample throughput for As speciation in response to changing regulations and increased need for biomonitoring.

#### **References**

- C. P. Verdon, K. L. Caldwell, M. R. Fresquez and R. L. Jones, Determination of seven arsenic compounds in urine by HPLC-ICP-DRC-MS: A CDC population biomonitoring method. Anal. Bioanal. Chem., 2009, 393(3), 939–947.
- B.P. Jackson, Fast ion chromatography-ICP-QQQ for arsenic speciation. *Journal of Analytical Atomic Spectrometry*, **2015**, 30, (6), 1405-1407.

### Scientists for a Day: Inspiring The Next Generation

Agilent's Tokyo Analytical Division (TAD) in Hachioji, Tokyo recently hosted a Science Day for 40 high school students interested in analytical methods and technologies.

The students started by brewing samples of several types of Japanese tea. Agilent engineers then showed them how to identify the specific teas using chromatography and mass spectrometry. The students also learned how to test soil samples using ICP-MS.

"Our 17 employee volunteers designed this workshop to show the students what forensic scientists actually do," said Hideki Seki, Communications Manager, Agilent Japan. "Our class looked just like a forensic lab. At the end of the session, the students went home inspired and happy."

## Four New Elements Verified by IUPAC

The periodic table now has its seventh row completed with the introduction of four new chemical elements with atomic numbers 113, 115, 117 and 118.

Elements 115, 117 and 118 were credited to a Russian-American team of scientists at the Joint Institute for Nuclear Research in Dubna, Russia and Lawrence Livermore National Laboratory in California, USA. A Japanese team at the Riken Institute was awarded credit for the discovery of element 113 by IUPAC.

The elements will be officially named within the next few months. IUPAC has invited the teams that discovered them to suggest names and symbols. Find out more at: http://www.iupac.org/

This information is subject to change without notice.

© Agilent Technologies, Inc. 2016 Printed in the U.S.A. February 08, 2016 5991-6568EN

# Fun In The Desert! Reflections from the 2016 Winter Conference on Plasma Spectrochemistry



Innovations in ICP-MS Lunch Seminar at WPC 2016

Almost 500 attendees participated at the 2016 Winter Conference in Tucson, Arizona, from January 10-16. There was a lot of excitement throughout the entire conference including the technical seminars, and social workshops, meetings. Hot topics included nanoparticle plasma analysis, new instrumentation, biological

& clinical applications, and speciation. Agilent held two lunch meetings on "Innovations in ICP-OES and ICP-MS Technology", with over 100 attendees in each session. There was also an "ICP-QQQ Workshop and User Meeting" to discuss the latest applications and workflows using MS/MS technology. Towards the end of the conference, Agilent held its annual customer appreciation dinner for around 140 guests. The Agilent team led a tribute to the late Dr. Joe Caruso, who had clearly touched so many in the plasma spectrochemistry community.

Thanks to everyone that participated and we look forward to seeing you again in 2017, in St. Anton, Austria!

https://ewcps2017.boku.ac.at/ewcps2017/EWCPS\_2107\_Sankt\_Anton.html

# **Conferences. Meetings. Seminars.**

PDAC, March 6-9, Toronto, ON, Canada, http://www.pdac.ca ACS Spring, March 13-17, San Diego, CA, US, www.acs.org European Symposium on Atomic Spectroscopy, March 31-2 April, Eger, Hungary, www.esas2016.mke.org.hu National Symposium on Environmental Nanotechnology and Nanoimpact, April 8-10, Xiamen, China, http://nsenn.xmu.edu.cn/index.asp Analytica Germany, May 10-13, Messe Munchen, Germany, http://www.analytica.de/index-2.html Spectr'Atom 2016, May 24-27, Pau, France, http://www.spectratom.com/ 13th European Workshop on Laser Ablation, July 12-15, Ljubljana, Slovenia, http://ewla2016.ki.si/ ASMS, June 5-9, San Antonio, TX, US, www.asms.org/

To view and download the latest ICP-MS literature, go to **www.agilent.com/chem/icpms** 

- **Application note:** Ultra-trace analysis of metals in mineral reference materials using the Agilent 7900 ICP-MS with UHMI, 5991-6406EN
- Application note: Gas chromatographic separation of metal carbonyls in carbon monoxide with detection using the Agilent 8800 ICP-QQQ, 5991-6432EN
- Application note: Routine soil analysis using an Agilent 8800 ICP-QQQ, 5991-6409EN

**Agilent ICP-MS Journal Editor** 

Karen Morton for Agilent Technologies e-mail: icpms@agilent.com



**Agilent Technologies**